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Edition 1.0 2018-08

TECHNICAL SPECIFICATION

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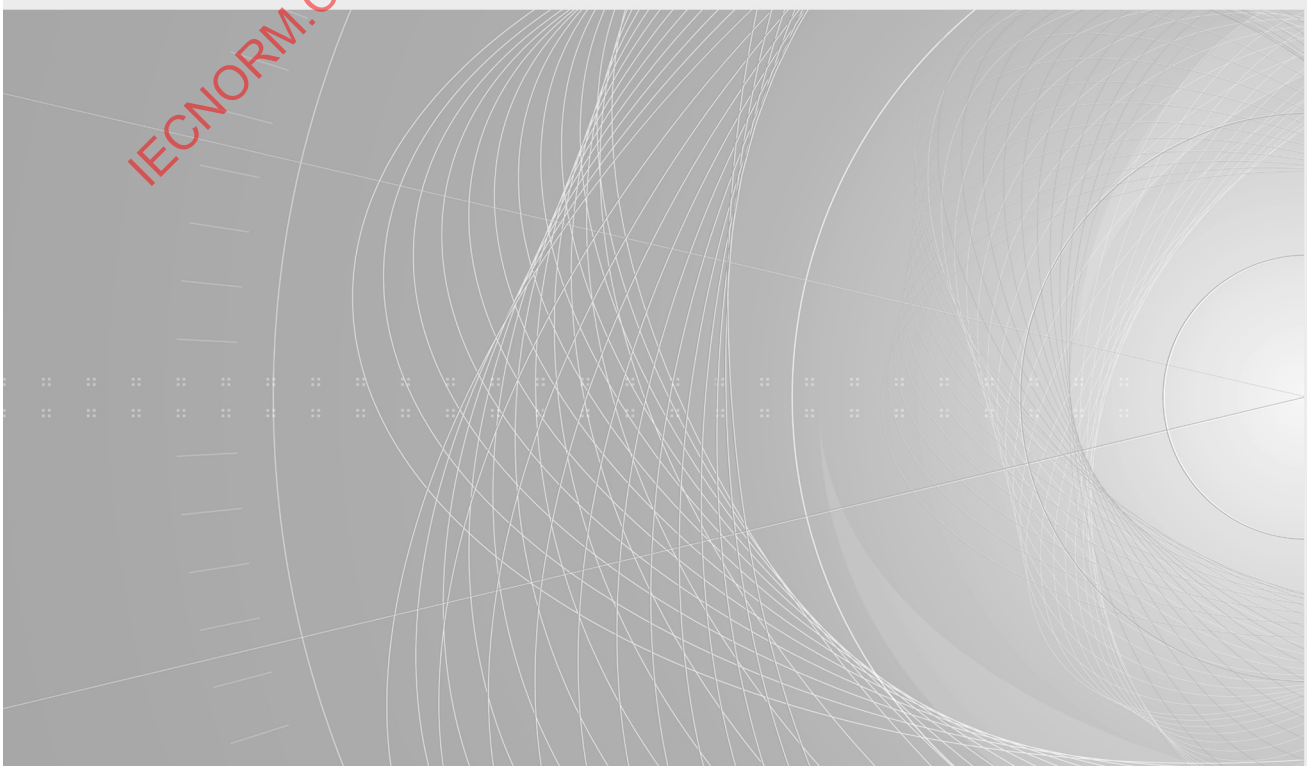
Nanomanufacturing – Key control characteristics – Part 4-7: Nano-enabled electrical energy storage – Determination of magnetic impurities in anode nanomaterials, ICP-OES method



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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 4-7: Nano-enabled electrical energy storage – Determination of magnetic impurities in anode nanomaterials, ICP-OES method

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Technical Specifications are subject to review within three years of publication to decide whether they can be transformed into International Standards.

IEC TS 62607-4-7, which is a Technical Specification, has been prepared by IEC technical committee 113: Nanotechnology for electrotechnical products and systems.

The text of this Technical Specification is based on the following documents:

Enquiry draft	Report on voting
113/405/DTS	113/430/RVDTS

Full information on the voting for the approval of this Technical Specification can be found in the report on voting indicated in the above table.

