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## Lithium manganese oxide

锰酸锂

（*English Translation*）

（送审稿）

YS/T 677—2016

Replace YS/T 677—2008

**Nonferrous Metal Industry Standard of the People’s Republic of China**

ICS 77.160

H 71

**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 standard is drafted in accordance with the rules given in the GB/T 1.1-2009.

This standard replaces the YS/T 677-2008 *Lithium manganese oxide*.

In addition to a number of editorial changes, the following technical deviations have been made with respect to the YS/T 677-2008.

—New normative references are added.

—New terms and definitions are added.

—The classification of the lithium manganese oxide products is added.

—The performance indexes of different types of lithium manganese oxide are added.

—The requirement of the magnetic impurities content is added.

—The content and the requirement of the main elements in the product are modified.

—The content and the requirement of the impurity elements in the product are modified.

—The requirement of the mole ratio of Li to Mn in the composition of the product is deleted.

—The cyclic test condition at 55℃ is added.

—Annex A to Annex D in the standard are deleted.

This standard was prepared by SAC/TC 243 National Nonferrous Metals Standardization Technical Committee.

This standard is drafted by the following organizations:

This standard is mainly drafted by the following people: \*\*，\*\*，\*\*

The previous editions of this standard are as follows:

—YS/T 677-2008.

**Lithium manganese oxide**

**1 Scope**

This standard specifies the terms, definitions, requirements, test methods, inspection rules, marking, packaging, transportation, storage, certificate of quality and contract/order content of lithium manganese oxide.

This standard is applicable to the cathode active material spinel lithium manganese oxide, used in lithium-ion batteries.

**2** **Normative** **References**

The following referenced documents are indispensable 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/T 1717, *Determination of pH value of an aqueous suspension of pigments*

GB/T 5162, *Metallic powders—Determination of tap density*

GB/T 5314, *Powders for powder metallurgical purposes—Sampling*

GB/T 6283, *Chemical products—Determination of water Karl Fischer method (general method).*

GB/T 13390, *Metallic powder－Determination of the specific surface area－Method of nitrogen adsorption*

GB/T 19077, *Particle size analysis—Laser diffraction methods*

*GB/T 20252，Lithium cobalt oxide*

GB/T 23365, *Electrochemical performance test of lithium cobalt oxide—Test method for specific capacity and charge-discharge efficiency of the first cycle*

GB/T 23366*, Electrochemical performance test of lithium cobalt oxide—Test method for discharge plateau capacity ratio and cycle life*

GB/T 24533-2009, *Graphite negative electrode materials for lithium ion battery*

JCPDS[[1]](#footnote-2))(35-0782), *X - ray powder diffraction standard pattern of spinel lithium manganese oxide*

**3 Terms and definitions**

For the purposes of this document, the terms and definitions given in GB/T 20252-2014 apply.

**4 Requirements**

**4.1 Product classification**

Lithium manganese oxide is divided into 2 types according to their properties and applications: the high capacity lithium manganese oxide, and the high power lithium manganese oxide.

**4.2 Chemical composition**

The chemical composition of the product shall meet the requirements in Table 1.

**Table 1** Chemical composition

|  |  |  |  |
| --- | --- | --- | --- |
| Chemical composition | | Content (mass ratio)/ % | |
| High capacity lithium manganese oxide | \*High power lithium manganese oxide |
| Main elements | Mn | 58.0±2.0 | 57.5±2.0 |
| Li | 4.2±0.4 | 4.1±0.4 |
| Impurities | K | ≤0.05 | ≤0.01 |
| Na | ≤0.3 | ≤0.1 |
| Ca | ≤0.03 | ≤0.03 |
| Fe | ≤0.01 | ≤0.01 |
| Cu | ≤0.005 | ≤0.005 |
| S | — | ≤0.167 |

**4.3 Moisture**

The moisture content of the high capacity lithium manganese oxide shall not be more than 0.07%. The moisture content of the high power lithium manganese oxide shall not be more than 0.05%.

**4.4 pH Value**

The pH value of the product shall be in the range of 7.0 to 11.0.

