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Nonferrous Metals Industry Standard of the People's Republic of China

YS/T 582—2013
Replacing YS/T 582—2006

Battery Grade Lithium Carbonate

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Foreword

This Standard is drafted in accordance with rules given in GB/T 1.1—2009.

This Standard replaces *Battery Grade Lithium Carbonate* (YS/T 582—2006). Compared with YS/T 582—2006, the main changes of this Standard are as follows:

—— Some product indexes are adjusted;

—— Requirements for magnetic impurity and hazardous material are added;

—— Measurement method for moisture is modified.

This Standard shall be under the jurisdiction of National Standardization Technical Committee of Nonferrous Metals (SAC/TC 243).

Drafting organizations of this Standard: Sichuan Tianqi Lithium Industries Inc., Foshan Brunp Recycling Technology Co., Ltd., Haimen Ronghui General Lithium Co., Ltd., and Jiangxi Ganfeng Lithium Co., Ltd.

The main drafters of this Standard: Jin Peng, Huo Liming, Li Nanping, Tu Mingjiang, Li Changdong, Jiang Hucheng, Song Shuangbing, Yu Haijun, Xie Shaozhong and Li Fangqin.

The historical version replaced by this Standard is as follows:

—— YS/T 582—2006.

Battery Grade Lithium Carbonate

1 Scope

This Standard specifies the requirements, testing method, inspecting rules, marking, packaging, transportation, storage and quality certificate as well as content of contract (or purchasing order) for battery grade lithium carbonate.

This Standard is applicable to battery grade lithium carbonate that is manufactured in various methods.

2 Normative Reference

The following documents are indispensable for the application of this document. For the dated documents so quoted, only the dated versions apply to this document. For the undated documents so quoted, the latest versions (including all modification sheets) apply to this document.

GB/T 191	Packaging - Pictorial Markings for Handling of Goods
GB/T 6284	Chemical Products for Industrial Use - General Method for Determination of Water Content - The Loss of Mass on Drying Method
GB/T 6678-2003	General Principles for Sampling Chemical Products
GB/T 11064 (All Parts)	Methods for Chemical Analysis of Lithium Carbonate, Lithium Hydroxide Monohydrate and Lithium Chloride
GB/T 19077.1	Particle Size Analysis - Laser Diffraction Methods - Part 1: General Principles
IEC 62321	Electrotechnical Products - Determination of Levels of Six Regulated Substances (Lead, Mercury, Cadmium, Hexavalent Chromium, Polybrominated Biphenyls, Polybrominated Diphenyl Ethers)

3 Terms and Definitions

The following terms and definitions are applicable in this Standard.

3.1 Magnetic impurity

The material which can be attracted by ferromagnet directly or indirectly. In this Standard, it refers to total content of three elements - iron, zinc and chromium.

4 Requirements

4.1 Chemical composition

Chemical composition of the product shall be as specified in Table 1. If required by the Buyer, hazardous substances shall comply with Annex II to Directive 2011/65/EU of the European Council.

Table 1 **%**

Li_2CO_3	Impurity content, not more than													
	Na	Mg	Ca	K	Fe	Zn	Cu	Pb	Si	Al	Mn	Ni	SO_4^{2-}	Cl^-
≥99.5	0.025	0.008	0.005	0.001	0.001	0.0003	0.0003	0.0003	0.003	0.001	0.0003	0.001	0.08	0.003

4.2 Magnetic impurity

Magnetic impurity content in the product shall be ≤0.0003%.

4.3 Water content

Water content in the product shall be ≤0.25%.

4.4 Particle size

$d_{10} \geqslant 1\ \mu m, 3\ \mu m \leqslant d_{50} \leqslant 8\ \mu m; 9\ \mu m \leqslant d_{90} \leqslant 15\ \mu m$。

4.5 Appearance quality

The product is white powder with no visible inclusions.

5. Test Methods

5.1 Chemical component analysis of the product shall comply with GB/T 11064. Determination of hazardous substance shall be in accordance with IEC62321.

5.2 Determination of magnetic impurity in the product shall be in accordance with Appendix A.

5.3 Determination of water content in the product shall be in accordance with GB/T6284.

5.4 Determination of particle size in the product shall be accordance with GB/T19077.1.

5.5 Appearance quality of the product shall be determined by visual inspection.

6. Inspection Rules

6.1 Inspection and acceptance

6.1.1 The product shall be inspected by the supplier who shall ensure its quality meets this Standard and contract (or purchasing order) requirements and complete quality certificate.