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

A list of all parts in the IEC TS 62607 series, published under the general title *Nanonmanufacturing – Key control characteristics*, can be found on the IEC website.

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

- transformed into an International Standard,
- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

A bilingual version of this publication may be issued at a later date.

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INTRODUCTION

Magnetic impurities often have significant influence on the performance of anode nanomaterials for nano-enabled electrical energy storage. If anode nanomaterials have high magnetic impurity content, it may lead to serious self-discharge and degrade the performance and cycle life. The magnetic impurities are also easy to aggregate, which can cause safety issues such as internal short-circuit fire, or even explosion. Therefore, accurate evaluation of magnetic impurity content is important for the safety and performance of energy storage devices. [1,2,3][1]

Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES) method is a mature and precise method to measure the concentration of metal elements in the tested materials, but the method to gather the magnetic substance has not been standardized. This document introduces a reliable method for gathering and determining the magnetic impurity content in the anode nanomaterials of an energy storage device. It will help to better control the quality of electrode nanomaterials.

This document introduces a determination of magnetic impurities using ICP-OES method for the electrochemical characterization of nano-enabled anode materials for electrical energy storage devices.

This standardized method is intended for use in comparing the characteristics of anode nanomaterials in the study stage, not for evaluating the electrode in end products.

The method is applicable to materials exhibiting function or performance only possible with nanotechnology, intentionally added to the active materials to measurably and significantly change the reliability or one or more physical properties of electrical energy storage devices.

In this context it is important to note that the percentage content of nanomaterials of the device in question has no direct relation to the applicability of this document, because minute quantities of nanomaterials are frequently sufficient to improve the performance significantly.

The fraction of nanomaterials in electrodes or electrode coatings is not of relevance for using this method.

[1] Numbers in square brackets refer to the Bibliography.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

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WARNING – Persons using this method should be familiar with normal laboratory practice. This document 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 and to ensure compliance with any national regulatory conditions.

IMPORTANT – It is absolutely essential that tests conducted according to this document be carried out by suitably trained staff.

1 Scope

This part of IEC TS 62607 provides a method for the determination of magnetic impurities in anode nanomaterials for energy storage devices using an Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES), including test overview, reagents, apparatus, test procedures, test results and test report.

This document applies to the determination of the total content of magnetic impurities (iron, cobalt, chromium, and nickel) ≥ 0,02 mg/kg which can be attracted by magnet.

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

ISO 6353-1:1982, *Reagents for chemical analysis – Part 1: General test methods*

ISO 18842:2015, *Aluminium oxide primarily used for the production of aluminium – Method for the determination of tapped and untapped density*

3 Terms and definitions

No terms and definitions are listed in this document.

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

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

4 Overview of the measurement

First, the magnetic impurities of samples are attracted by a magnet. And then the magnetic impurities are dissolved in acidic solution. Lastly ICP-OES is used to determine the content of iron, cobalt, chromium, and nickel.

5 Reagents

5.1 Nitric acid

$c(HNO_3)$ = 15 mol/L, $\rho \approx$ 1,40 g/mL. It shall be analytical reagent grade.

5.2 Hydrochloric acid

c(HCl) = 12 mol/L, $\rho \approx$ 1,18 g/mL. It shall be analytical reagent grade.

5.3 Nitrohydrochloric acid

Solution obtained by mixing one volume of nitric acid with three volumes of hydrochloric acid.

5.4 Ethanol

$w(C_2H_6O) \geq$ 99,7 %. It shall be analytical reagent grade.

5.5 Argon gas

It shall have volume fraction of not less than 99,99 %.

5.6 Standard solutions

5.6.1 Iron standard solution

In accordance with ISO 6353-1:1982, concentration 0,001 g/mL.

5.6.2 Cobalt standard solution,

In accordance with ISO 6353-1:1982, concentration 0,001 g/mL.

5.6.3 Chromium standard solution,

In accordance with ISO 6353-1:1982, concentration 0,001 g/mL.

5.6.4 Nickel standard solution,

In accordance with ISO 6353-1:1982, concentration 0,001 g/mL.

Subject to specific instructions, pure reagents and grade 3 water as specified in ISO 3696:1987 shall be used in the process.