**4.5 Magnetic Impurities**

The magnetic impurities content of the product for which the buyer side requires, shall be determined by both the supplier and the buyer and specified in the contract.

\*The magnetic impurities content of the product shall be determined by both the supplier and the buyer and specified in the contract when the buyer side requires.

**4.6** **Particle Size Distribution**

The characteristic value of the particle size distribution shall meet the requirements in Table 2.

**Table 2** Particle size distribution

|  |  |  |  |
| --- | --- | --- | --- |
| Product classification | | High capacity lithium manganese oxide | High power lithium manganese oxide |
| Particle size distribution | D50/μm | 6.0~14.0 | 10.0~14.0 |
| Dmax/μm | ≤100.0 | ≤60.0 |

**4.7** **Tap density**

The tap density of the product shall meet the requirements in Table 3.

**Table 3** Tap density

|  |  |  |
| --- | --- | --- |
| Product classification | High capacity lithium manganese oxide | High power lithium manganese oxide |
| Tap density( g/cm3) | ≥1.1 | ≥1.8 |

**4.8 Specific surface area**

The specific surface area of the product shall meet the requirements in Table 4.

**Table 4** Specific surface area

|  |  |  |
| --- | --- | --- |
| Product classification | High capacity lithium manganese oxide | High power lithium manganese oxide |
| Specific surface area/( m2/g) | 0.4~1.2 | 0.2~0.7 |

**4.9 Appearance**

The product shall be uniform black powder and agglomeration is not allowed.

**4.10 Crystal structure**

The crystal structure of the product shall be in accordance with the JCPDS standard (35-0782), and the impurity phase shall not be detected compared with the standard pattern.

**4.11 Specific capacity**

The initial specific discharge capacity of the product under specified conditions shall meet the requirements in Table 5.

**Table 5** Initialspecific discharge capacity

|  |  |  |
| --- | --- | --- |
| Product classification | High capacity lithium manganese oxide | High power lithium manganese oxide |
| Initial specific discharge capacity/( mAh/g) | ≥110 | ≥100 |

**4.12 Efficiency**

The first efficiency of the product under specified conditions shall not be less than 90%.

**4.13 Plateau capacity ratio**

The plateau capacity ratio of the product under specified conditions shall not be less than 90% after 10 cycles, and 85% after 100 cycles.

**4.14 Cycle Life**

The cycle life of the product shall meet the requirements in Table 6, when the discharge capacity reaches 80% of the discharge capacity of the first cycle.

**Table 6** Cycle life

|  |  |  |
| --- | --- | --- |
| Product classification | High capacity lithium manganese oxide | High power lithium manganese oxide |
| Cycle life, numbers | ≥500 | ≥1000 |

**4.15 High temperature cycle life**

The cycle life of the high power lithium manganese oxide tested at 55℃ shall not be less than 300 cycles, when the discharge capacity reaches 80% of the discharge capacity of the first cycle.

**5 Test methods**

**5.1 Chemical composition**

The test on the chemical composition is carried out according to the methods agreed by both suppliers and buyers.

**5.2 Moisture**

The determination of the moisture content is carried out according to GB/T 6283.

**5.3 pH value**

The determination of pH value of the product is carried out according to GB/T 1717.

**5.4 Magnetic impurities**

The determination of the magnetic impurities content of the product is carried out according to the provisions in Annex K of GB/T 24533-2009.

**5.5 Particle size distribution**

The determination of the size distribution of the product is carried out according to GB/T 19077.

**5.6 Tap density**

The determination of the tap density of the product is carried out according to GB/T 5162.

**5.7 Specific surface area**

The measurement of the specific surface area of the product is carried out according to GB/T 13390.

**5.8 Appearance**

Visual inspection of the product appearance.

\*The determination of the product appearance is carried out by visual inspection.

\*The product appearance is detected by visual inspection.

**5.9 Crystal structure**

The crystal structure of the product is detected by X-ray diffractometer.

**5.10 Specific capacity**

The determination of the first specific discharge capacity of the product is carried out according to GB/T 23365. The charging and discharging voltage range is 3.00 V-4.30 V with the other conditions remain unchanged.

