Agilent

Trusted Answers

Standard Operating Procedure

Determination of elements in lithium iron phosphate cathode materials for lithium ion batteries

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Contents

Determination of elements in lithium iron phosphate cathode materials for lithium-ion batteries

1 Purpose

This Standard Operating Procedure (SOP) describes the requirements for the determination of elements in lithium iron phosphate (LFP) cathode materials using an Agilent 5800 ICP-OES instrument. These materials are used in the manufacture of lithium-ion batteries. This procedure is used for quality inspection and product acceptance testing. The procedure will determine the concentration of the major elements, Li, Fe, and P, and a full suite of 28 trace elements Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Hg, K, Mg, Mn, Mo, Na, Ni, Pb, S, Sb, Se, Si, Sn, Sr, Ti, V, W, and Zn.

2 Scope

Staff from the XXX departments at the XXX sites of the <company name> use this procedure.

This procedure is based on the Chinese Standard GB/T 30835-2014 (implemented on 1 April 2015).

This procedure is suitable for samples of lithium iron phosphate that are used as the cathode (positive electrode) in lithium-ion batteries.

3 Out of scope

The following is out of scope for this SOP: Analysis of other types of cathode materials.

4 Process owner

Title of the process owner

5 Roles and responsibilities

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

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.

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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 always be used 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

Lithium iron phosphate represents a hazard with potentially damaging effect to lungs, skin, and eyes. Safe handling procedures should be employed at all times when handling samples. This includes the use of closely fitted dust masks, gloves, and protective clothing.

6.2 Equipment and reagents

Assemble the following equipment:

- One 500 mL volumetric flask and one 1000 mL volumetric flask
- PTFE weighing spatulas
- Fifteen (15) x 50 mL stoppered quartz volumetric flasks for intermediate stock and calibration standards
- 50 mL volumetric flasks or 50 mL polypropylene test tubes for QCs and sample solutions (minimum 1 per sample)
- Calibrated mechanical pipettes and trace metal grade pipette tips in the following ranges:
  20 to 200 µL
  100 to 1000 µL
  1000 to 5000 µL
- Talc powder-free disposable gloves.
- Disposable syringes and disposable 0.45 µm disc filters. Hydrophilic PTFE filters are recommended. (Filtering may be required if LFP samples has been loaded onto carbon particles)

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You will also need access to:

- Electronic balance
- Microwave assisted digestion system
- Ultrapure Water System equivalent to ASTM Type 1 (ASTM D 1193) >18 MΩ/centimeter resistivity
- Extraction fume hood
- An Agilent 5800 ICP-OES with default sample introduction components supplied with the instrument
- Argon humidifier
- Optional but recommended: an Agilent SPS 4 autosampler

6.3 Calibration standards and sample preparation

6.3.1 Reagents

Assemble the following reagents:

- Ultrapure water
- Nitric Acid (HNO_3), concentrated (reagent grade for trace metal analysis or equivalent recommended)
- Hydrochloric Acid, (HCl) concentrated (reagent grade for trace metal analysis or equivalent recommended)

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.

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:

5% Aqua Regia (3:1 HCL to HNO_3) solution in ASTM Type1 ultrapure water.
Prepare by transferring 37.5 mL of concentrated hydrochloric acid and 12.5 mL of concentrated nitric acid to a 1000 mL volumetric flask. Make up to 1000 mL by adding ultrapure water.

Also, you will need to prepare enough of this same solution for rinse (about 4 L) for the autosampler. A smaller volume may be prepared, if manually sampling.

6.3.3 Calibration stock solutions

Assemble or prepare the following stock solutions:

- Multi-element Quality Control Standard, (QC27) containing 100 µg/mL of Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Si, Se, Sr, Ti, Tl, V, Zn in a matrix of 5% HNO_3 with trace hydrofluoric acid (Available from Agilent - part number 5190-9418)

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- Mercury, Hg, single element standard in 5% HNO_3, 1,000 µg/mL (Available from Agilent – part number 5190-8485)
- Tin, Sn, single element standard 1,000 µg/mL in 20% HCL (Available from Agilent – part number 5190-8543)
- Tungsten, W, single element standard 1,000 µg/mL in 5% HNO_3 with trace hydrofluoric acid (Available from Agilent – part number 5190-8547)
- Iron, Fe, single element standard 10,000 µg/mL in 5% HNO_3 (Available from Agilent - part number 5190-8403)
- Lithium, Li, single element standard 1,000 µg/mL 5% HNO_3 (Available from Agilent - part number 5190-8289)
- Phosphorus, P, single element standard 10,000 µg/mL 5% HNO_3 (Available from Agilent - part number 5190-8429)
- Sulfur, S, single element standard 1,000 µg/mL in H_2O (Available from Agilent – part number 5190-8529)
- Yttrium, Y, single element standard 10,000 µg/mL 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)

Note: The SO_4^{2-} content of the sample can be calculated based on the measured S content. Preparation of a separate standard calibration curve is advised when measuring sulfur at µg/mL levels. Avoid using calibration solutions that contain both Pb and S if an accurate measurement of S is required. Agilent supplies a multi-element calibration standard containing S without Pb (part number 8500-6942)

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 75 mg/L Rb internal standard is described in this standard operating procedure.

6.3.4 Preparing intermediate stock solutions A and B

Make up two intermediate stock solutions (A and B) as follows:

- **Intermediate Stock A** – Trace Elements – 10 ppm: Add 5 mL of the Multi-Element Quality Control Standard, (QC27), and 0.5 mL of each standard for Hg, Sn, and W to a 50 mL volumetric flask. Make up to 50 mL with 5% Aqua Regia blank solution
- **Intermediate Stock B** – Sulfur – 10 ppm: Add 0.5 mL of the sulfur standard to a separate 50 mL flask. Make up to 50 mL with ultra pure water.

6.3.5 Prepare multi-element calibration

Prepare the multi-element calibration standards listed in the table below from the stock solutions described in 6.3.3, the intermediate stock solutions described in 6.3.4, and the blank solutions described in 6.3.2. Use the 5% Aqua Regia blank solution as the diluent for the calibration standards.

Prepare each standard (and the Blank) in a 50 mL quartz volumetric flask which has been rinsed three times with the blank solution. Label each volumetric flask with the standard number e.g. Std 2. Note: If preparing in flask and using an autosampler, you will need to transfer each standard to a tube that can be placed on the autosampler rack.

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