6 Apparatus

6.1 ICP-OES

The technique ICP-OES is based on the measurement of emission at one wavelength highly selective for a specific element. For the determination of magnetic impurities, therefore, the test wavelengths are chosen in accordance with Table 1.

Table 1 –Recommended test wavelengths for the determination of magnetic impurities

Metal element	Wavelength nm
iron	238,204
cobalt	228,616
chromium	267,716
nickel	231,604

6.2 Conical flask

The conical flask is transparent and has a volume of 250 mL.

6.3 Sample tank

The sample tank is made of plastic material and has a volume of 500 mL, with inner and outer cover. The sample tank should be good sealing. Its calibre is consistent with the calibre of conical flasks.

6.4 Magnet

The magnet has 0,6 T of magnetic field intensity (allowable deviation within 5 %). Its diameter is 15 mm to 20 mm and its length is 45 mm to 55 mm. The magnet should be completely encapsulated with polytetrafluoroethylene materials, resistant to strong acid and alkali.

NOTE The magnetic field intensity (0,6 T) used in this document refers to the maximum magnetic field intensity of the magnet, which is probed at the surface of the magnet by the magnetometer.

6.5 Electronic balance

The balance has a resolution of 0,001 g.

6.6 Scroll equipment

The scroll mode of the equipment is shown in Annex A. The range of rotational speed is 60 rev/min to 100 rev/min.

6.7 Ultrasonic cleaning equipment

An ultrasonic cleaning device commonly used in the laboratory is applied to this document.

6.8 Heating device

The heating device has a capability for adjustment of heating temperature to boil aqueous solution, such as a hot plate or electric furnace.

7 Preparation of series of standard solutions

Prepare a series of standard solutions, and make sure that the concentration of the sample solution is in the linear range of the calibration curve. Beside blank solution, not less than three calibration standard solutions are prepared.

8 Test procedure

8.1 Cleaning the magnet

Put the magnet into a clean conical flask, add enough nitrohydrochloric acid and water to completely immerse the magnet. Heat the conical flask on a heating device until the solution boils gently. Keep it boiling for at least 30 min, ensuring that the solution does not dry out. During the heating process, the conical flask shall be shaken in a circular motion at least three times to avoid local overheating. After heating, cool the conical flask to room temperature in the atmosphere, and then wash the magnet three times with water. Reserve the magnet for the following steps.

NOTE During the process of pouring out the cleaning solution, place another magnet at the bottom of the outside of the conical flask to attract the inside magnet, which can prevent the magnet from being removed from the flask.

WARNING: When shaking the conical flask, protective measures shall be taken to avoid thermal burns on hands.

8.2 Test sample

Weigh the amount of the sample in a clean sample tank. The mass of the test sample is determined by the untapped bulk density of the sample. The method for measuring the untapped bulk density shall be in accordance with “untapped bulk density determination” method in ISO 18842:2015. See Table 2.

Table 2 – Correspondence of the measured mass to the untapped bulk density of the sample

Untapped bulk density g/cm^3	Measure mass g
< 0,5	50 ± 1,00
0,5 to 1,0	100 ± 2,00
> 1,0	200 ± 5,00

8.3 Attraction of magnetic impurities

Add enough ethanol (at least half of the volume of the sample tank) to the sample tank containing samples, and add the clean magnet to it. Cover securely, shake vigorously, and put the samples on a rolling device (see Annex A). Set the scrolling speed in the range of 60 rev/min to 80 rev/min. Roll for at least 30 min. The rolling process shall involve the samples being shaken vigorously at least three times.

NOTE For the validation of this attraction procedure, see Annex D.

8.4 Remove nonmagnetic impurities

After the rolling process, remove the magnet from the sample tank (this removal process is shown in Annex B) and wash it with water. During the removal process, avoid any physical contact with the magnet inside and outside the flask and superficial fracture of conical flask caused by the collision between magnet and conical flask. Add enough ethanol to the conical flask to completely immerse the magnet and clean them with the ultrasonic cleaning equipment until the magnet and conical flask are clean (usually ultrasonic cleaning for 1 min), and then wash the magnet and conical flask with water three times. In this process, the waste and ethanol should be properly disposed of (recycled if possible).

