Standard Operating Procedure

Determination of elements in graphite and silicon-carbon anode materials for lithium ion batteries

Contents

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1 Purpose

This Standard Operating Procedure (SOP) describes the requirements for the ICP-OES determination of elements in samples of anode materials which are graphite-based, including silicon-carbon. These materials are used for lithium-ion batteries. This procedure is used for quality inspection and product acceptance testing. The procedure will determine the concentration of the elements, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Ga, K, Hg, Li, Mg, Mg, Mn, Mo, Na, Ni, Pb, Sb, Sr, Ti, V, and Zn.

2 Scope

Staff from the XXX department(s) at the XXX site(s) of the <company name> use this procedure.

This procedure is based on the Chinese Standard GB/T 24533-2019 (implemented on 1 February 2020) and GB/T 38823-2020 (implemented on 1 December 2020).

This procedure is suitable for samples of graphite-based, including silicon-carbon, anode materials that are used as the negative electrode in lithium-ion batteries.

3 Out of scope

The following is out of scope for this SOP:

Sample preparation methods
The sample preparation methods for ICP-OES analysis described in this SOP should be used for reference only. The procedures are based on the sample preparation methods for elemental analysis using ICP-OES outlined in GB/T 24533-2019 and GB/T 38823-2020. For detailed procedures for each group of elements, refer to the documents listed below.

- Trace elements Fe, Na, Cr, Cu, Ni, Al, and Mo in graphite: GB/T 24533-2019 Appendix H
- S in graphite: GB/T 24533-2019 Appendix J
- Magnetic matter (Fe+Co+Cr+Ni+Zn) in graphite: GB/T 24533-2019 Appendix K
- Regulated matter Cd, Pb, and Hg in graphite and silicon-carbon: GB/T 26125
- Trace elements Fe, Co, Cu, Ni, Al, Cr, and Zn in silicon-carbon: GB/T 38823-2020 Appendix C
- Magnetic matter (Fe+Co+Cr+Ni+Zn) in silicon-carbon: GB/T 33827

4 Process owner

Title of the process owner

5 Roles and responsibilities

The following table lists the roles and responsibilities of this SOP.

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Role	Responsibilities
Laboratory Manager /Supervisor – select appropriate title	Ensures that this procedure is kept up to date and Analysts are suitably trained to follow it. Ensures that the ICP-OES instrument is maintained as per the manufacturer's recommendations and suitable consumables and spares are available. Ensures Good Laboratory Practice has been implemented and is being followed, as per OECD guidelines or those issued by a relevant authority.
Analyst	To follow the instructions in this procedure and accurately create any required records.

6 Procedure

6.1 Safety instructions

All staff must refer to current OHS procedures available <insert where to find them>

Where applicable, refer to relevant sections in material safety data sheets of the materials used for first aid, handling and storage, and exposure control/personal protection measures.

Related safety documents/requirements include:

<insert number and title for relevant OHS SOPs and include them in the References section at the end of this SOP>

WARNING!

Cryogenic and suffocation hazard

Liquid argon represents a potential cryogenic and suffocation hazard. Safe handling procedures should be employed at all times when handling liquid argon tanks and fittings, and appropriate gas monitoring equipment (e.g. O_2 sensors) should be installed in laboratories where such gases are stored and used.

WARNING!

Inhalation hazard

Graphite powder represents an inhalation hazard with potentially damaging effect to lungs. Safe handling procedures should be employed at all times when handling graphite powder. This includes the use of a closely-fitted dust mask.

6.2 Equipment and reagents

Assemble the following equipment:

- One 500 mL wash bottle
- Twelves (12) x 50 mL stoppered quartz volumetric flasks for standards and QC solutions
- 50 mL polypropylene test tubes for sample solutions, minimum 1 per sample. (50 mL volumetric flasks can also be used, if preferred)

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- Calibrated mechanical pipettes and trace metal grade pipette tips in the following ranges:
  10-100 µL
  100-1000 µL
  1000-5000 µL
  1000-10000 µL
- Disposable syringes and disposable 0.45 µm disc filters. Hydrophilic PTFE filters are recommended.
- Talc powder-free disposable gloves.
- Dust mask

You will also need access to:

- A microwave digester
- An electronic balance, 0.0001 g accuracy
- Ultrapure Water System equivalent to ASTM Type 1 (ASTM D 1193) >18 MΩ/centimeter resistivity
- Extraction fume hood
- An Agilent 5800 ICP-OES, with the default sample introduction components supplied with the instrument
- Optional but recommended: an Agilent SPS 4 autosampler