6.1.2 The buyer shall inspect the product received in accordance with this Standard, and in case of any discrepancy with this Standard or quality certificate, shall inform the supplier of such discrepancy within 3 months of receiving the product for negotiated settlement by both parties. If arbitration is needed, sampling for arbitration shall take place at the buyer's premises.

6.2 Batching

The product shall be delivered for acceptance in batches, each comprising the same mixture. Net weight of each batch shall be 2t-10t.

6.3 Inspection items and sampling

Inspection items and sample numbers shall be as specified in Table 2.

Table 2

Inspection item	Sampling requirement	Required clause No.	Clause No. of test method
Chemical composition	As specified under 7.6 of GB/T 6678—2003, stainless steel or UPVC sampler shall be used by inserting sampling tube to 2/3 in the center of the bag; the samples obtained are mixed evenly and quartered to about 200 g.	4.1	5.1
Magnetic impurity		4.2	5.2
Water content		4.3	5.3
Particle size		4.4	5.4
Appearance quality		4.5	5.5

6.4 Determination of inspection result

6.4.1 If the inspection result of chemical composition of the product fails, then twice as many samples shall be taken from the same batch for repetitive inspection on nonconforming

item; if there is still any inspection result that fails, then this batch is determined as nonconforming.

6.4.2 If appearance quality of the product fails the inspection, then twice as many samples shall be taken from the same batch for repetitive inspection on nonconforming item; if there is still any inspection result that fails, then this batch is determined as nonconforming.

6.4.3 If magnetic impurity in the product fails the inspection, then twice as many samples shall be taken from the same batch for repetitive inspection on nonconforming item; if there is still any inspection result that fails, then this batch is determined as nonconforming.

6.4.4 If water content of the product fails the inspection, then this batch is determined as nonconforming.

6.4.5 If particle size of the product fails the inspection, then this batch is determined as nonconforming.

7 Marking, Packaging, Transportation, Storage and Quality Certificate

7.1 Marking

Product packing bag shall indicate:

a) Product name;

b) Batch No.;

c) Gross weight;

d) Net weight;

e) Main content;

f) Supplier name;

g) Applicable standard;

h) Place of origin;

i) "Keep away from rain" symbol specified in GB/T191.

7.2 Packaging

The product is packed in a bag with a plastic inner layer and a plastic woven outer layer (or packed in film coating bag). The top of the inner bag is tied closely or heat sealed while the top of the outer bag knitted firmly. Net weight per bag shall be as required by the customer.

7.3 Transportation

During transportation, the product shall be kept away from acid; during handling, the packing bag shall not be damaged and the product shall be protected from moisture.

7.4 Storage

The product shall be stored in a dry place with no acid corrosion atmosphere.

7.5 Quality certificate

Each batch of product shall be accompanied by quality certificate indicating:

a) Supplier name, address, phone and fax numbers;

b) Product name;

c) Reference number of this Standard;

d) Batch No.;

e) Release date;

f) Inspection result.

8 Content of Contract (or Purchasing Order)

Content of Contract (or Purchasing Order) shall include:

a) Product name;

b) Quantity;

c) Reference number of this Standard;

d) Other information.

Appendix A

(Normative)

Determination of Magnetic Impurity Level in Battery Grade Lithium Carbonate by ICP-OES (Inductively Coupled Plasma Optical Emission Spectrometer)

A.1 Method summary

Magnetic impurities in the specimen are adsorbed by magnetic bar, decomposed by aqua regia and examined by ICP-OES which determines the content of iron, zinc and chromium; total content of the three elements is the content of magnetic impurities.

A.2 Reagent

A.2.1 Grade I water

A.2.2 Hydrochloric acid (ρ=1.19 g/mL), GR.

A.2.3 Nitric acid (ρ=l.42 g/mL), GR.

A.2.4 Nitric acid (1 + 1).

A.2.5 Hydrochloric acid (1 + 1).

A.2.6 Aqua regia: mix nitric acid (A.2.3) with hydrochloric acid (A.2.2) as per a volume ratio of 1:3 before use.

A.2.7 Iron standard storage solution: place 1.0000 g pure metal wire (iron mass fraction ≥9.99%) in 200 mL beaker; add 20 mL hydrochloric acid (A.2.5); put the beaker on water bath until the solution is clear and cooled to room temperature. Move the solution into 1000 mL volumetric flask, add water to scale and shake up. 1 mL of this solution contains 1 mg iron.