NOTE During the process of pouring out the cleaning solution, place another magnet at the bottom of the outside of the conical flask to attract the inside magnet, which can prevent the magnet from being removed from the flask.

8.5 Extraction of magnetic impurities

After cleaning, add enough nitrohydrochloric acid and water to completely immerse the magnet in the conical flask. Heat the conical flask on a heating device until the solution boils gently. Keep it boiling for at least 30 min (ensuring that the solution does not dry out). During the heating process, the conical flask shall be shaken in a circular motion at least three times to avoid local overheating, and the acid shall completely cover the surface of the magnet. After all particles have dissolved, the conical flask is cooled to room temperature in the atmosphere. Remove the solution after cooling to a 100 mL volumetric flask. Use a small amount of water to wash the conical flask and magnet three to four times, then transfer the water to the volumetric flask, and dilute it with water to 100 mL.

8.6 Preparation of blank sample

Carry out the procedures as described in 8.1, 8.3, 8.4 and 8.5 with no sample. The extract thus obtained at the end of step 8.5 is referred to as blank sample.

8.7 Test

ICP-OES is used to test the content of the magnetic materials. An example of the parameter settings of the instrument can be found in Table 3. Test the series of standard solutions in turn and draw calibration curve. Test blank sample and samples in the same way. The result of the samples is calculated by deduction of the blank sample result.

Table 3 – Instrument operating parameters

Operating parameter	Set value
Flow rate of plasma	15 L/min
Auxiliary flow rate	0,2 L/min
Flow rate of nebulizer	0,8 L/min
Power of the RF generator	1 300 W
Flow rate of sample	1,5 mL/min
Observation direction	Axial

During the test, these parameters should be appropriately adjusted according to the instrument as needed, in order to achieve optimum measurement conditions of the metals.

NOTE The specific steps for preparation of a series standard solution and test procedure are given in Annex C.

9 Data analysis and calculation of results

Formula (1) and Formula (2) are used to calculate the content of the magnetic impurities:

$$X_i = \frac{(c_i - c_0)v}{m} \quad (1)$$

$$X = X_{Fe} + X_{Co} + X_{Ni} + X_{Cr} \quad (2)$$

where

X_i is the content of a particular element in the sample, mg/kg;

c_i is the concentration of an element of the sample solution as determined from the calibration curve, μg/mL;

c_0 is the concentration of an element of the blank solution as determined from the calibration curve, μg/mL;

v is the volume of the sample solution, mL;

m is the mass of sample, g;

X is the total mass of the magnetic material, mg/kg.

The amount of elements below their instrument detection limit shall not be calculated in the total amount of magnetic impurities.

10 Test report

The test report shall include but is not limited to the following information:

- report number;
- test date;
- test person, the reviewers;
- measurement of the ambient temperature and humidity;
- sample description, including the manufacturer, the name, model, etc.;
- apparatus description, including the manufacturer, the name, model, etc.;
- test results.

Annex A
(informative)

Diagram of the scroll device

According to Figure A.1, the sample tank is placed on the scroll bar of the rolling device, which can prevent the sample from sliding.

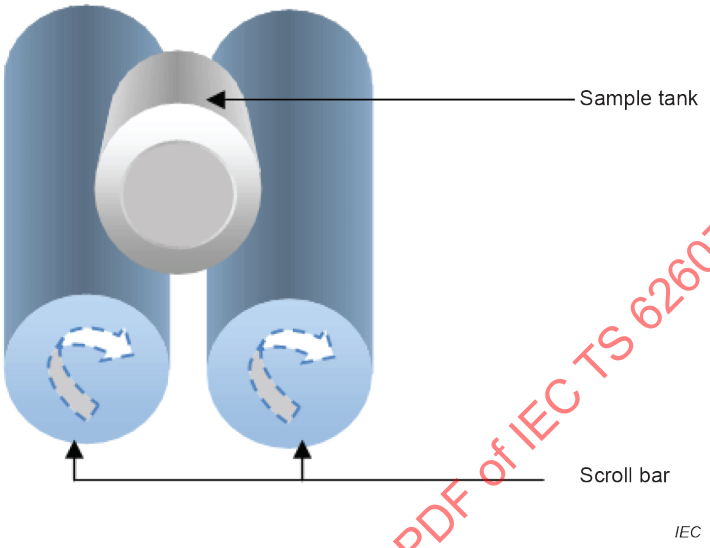

Figure A.1 – Diagram of sample tank on the rolling device

Annex B
(informative)

