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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 43965-2024

Electronic grade ethyl orthosilicate

电子级正硅酸乙酯

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3 Terms and definitions

3.1 The document states that it has no terms and definitions to define.

4 Technical requirements

4.1 Electronic grade ethyl orthosilicate is to meet Table 1, which sets the indicators for three grades: for photovoltaic use, marked 6N; for flat panel display use, marked 8N; and for integrated circuit use, marked 9N. The table starts with the purity of the ethyl orthosilicate itself, expressed as a percentage, at not less than 99.90 for the photovoltaic grade, not less than 99.95 for the flat panel display grade and not less than 99.99 for the integrated circuit grade.

4.1 (continued) The largest block of the table is the cation content, in micrograms per kilogram, listed element by element for forty-seven elements. In the order printed these are boron, aluminium, antimony, arsenic, barium, beryllium, bismuth, cadmium, calcium,

cerium, chromium, cobalt, copper, gallium, germanium, gold, hafnium, indium, iridium, iron, lead, lithium, magnesium, manganese, mercury, molybdenum, nickel, niobium, palladium, platinum, potassium, rhenium, rhodium, rubidium, silver, sodium, strontium, tantalum, thallium, thorium, tin, titanium, tungsten, uranium, vanadium, zinc and zirconium. In the extraction the element names and the three columns of limits run as separate blocks and the first of those blocks holds one value fewer than there are elements, so the individual element limits cannot be matched to their rows with confidence and are not reproduced here. The two summary rows that close the block can be read: the total cation content is not more than 1 000 micrograms per kilogram for the photovoltaic grade, not more than 10 for the flat panel display grade and not more than 1 for the integrated circuit grade; and the purity expressed on a cation basis is not less than 99.9999 % for the photovoltaic grade, not less than 99.999999 % for the flat panel display grade and not less than 99.9999999 % for the integrated circuit grade.

4.1 (continued) The rest of the table is readable. The chloride content, in micrograms per kilogram, is not more than 500 for the photovoltaic grade, not more than 500 for the flat panel display grade and not more than 50 for the integrated circuit grade. The colour, in Hazen units, is 5 to 10 for all three grades. The water content, in milligrams per kilogram, is not more than 50, not more than 50 and not more than 5 respectively. The particle count, in pieces per millilitre, is given for four size bands. For particles from 0.2 micrometres up to 0.3 micrometres the limits are not more than 100, not more than 50 and not more than 20; from 0.3 micrometres up to 0.5 micrometres, not more than 50, not more than 30 and not more than 10; from 0.5 micrometres up to 1.0 micrometres, not more than 30, not more than 20 and not more than 5; and for particles of 1.0 micrometres and above, not more than 20, not more than 5 and not more than 2.

5 Test methods

5.1 Test environment. Cation and particle determinations are to be carried out in a cleanroom of at least ISO class 3 as defined in GB/T 25915.1. Solution preparation, sample handling, content testing and chloride testing for the cation and particle determinations should be carried out in a cleanroom environment of at least ISO class 2 as defined in GB/T 25915.1. Water testing is carried out in a nitrogen-purged glove box, the pressure inside the box being set at 0.42 mbar, the oxygen content not more than 2 parts per million by volume and the water content not more than 2 parts per million by volume.

5.2 Purity of ethyl orthosilicate. The reagents are high purity nitrogen and high purity helium, both of purity greater than 99.999 %. The column is a fused silica capillary 30 m long with an internal diameter of 530 micrometres whose inner wall has been film treated, or another equivalent capillary column. Detection is by gas chromatography with a flame ionization detector and electronic pneumatic control. The chromatographic conditions preferred are set in Table 2: injector temperature 200 °C, initial column temperature 80 °C,

detector temperature 250 °C, injection volume 0.2 microlitres, carrier gas flow 40 millilitres per minute, split flow 35 millilitres per minute, hydrogen purity not less than 5N and nitrogen carrier gas purity not less than 5N. Impurities are measured with a temperature programme, the oven programme following Table 3, which gives two steps: step one at a rate of 0 degrees Celsius per minute to a value of 80 °C held for 3 min, and step two at a rate of 10 degrees Celsius per minute to a value of 180 °C held for 8 min. The test runs in four stages: the operating parameters of the chromatograph are set until the baseline is stable and normal; a 5 microlitre syringe is flushed several times with the sample to be tested and 0.2 microlitres injected each time, the resulting chromatogram of the ethyl orthosilicate sample being as shown in Annex A while the computer collects the peak area or peak height response of every organic component; after the run the purity is obtained by area normalization; and the sample is tested three times, the mean of the three results being taken as the final result.

