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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 14602-2014

Replacing GB/T 14602-1993

Gases for electronic industry - Hydrogen chloride

电子工业用气体 氯化氢

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1 Scope

The standard sets the technical requirements, the test methods and the rules for marking, packaging, storage and transport and for safety of hydrogen chloride.

It applies to hydrogen chloride products obtained by purification from industrial hydrogen chloride as raw material. The product is used mainly for vapour-phase polishing, epitaxy and etching processes in the microelectronics industry, and can also be used in cemented carbides, glass surface treatment, pharmaceutical intermediates, fine chemical manufacture, scientific research and similar fields.

The molecular formula is given as HCl and the relative molecular mass as 36.458, calculated from the 2009 international relative atomic masses.

2 Normative references

The listed documents are indispensable for the application of the document. For dated references only the edition cited applies; for undated references the latest edition, including

all amendments, applies.

The references given are: GB 190 Packaging marks for dangerous goods; GB/T 3723 General rules for the safe sampling of chemical products for industrial use; GB 5099 Steel seamless gas cylinders; GB/T 5832.3 Determination of trace moisture in gases - Part 3: Cavity ring-down spectroscopy; GB 7144 Colour marking for gas cylinders; GB 14193 Regulations for the filling of liquefied gas cylinders; GB 15258 General rules for the preparation of chemical safety labels; GB 16804 Gas cylinder warning labels; GB/T 26571 Specification for the storage period of special gases; GB/T 28726-2012 Gas analysis - Helium ionization gas chromatography; together with the Regulations on the safety supervision of gas cylinders (2000 edition), the Regulations on the safe management of hazardous chemicals (2002 edition) and the Regulations on the safety supervision of special equipment (2009 edition).

3 Technical requirements

The technical requirements for hydrogen chloride are to satisfy Table 1, headed technical indices.

Table 1 sets one column of items against two indicator columns. The two indicator columns carry no heading of their own in either the first part of the table or its continuation, so the grades they correspond to cannot be read from the pages available; the values below are therefore reported as the first and the second indicator column, in that printed order.

The items and their limits are: hydrogen chloride purity, as a volume fraction in units of ten to the minus second, not less than 99.999 in the first column and 99.999 5 in the second; oxygen plus argon (O2 + Ar) content, as a volume fraction in units of ten to the minus sixth, less than 1.0 and 0.5; nitrogen (N2) content, less than 2.0 and 2.0; carbon dioxide (CO2) content, less than 2.0 and 1.0; carbon monoxide (CO) content, less than 1.0 and 0.5; hydrocarbons (CH4 plus C2H2) content, less than 1.0 and 0.5; and moisture (H2O) content, less than 1.0 and 0.5, all on the same volume fraction basis.

The table further sets total impurity content, as a volume fraction in units of ten to the minus sixth, with no limit stated in the first column and not more than 5 in the second; iron (Fe) content not more than 0.5 mg/L and 0.1 mg/L; other metallic elements (manganese, cobalt, zinc, copper, chromium, nickel) content not more than 0.1 mg/L in both columns; and particles, to be agreed between supplier and purchaser in both columns.

4.1 Sampling and assessment

Hydrogen chloride products are to be inspected and accepted one by one. Where any single index of the inspection result fails to meet the technical requirements of the standard, the product is judged non-conforming.

The safety of hydrogen chloride sampling is to comply with the relevant provisions of GB/T

3723.

4.2 Purity of hydrogen chloride

The total impurity content is obtained from an equation that sums six measured impurity contents, whose symbols are defined as the oxygen plus argon content, the nitrogen content, the carbon dioxide content, the carbon monoxide content, the hydrocarbon content and the moisture content, each expressed as a volume fraction in units of ten to the minus sixth.

The purity of hydrogen chloride is then obtained from a second equation that subtracts the converted total impurity content from 100. The symbol is defined as the hydrogen chloride purity expressed as a volume fraction in units of ten to the minus second.