Magnet removal method

After rolling, place the samples flat on the test bench.

a) Attract the internal magnet using another magnet.

b) Hold the sample tank, unscrew the cover of the sample tank, and place the cover on the test bench.

c) Select a clean conical flask, put the flask upside down and insert the lip of the flask just inside the sample tank as shown in Figure B.1(5). Hold the conical flask and sample tank in one hand.

d) With the other hand, hold the magnet on the outside of the sample tank to attract the internal magnet. Slowly move the internal magnet up using the magnet on the outside of the sample tank until the internal magnet is inside the conical flask.

e) Revert the conical flask to its normal position and remove the outside magnet. The internal magnet has now been transferred from the sample tank to the conical flask.

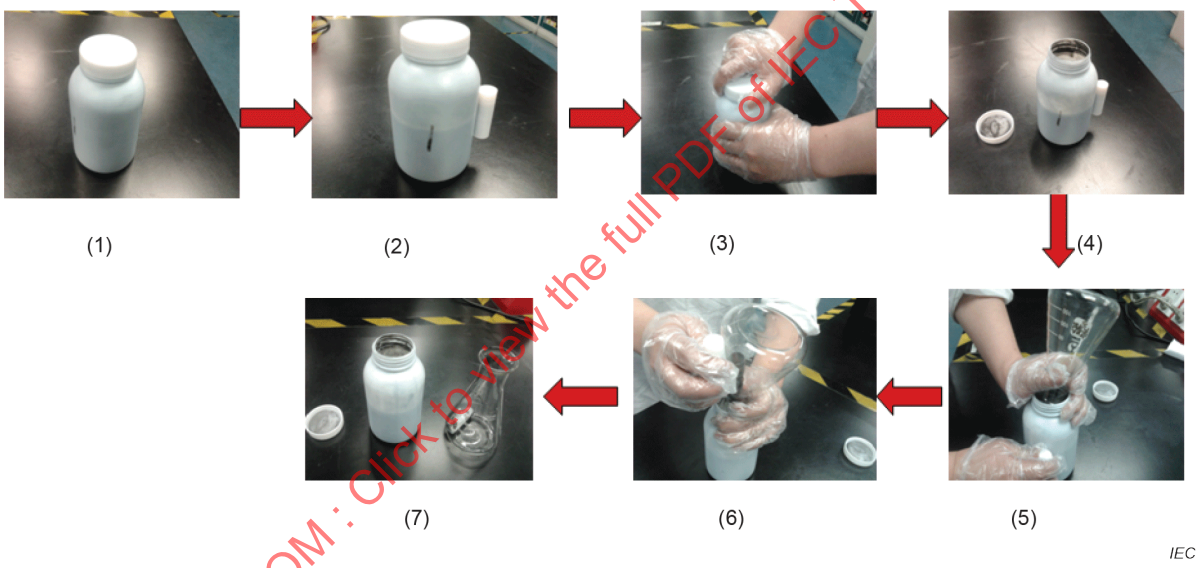

Figure B.1 – Diagram of magnet removal

Annex C
(informative)

Case study

C.1 Preparation of series of standard solutions

A series of standard solutions were prepared by diluting of standard solution as specified in Clause 7.

a) 5,0 mL iron, cobalt, chromium, and nickel standard solutions were placed in a 100 mL one-mark volumetric flask. 2,0 mL nitric acid was then added to the volumetric flask.

b) The solution was diluted with water to the mark. The standard stock solution of iron, cobalt, chromium, nickel concentration was 50,00 µg/mL.

c) 0,00 mL(calibration blank), 0,20 mL, 0,50 mL, 1,00 mL, and 2,00 mL standard stock solutions were measured and added to five 100 mL one-mark volumetric flasks. 2,0 mL nitric acid was added to each volumetric flask.

d) Each solution was diluted with water to the mark. A series of standard solutions of iron, cobalt, chromium, nickel with concentration of 0,00 µg/mL (calibration blank), 0,10 µg/mL, 0,25 µg/mL, 0,50 µg/mL, 1,00 µg/mL were now obtained.