5.3 Cation content. The reagents and materials preferred are ultrapure water meeting grade EW-I of GB/T 11446.1; a sampling bottle of perfluoroalkoxy resin; ultrapure nitric acid with each single metal ion below 10 nanograms per litre; ultrapure hydrofluoric acid with each single metal ion below 10 nanograms per litre; a nitric acid solution made by weighing 10 millilitres of 69 % electronic grade nitric acid into a 500 millilitre sampling bottle, adding 100 millilitres of ultrapure water and mixing; and a mixed nitric and hydrofluoric acid made by weighing 10 millilitres of 49 % electronic grade hydrofluoric acid into that nitric acid solution and adding a further 100 millilitres of ultrapure water and mixing. Before the metal ion and boron content of the product is measured the sample is pretreated, the whole pretreatment being carried out under class 100 ultraclean conditions. The inductively coupled plasma mass spectrometer is tuned before every run and warmed up for 0.5 h, after which a blank is measured and injection begins only once the baseline noise or the blank parameters are stable. Pretreatment has two steps: 5 g of the ethyl orthosilicate sample is weighed into a 100 millilitre open perfluoroalkoxy resin vessel and heated to 150 °C so that the sample evaporates completely; then 10 millilitres of the prepared mixed acid is added to the evaporated vessel and shaken well so that the metal ions in the sample dissolve fully in the mixed acid. The determination follows SJ/T 11637, with five mixed element standard solutions of proportionate mass concentration and a working curve whose linear coefficient is not less than 0.995. Another equivalent method may be used; where the result is disputed, the method of SJ/T 11637 is the arbitration method.

5.4 Chloride content. The determination runs in six steps: the ion chromatograph is brought to suitable working conditions; the ethyl orthosilicate is mixed with isopropanol in the ratio one to one; the mixture passes in turn through the sample loop and the concentrator column so as to be enriched; the concentrator column is backflushed with eluent into the ion chromatographic system for measurement; a Gaussian chromatographic peak is formed in the conductivity detector; and, the peak area or peak height being proportional to the concentration of the component, the chloride content is calculated from the peak area.

5.5 The colour is determined as GB/T 3143 requires.

5.6 The water content is determined as GB/T 26793 requires. Another equivalent method may be used; where the result is disputed, the method of GB/T 26793 is the arbitration method.

5.7 The particle count is determined as SJ/T 11638 requires.

6 Evaluation of the measurement results

6.1 The method detection limit for the cation content of ethyl orthosilicate is to meet GB/T 37837.

6.2 Precision is measured independently on the sample, with not fewer than seven determinations, and is expressed as the relative standard deviation. The calculation of the relative standard deviation is to meet GB/T 37837 and the relative standard deviation of the test result is to be not more than 5 %.

6.3 Spike recovery is obtained by taking two portions of the same sample, spiking one of them with standard solution and injecting both under the same conditions; the difference between the concentration found with the spike and the concentration found without it, divided by the theoretical value of the spike, is the spike recovery of the sample. It is to be kept within 80 % to 120 %. Spike recovery is prescribed only for the determination of the cation content and the chloride content.

7 Inspection rules

7.1 An inspection batch is made up of all the product filled from the same finished product tank, produced continuously and steadily on the same production line by the same process. Every indicator of Table 1 is to be tested on each inspection batch.

7.2 The first and the last item of each inspection batch are to be tested against the indicators, while the sampling of the items in between follows the sampling plan of GB/T 2828.1 with the acceptance quality limit calculated at 1.0, the sample size meeting Table 4. That table pairs the batch size, that is the total number of items, with the sample size: 2 to 8 items, 2; 9 to 15, 3; 16 to 25, 5; 26 to 50, 8; 51 to 90, 13; 91 to 150, 20; 151 to 280, 32; and 281 to 500, 50. The series is carried on under the heading Table 5, again titled sampling quantity, with 501 to 1 200 items, 80; 1 201 to 3 200, 125; 3 201 to 10 000, 200; 10 001 to 35 000, 315; and 35 000 to 150 000, 500.

7.3 Where any one item of the test result on a sample fails to meet Table 1, the batch is to be sampled and tested again; if after three samplings any one indicator still fails, every filled cylinder is to be sampled and tested, and if any one indicator still fails the batch is non-conforming.

7.4 Sampling safety is to meet the provisions of GB/T 3723 on hazardous substances and