**5.11 Efficiency**

The determination of the first efficiency of the product is carried out according to GB/T 23365. The charging and discharging voltage range is 3.00 V-4.30 V with the other conditions unchanged.

\*The determination of the first efficiency of the product is carried out according to GB/T 23365. The charging and discharging voltage range are modified to 3.00 V-4.30 V with the other conditions unchanged.

**5.12 Plateau capacity ratio**

The measurement of the capacity ratio of the product platform is carried out according to the provisions of GB/T 23366. The charging and discharging voltage range is 3.00 V-4.20 V with the other conditions remain unchanged.

**5.13 Cycle life**

The determination of the cycle life of the product is carried out according to GB/T 23366. The charging and discharging voltage range is 3.00 V-4.20 V with the other conditions remain unchanged.

**5.14 High temperature cycle life**

The determination of the high temperature cycle life of the product is carried out according to GB/T 23366. The test temperature is at 55℃ and the charging and discharging voltage range is 3.00 V-4.20V with the other conditions remain unchanged.

**6 Inspection rules**

**\*6 Inspection provisions**

**6.1 Inspection and acceptance**

**6.1.1** The supplier shall check the product and fill in the quality certificate to insure that the quality of the product is in accordance with this standard and the contract (or order).

**\*6.1.1** The certificate of quality of the product shall be checked by the supplier to insure that the quality of the product is in accordance with this standard and the contract (or order).

**6.1.2** The buyer shall check the received product according to this standard, and negotiate with the supplier within 3 months after receiving the product, in the case that the check result doesn’t accord to this standard or the contract (or order). If any arbitration is needed, samples shall be obtained from both sides.

**\*6.1.2** The received product shall be checked by the buyer according to this standard, and negotiate with the supplier within 3 months after receiving the product, in the case that the check result doesn’t accord to this standard or the contract (or order). If any arbitration is needed, sampling shall be conducted by both sides.

**\*6.1.2** The received product shall be checked by the buyer according to this standard. When the check result doesn’t accord to this standard or the contract (or order), the supplier shall negotiate within 3 months after receiving the product. If any arbitration is needed, sampling shall be conducted by both sides.

**\*6.1.2** The received product shall be checked by the buyer according to this standard, and the supplier shall be notified within 3 months after receiving the product, in the case that the check result doesn’t accord to this standard or the contract (or order). The discordance shall be resolved through consultation. If any arbitration is needed, sampling shall be conducted by both sides.

**6.2 Batches**

The product shall be submitted for the acceptance in batches and each batch shall be composed of the same mixture, with weight of not more than 5 000 kg. If the buyer has special requirement, it shall be settled based on the negotiation between both sides.

\*The product shall be submitted for the acceptance in batches and each batch shall be composed of the mixture from the same type, with weight of not more than 5 000 kg. If the buyer has special requirement, it shall be settled based on the negotiation between both sides.

**6.3 Product inspection**

**6.3.1 Inspection classifications**

The inspection in this standard is classified into: inspection by batch, and periodic inspection.

\*The inspection in this standard is classified into: inspection by batch, periodic inspection.

**6.3.2 Inspection by batch**

Each batch of the product shall be tested.

**6.3.3 Periodic Inspection**

The items of the periodic inspection are determined according to their difficulty degree and stability. In the case of the normal production, the inspection shall be carried out once a month. The periodic inspection shall be carried out when the raw materials or production processes significantly change or the production is resumed after long term shut down. It shall be indicated in the contract if there are special requirements for the periodic inspection from the buyer.

\*The items of the periodic inspection are determined according to their difficulty degree and stability. In the case of the normal production, the inspection shall be carried out once a month. The periodic inspection shall be carried out when the raw materials or production processes are significantly changed or the production is resumed after long term shut down. It shall be indicated in the contract if there are special requirements for the periodic inspection from the buyer.