C.2 Cleaning the magnet

a) Three magnets were put into three clean conical flasks, numbered 0, 1 and 2, and then 8 mL nitrohydrochloric acid and enough water were added to completely immerse the magnet in each conical flask as shown in Figure C.1(1).

b) The conical flasks were heated on a heating device as shown in Figure C.1(2) until the solution boiled gently. It was kept boiling for 30 min. During the heating process, the conical flasks were shaken in a circular motion at least three times to avoid local overheating.

c) After heating, the conical flasks were cooled to room temperature in the atmosphere, and then the magnets were washed three times with water as shown in Figure C.1(3). The magnets were reserved for the following steps.

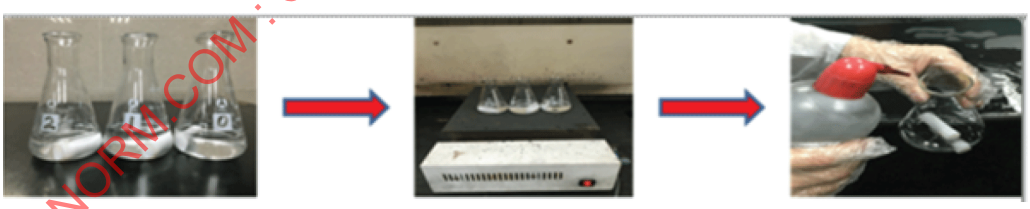

Figure C.1 – Procedure for cleaning the magnet

C.3 Weighing sample

Three clean sample tanks were taken, numbered 0, 1 and 2. Sample tank 0 was prepared for blank test. Sample tanks 1 and 2 were prepared for sample repeatability test. Weighing sample as specified in Table 2. Untapped bulk density of materials used in this test was in the range 0,5 g/cm^3 to 1,0 g/cm^3. The recorded sample masses were 0 g, 100,096 2 g, 100,861 8 g in sample tanks 0, 1 and 2, respectively, as shown in Figure C.2.

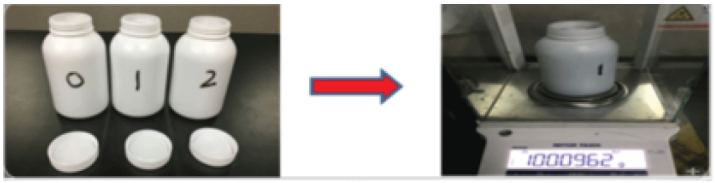

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Figure C.2 – Procedure for weighing sample

C.4 Attraction of magnetic impurities

a) The three clean magnets in Clause C.2 were put into each sample tank in Clause C.3 with one-to-one correspondence, as shown in Figure C.3(1).

b) 300 mL ethanol was added to each sample tank as shown in Figure C.3(2).

c) The covers of the sample tanks were unscrewed, and then the sample tanks were put on the scroll device as shown in Figure C.3(3). The rolling speed was set as 70 rev/min. And then the sample tanks were rolled for 30 min. During the rolling process, sample tanks were shaken vigorously at least three times.

(1) (2) (3)

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Figure C.3 – Procedure for attraction of magnetic impurities

C.5 Remove nonmagnetic impurities

a) The magnets from the sample tanks 0, 1 and 2 were transferred to the conical flasks marked with the corresponding number as shown in Figure C.4(1). The transfer method was as specified in Annex B.

b) The powder on the walls of conical flasks was cleaned with water as shown in Figure C.4(2).

c) Enough ethanol was added to completely immerse the magnets in the conical flasks. The magnets and conical flasks were cleaned using ultrasonic cleaning devices for 1 min as shown in Figure C.4(3). This process was repeated three times.

d) The magnets were cleaned with water three times as shown in Figure C.4(4).