For magnetic matter analysis (optional):

- A roller mixer, rotation rate range 60 to 100 rotations per min
- A hot plate or similar heating device with adjustable temperature control
- An ultrasonic bath, 28 to 33 kHz
- Glass conical flasks
- 500 mL cylindrical PTFE bottles with well-sealed double lids
- Magnetic stirrer bars with PTFE exterior (45 to 55 mm long x 15 to 20 mm in diameter)

6.3 Calibration standards, QC samples, and sample preparation

6.3.1 Reagents

Assemble the following reagents:

- Reagent water equivalent to ASTM Type 1 (ASTM D 1193) ≥18 MΩ/centimeter resistivity
- Nitric Acid (HNO_3), concentrated (SuperPure or equivalent recommended (68 to 70%))
- Hydrochloric Acid (HCl), concentrated (SuperPure or equivalent recommended (37%))
- Aqua regia (A combination of HCl and HNO_3 at a ratio of 3:1 V/V). If acids of lower concentrations are used, calculate the volumes of the acids so that the molar ratio of HCl to HNO_3 is close to 3:1.

WARNING 	**Chemical Hazard** Nitric acid, hydrochloric acid, and strong alkali solutions are very corrosive and can cause severe burns when they come into contact with the skin. Preparation solutions should be done under an extraction fume hood. It is essential that appropriate protective clothing is worn at all times when handling these acids. If acid contacts the skin, wash off with copious amounts of water and seek medical attention immediately.

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For magnetic matter analysis (optional):

- Anhydrous alcohol

6.3.2 Prepare blank solution

Prepare the following blank solution for calibration. It is recommended to prepare a fresh blank on the same day as analysis.

24% aqua regia in ASTM Type1 ultrapure water. Prepare by diluting 30 mL of HNO_3 and 90 mL HCl to 500 mL wash bottle. Make up to 500 mL by adding ultrapure water.

6.3.3 Prepare calibration stock solutions

Assemble or prepare the following stock solutions for calibration.

- Multi-element Standard 2A. (Available from Agilent - part number 8500-6940)
    - Bottle 1: containing 10 mg/L of Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cs, Cu, Fe, Ga, K, Li, Mg, Mn, Na, Ni, Pb, Se, Sr, Tl, V, Zn in a matrix of 5% HNO_3
    - Bottle 2: 10 mg/L Hg in a matrix of 5% HNO_3
- Molybdenum, Mo, single element standard 1,000 ug/mL in 1% NH_4OH (Available from Agilent - part number 5190-8487)
- Antimony, Sb, single element standard 1,000 ug/mL in 1% HNO_3, trace tartaric acid (Available from Agilent - part number 5190-8244)
- Titanium, Ti, single element standard 1,000 ug/ml in H_2O (Available from Agilent – part number 5190-8545)
- Yttrium, Y, single element standard 10,000 µg/mL in 5% HNO_3 (Available from Agilent - part number 5190-8233)
- Rubidium, Rb, single element standard 10,000 µg/mL in 5% HNO_3 (Available from Agilent - part number 5190-8441)
- Barium, Ba, single element standard 1,000 ug/mL in 5% HNO_3 (available from Agilent – part number 5190-8248)
- Sodium, Na, single element standard 1,000 ug/ml in 5% HNO_3 (available from Agilent – part number 5190-8525)
- Potassium, K, single element standard 1,000 ug/ml in 5% HNO_3 (available from Agilent – part number 5190-8503)

Note: The use of an internal standard is strongly recommended to correct for any unexpected matrix interferences that might be present in the samples. The use of a mixed 5 mg/L Y and 100 mg/L Rb internal standard is described in this standard operating procedure.

6.3.4 Prepare intermediate stock solution A

Intermediate Stock A: Mo, Sb, and Ti – 10 mg/L. Add 0.5 mL of each of the single element standards for Mo, Sb and Ti to a 50 mL volumetric flask. Make up to 50 mL with the 24% Aqua Regia blank solution.

6.3.5 Prepare multi-element calibration standards:

Prepare the multi-element calibration standards listed in the table below from the stock solutions described in 6.3.3, the intermediate stock solution described in 6.3.4 and the blank solution described in 6.3.2. Use the 24% Aqua Regia blank solution as the diluent for the calibration standards.

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