**6.3.4 Inspection items and sampling**

Product inspection items and sampling are shown in Table 7**.**

**Table 7** Inspection items and sampling

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Test items | Sampling method | Sample number | Required  number | Test method number | Test classification |
| Chemical composition | Sampling according to the method specified in GB/T 5314 | One portion from each batch | 4.2 | 5.1 | Inspection by batch |
| Moisture content | One portion from each batch | 4.3 | 5.2 | Inspection by batch |
| pH Value | One portion from each batch | 4.4 | 5.3 | Inspection by batch |
| Magnetic impurities content | One portion from each batch | 4.5 | 5.4 | Inspection by batch |
| Particle size distribution | Five portion from each batch | 4.6 | 5.5 | Inspection by batch |
| Tap density | Two portion from each batch | 4.7 | 5.6 | Inspection by batch |
| Specific surface area | One portion from each batch | 4.8 | 5.7 | Inspection by batch |
| Appearance | — | Each bag | 4.9 | 5.8 | Inspection by batch |
| Crystal structure | Sampling according to the method specified in GB/T 5314 | One portion from each batch | 4.10 | 5.9 | Periodic inspection |
| Specific capacity | One portion from each batch | 4.11 | 5.10 | Periodic inspection |
| Efficiency | One portion from each batch | 4.12 | 5.11 | Periodic inspection |
| Plateau capacity ratio | One portion from each batch | 4.13 | 5.12 | Periodic inspection |
| Cycle life | One portion from each batch | 4.14 | 5.13 | Periodic inspection |
| High temperature cycle life | One portion from each batch | 4.15 | 5.14 | Periodic inspection |

**6.4 Judgment on inspection results**

**6.4.1** One batch of the product is judged as unqualified, if any of the chemical composition, the moisture content, the pH value, the magnetic impurities content, the particle size distribution, the tap density, the specific surface area and the crystal structure is tested to be unqualified.

**6.4.2** The disqualification of the product appearance will disqualify the whole barrel (bag) of the product.

**\*6.4.2** The whole barrel (bag) of the product is judged as unqualified if the product appearance is tested to be unqualified.

**\*6.4.2** The whole barrel (bag) of the product is unqualified in case product appearance is tested to be unqualified.

**6.4.3** Make six coin cells according to the method in GB / T 23365. Three batteries are picked randomly to do the specific capacity and the first cycle coulombic efficiency test. If two batteries can’t reach the requirement of this standard, this batch of the product is judged as unqualified. However, repeated test is allowed for the other three batteries, and if two of them can reach the requirement of this standard, this batch of the product is judged as qualified.

**6.4.4** Make six test batteries according to the method in GB / T 23366. Three batteries are picked randomly to do the plateau capacity ratio, the cycle life and the high temperature cycle life test. If two batteries can’t reach the requirement of this standard, this batch of the product is judged as unqualified. However, repeated test is allowed for the other three batteries, and if two of them can reach the requirement of this standard, this batch of the product is judged as qualified.

**7 Marks, packaging, transportation, storage and quality certificate**

**7.1 Marks**

On the outer package, the followings shall be marked:

1. product name;
2. supplier name, address;
3. batch number, type;
4. net weight;
5. test date;
6. damp proof mark;
7. this standard number, YS/T 677-2016.

**7.2 Packaging**

Products are packed using aluminum plastic bag, and put into the barrel after thermoplastic sealing. A barrel of the product has a net weight of 25.00 kg. The packaging can also be settled by negotiation to satisfy the buyer’s requirement.

**7.3 Transportation and storage**

**7.3.1** Damage to the packaging shall be avoided during transportation process.

**7.3.2** Products shall be avoided of damp during storage. Guarantee period is one year from the date of the production.

**\*7.3.2** Products shall be avoided of damp during storage. Guarantee period is one year since the date of the production.

**7.4 Quality certificate**

Quality certificate shall be attached to each batch of the products, which indicates:

1. supplier name, address, telephone and fax;
2. product name;
3. batch number, type;
4. net weight and number;
5. partition test result; Seal from the technical supervision department; (\*various analysis and test result; seal of the quality assurance department;)
6. this standard number, YS/T 677-2016;
7. production date.

**8 Contract (or order) contents**

The contract (or order) for the product listed in this standard shall include the following contents:

1. product name;
2. type and quantity;
3. tish standard number, YS/T 677-2016;
4. others.

1. )  Joint Committee on Powder Diffraction Standards [↑](#footnote-ref-2)