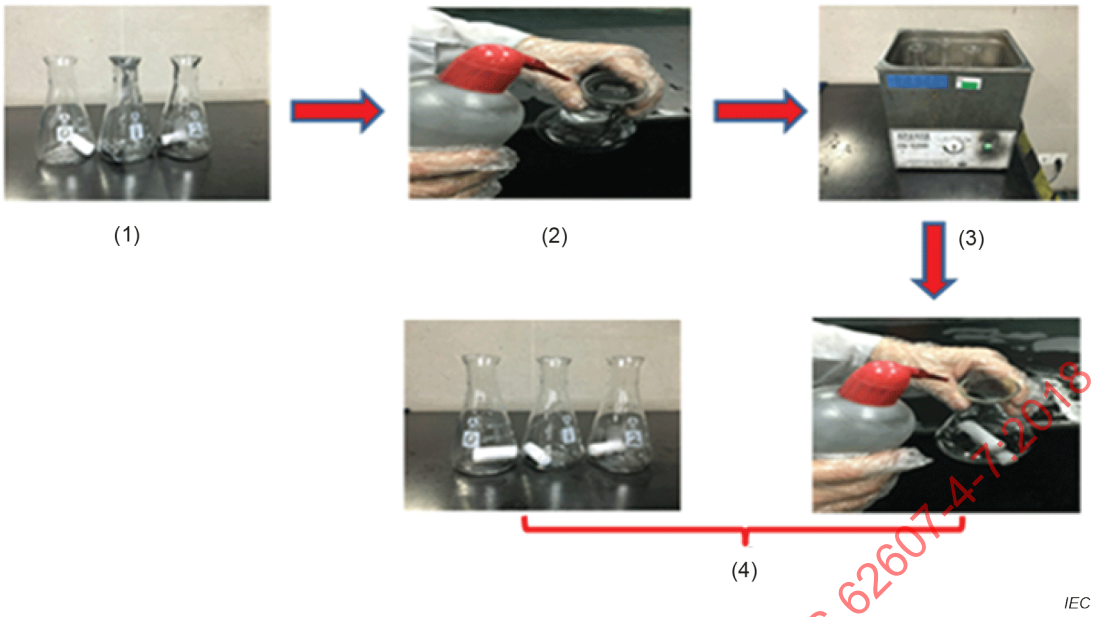

Figure C.4 – Procedure for removing nonmagnetic impurities

C.6 Extraction of magnetic impurities

a) 8 mL nitrohydrochloric acid was added in each conical flask and enough water was added to completely immerse the magnets as shown in Figure C.5(1).

b) The conical flasks were heated on a heating device as shown in Figure C.5(2) until the solution boiled gently. It was kept boiling for 30 min. During the heating process, the conical flasks were shaken in a circular motion at least three times to avoid local overheating.

c) After heating, the conical flasks were cooled to room temperature in the atmosphere, and then solution was transferred to three one-mark volumetric flasks with one-to-one correspondence. The solution was then diluted with water to 100 mL as shown in Figure C.5(4).

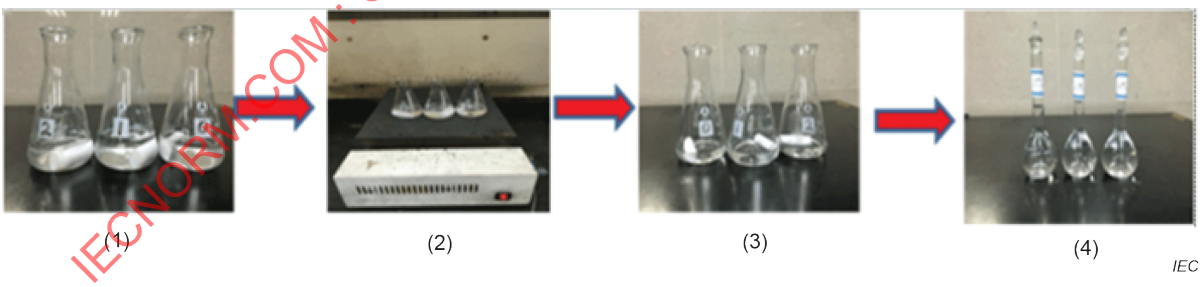

Figure C.5 – Procedure for extraction of magnetic impurities

C.7 Test

As specified in 8.7, the results of the samples which were calculated by deduction of the blank sample result are shown in Table C.1.