A.2.8 Zinc standard storage solution: place 1.0000 g pure metal zinc (purity 99.99%) in 200 mL beaker; add 20 mL hydrochloric acid (A.2.5); put the beaker under low temperature until the solution is clear and cooled to room temperature. Move the solution into 1000 mL volumetric flask, add water to scale and shake up. 1 mL of this solution contains 1 mg zinc.

A.2.9 Chromium standard storage solution: place 1.0000 g pure metal chromium (purity 99.99%) in 200 mL beaker; add 50 mL hydrochloric acid (A.2.5); put the beaker under low temperature until the solution is clear and cooled to room temperature. Move the solution into 1000 mL volumetric flask, add water to scale and shake up. 1 mL of this solution contains 1 mg chromium.

A.2.10 Mixed standard solution A: move 20.00 mL of each standard storage solution (A.2.7~A.2.9) into 200 mL volumetric flask; add 20 mL nitric acid (A.2.4); add water to scale and shake up. 1 mL of this solution contains 100 μg iron, zinc and chromium.

A.2.11 Mixed standard solution B: move 10.00 mL mixed standard solution A (A.2.10) into 100 mL volumetric flask; add 20 mL nitric acid (A.2.4); add water to scale and shake up. 1 mL of this solution contains 10 μg iron, zinc and chromium.

A.3 Instrument and material

A.3.1 Inductively Coupled Plasma Optical Emission Spectrometer

A.3.2 Ultrasonic generator (100 W, 40 kHz)

A.3.3 Magnetic bar (6000 Gs~8500 Gs, Φ30 mmX100mm).

A.3.4 Argon [φ (Ar) ≥99.999%].

A.4 Specimen

Battery grade lithium carbonate

A.5 Analysis steps

A.5.1 Sample

Take 200 g~250 g specimen to the accuracy of 0.1 g; prepare duplicate samples.

A.5.2 Blank experiment

Conduct blank experiment along with the specimen.

A.5.3 Determination

A.5.3.1 Put sample (A.5.1) into 500 mL plastic wide-mouth bottle; place magnetic bar into the bottle and slowly add 250 mL grade I water (A.2.1); put in electric magnetic stirrer to stir for 30 min at 100 r/min.

A.5.3.2 Take out the magnetic bar; remove lithium on the surface of the magnetic bar with flushing water; put the bar into 500 mL glass beaker; add 150 mL~200 mL water for ultrasonic cleaning for 2.0 min.

A.5.3.3 Add 15 mL aqua regia (A.2.6) in the glass beaker; place the beaker on heating plate to heat to slight boiling for 20 min; cool to room temperature.

A.5.3.4 Remove the magnetic bar from glass beaker and wash it 3-5 times; move sample solution into 250 mL volumetric flask; measure the solution with ICP-OES according to analysis spectral line in Table A.1 in parallel with blank experiment (A.5.2).

Table A.1

Element	Fe	Cr	Zn
Wave length/nm	259.9	267.7	213.8

A.5.4 Drawing working curve

A.5.4.1 Add 15 mL prepared aqua regia (A.2.6) in 6 glass beakers respectively; place them on heating plate to heat to slight boiling for 20 min. After being cooled to room temperature, the solution is moved into six 100 mL volumetric flasks; add 0.00 mL, 0.20 mL, 0.50 mL, l.00 mL, 2.00 mL, 5.00 mL mixed standard solution B (A.2.11) respectively into the flasks; add water to scale and shake up. Concentration of each element in standard solution is shown in Table A.2.

Table A.2

Standard series	1	2	3	4	5
Element concentration/ (μg/mL)	0.020	0.050	0.100	0.200	0.500

A.5.4.2 Measure standard series solution with ICP-OES according to analysis spectral line in Table A.1. Draw working curve with element concentration in standard solution as x-coordinate and emission intensity as y-coordinate.

A.6 Calculation of analysis result

The content of each element is calculated as their mass fraction ω_x expressed in % according to Eq. (A.1); total content of iron, zinc and chromium is the content of magnetic impurities:

$$w_X = \frac{(\rho_1 - \rho_0) \cdot V_1}{m_0 \times 10^6} \times 100 \qquad \cdots\cdots (A.1)$$

where

ρ_1——element concentration in the test solution obtained from working curve, in μg/mL;

ρ_0——element concentration in blank solution obtained from working curve, in μg/mL;

V_1——volume of test solution, in mL;

m_0——sample amount, in g.

A.7 Admissible error

The difference of analysis results between labs shall not exceed the admissible error given in Table A.3.

Table A.3

Mass fraction of magnetic impurities/%	Admissible error
0.0002~0.0010	0.0001