4.3 Determination of the oxygen plus argon, nitrogen, carbon monoxide, carbon dioxide and hydrocarbon contents

The oxygen plus argon, nitrogen, carbon monoxide, carbon dioxide and hydrocarbon (CH4, C2H2) contents in hydrogen chloride are determined by the pre-cut sample introduction method specified in GB/T 28726-2012.

The pre-cut column is about 2.0 m long with an internal diameter of about 2 mm, is made of Monel 400 alloy and is packed with Porapak Q of 0.18 mm to 0.25 mm particle size, or an equivalent chromatographic column.

Chromatographic column I is about 2 m long with an internal diameter of about 2 mm, is made of Monel 400 alloy and is packed with 5A molecular sieve of 0.18 mm to 0.25 mm particle size, or an equivalent column; it is used to analyse the oxygen plus argon, nitrogen and carbon monoxide contents.

Chromatographic column II is about 2 m long with an internal diameter of about 2 mm, is made of Monel 400 alloy and is packed with Hayesep Q of 0.18 mm to 0.25 mm particle size, or an equivalent column; it is used to analyse the carbon dioxide and hydrocarbon (CH4, C2H2) contents.

The gas standard sample has component contents with volume fractions from 1 to 5 in units of ten to the minus sixth, with nitrogen as the balance gas.

Other equivalent methods are permitted for determining these contents in hydrogen chloride; where the results are disputed, the method specified in the standard is the arbitration method.

4.4 Determination of the moisture content

The determination is carried out according to GB/T 5832.3. Other equivalent methods may

be used to determine the water content in hydrogen chloride; where the results are disputed, the method specified in GB/T 5832.3 is the arbitration method.

4.5 Determination of the iron and other metallic element contents

Inductively coupled plasma mass spectrometry is used to determine the contents of manganese, cobalt, zinc, copper, chromium, nickel and similar metallic elements in hydrogen chloride. The detection limit is not greater than 0.01 mg/L; the balance has a sensitivity of 0.01 g; an absorption system is required; and the metallic element standard solutions are to have element contents close to the corresponding contents in the sample being tested.

Determination conditions: the quantity of hydrogen chloride absorbed is not more than 5 g, the deionized water is not more than 50 mL, and the high-purity nitrogen flow is 20 mL/min.

Sample absorption: a diagram of the absorption device is given in Annex A. Exactly 50.00 mL of deionized water is placed in the absorption system and the initial mass of the absorption system, comprising the gas washing bottle, the absorption bottle and the buffer bottle, is weighed accurately. The hydrogen chloride sample is passed slowly through the absorption system and absorption is stopped once more than 5 g of hydrogen chloride has been absorbed. The high-purity nitrogen cylinder valve is opened to purge the line for 1 min and gas flow is then stopped. The final mass of the absorption system is weighed accurately, and the solution in the absorption system is the sample solution for the determination of metallic elements.

Determination procedure: the instrument is started according to the instructions for the inductively coupled plasma mass spectrometer and its components are adjusted to the determination conditions; determination may begin once the instrument is stable. For the standard samples, the metallic element standard solutions are injected and the absorbance signal of the absorption peak of each metallic element is recorded; each standard solution is injected at least twice until the relative deviation between two parallel determinations is not greater than 5 %, and the mean is taken. For the samples, the absorbed sample solution is injected under the same determination conditions as the standard solutions, the absorbance signals of the absorption peaks of the different metallic elements are recorded, injection is repeated at least twice until the relative deviation between two parallel determinations is not greater than 5 %, and the mean is taken.

Treatment of results: the content of a metallic element in hydrogen chloride is obtained from an equation whose symbols denote the content of the metallic element in hydrogen chloride in milligrams per litre, the signal intensity of the sample, the signal intensity of the standard, the content of the standard in milligrams per litre, and the mass in grams of the hydrogen chloride that has passed through the gas absorption bottle. That mass is in turn obtained as the difference between the final mass of the absorption system and its initial mass, both in grams.