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PEOPLE'S REPUBLIC OF CHINA**

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Electronic gas - Tetrafluoromethane

电子气体 四氟甲烷

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1 Scope

The document fixes the technical requirements, the inspection rules and the requirements for marking, packaging, transport, storage and safety information of electronic grade tetrafluoromethane, and describes the associated test methods.

It applies to tetrafluoromethane prepared by purification of industrial grade tetrafluoromethane. The terms and definitions of GB 30000.25 apply; the document defines no terms of its own.

4 Technical requirements

The whole of the product specification rests on one table of limits, with no division of the product into grades or classes. Table 1 has two columns, item and requirement, and eleven rows.

Purity of tetrafluoromethane (CF₄), as volume fraction in units of 10 to the minus 2, is not less than 99.999. The impurity limits, as volume fraction in units of 10 to the minus 6, are: oxygen plus argon less than 1; nitrogen less than 4; carbon monoxide less than 0.1; carbon dioxide less than 1; sulfur hexafluoride less than 5; other halogenated compounds less than 2; moisture less than 2.

Acidity, calculated as HF and given as mass fraction in units of 10 to the minus 6, is less than 0.1. Total impurity content, as volume fraction in units of 10 to the minus 6, is not greater than 10. Particle content is left to agreement between supplier and purchaser.

A footnote to the table defines other halogenated compounds as hexafluoroethane, octafluoropropane, chlorotrifluoromethane and trifluoromethane.

5 Test methods

A warning opens the clause: some of the test procedures described can lead to hazardous situations, and the user is to take appropriate safety and health protection measures.

Unless otherwise stated, reagents are of analytical grade and water is grade three water as defined in GB/T 6682; standard solutions, preparations and products are prepared in accordance with GB/T 601 and GB/T 603.

The gaseous impurities are determined by the cut injection method of GB/T 28726, that is helium ionisation gas chromatography. Other equivalent methods are permitted, and in case of dispute the method of this document is the arbitration method. Two pre-separation columns and three analytical columns are specified by length, internal diameter, packing and particle size range: a modified carbon molecular sieve column of about 2 m and a porous polymer column of about 5 m for pre-separation; a 5A molecular sieve column of about 3 m for oxygen plus argon, nitrogen and carbon monoxide; a porous polymer column of about 7 m for carbon dioxide, nitrous oxide and sulfur hexafluoride; and a capillary column of about 50 m internal diameter 0.32 mm coated with bonded silica gel for the four halogenated compounds. The component content of the standard sample lies between 1 and 5 in units of 10 to the minus 6 by volume, with helium as the balance gas.

Moisture is determined in accordance with GB/T 5832.3, that is by cavity ring-down spectroscopy; equivalent methods are permitted and GB/T 5832.3 is the arbitration method.

Acidity is measured by vaporising the test sample and bubbling it through porous gas washing bottles containing water so that the acidic species are absorbed, then titrating with

sodium hydroxide standard titration solution against bromocresol green. The reagents are sodium hydroxide standard titration solution at 0.01 mol/L and bromocresol green indicator solution at 5 g/L. The apparatus is a 316L stainless steel sampling cylinder of 500 mL or 1000 mL, a 316L stainless steel two-way control valve, porous gas washing bottles of 250 mL, a micro burette of 2 mL or 5 mL with a smallest scale division of 0.01 mL, and an electronic balance of maximum weighing not less than 3000 g and accuracy 0.1 g.

In the procedure, four washing bottles each receive 100 mL of water, the cylinder and valve are weighed, about 100 g of sample is bubbled through the four bottles over 15 min to 20 min and the cylinder and valve are weighed again. The solutions of the third and fourth bottles are checked with indicator against each other before the first and second are combined and titrated to a blue end point; the third and fourth then serve as the blank. The symbols of the calculation are the concentration of the sodium hydroxide solution in mol/L, the volumes consumed by the test portion and by the blank in mL, the molar mass of hydrogen fluoride taken as 20.01 g/mol, and the initial and final masses in g. The result is the arithmetic mean of parallel determinations, whose relative deviation is not to exceed 40 %.

Total impurity content is the sum of the seven measured impurity contents. Purity is obtained from the total impurity content by the equation given in the document.

6 Inspection rules

A batch is the cylinder-packed tetrafluoromethane produced continuously and stably on one production line, or the product of one storage tank or one tank car. Rounding follows the comparison method of GB/T 8170.

For cylinder product, the oxygen plus argon, nitrogen and moisture contents are checked cylinder by cylinder, and a result outside Table 1 rejects the product. The remaining impurities of Table 1 are checked on a random sample whose size comes from Table 2, which has two columns and three rows: from 1 to 10 cylinders in the batch, at least 1 cylinder is sampled; from 11 to 20 cylinders, at least 2; above 20 cylinders, at least 5.

If the sample fails, a fresh random sample of double the size is drawn from the same batch. If it conforms, the batch passes apart from the failing cylinder; if it fails again, the batch is rejected. Storage tank and tank car product is inspected and accepted unit by unit.

Sampling safety follows GB/T 3723. Measures are to be taken to treat the tail gas of the determination so that tetrafluoromethane is not released to the environment.

7 Marking, packaging, transport, storage and safety information

The certificate of conformity issued with the product carries at least the product name, the manufacturer name and the hazardous chemical production licence number; the production date or batch number and the expiry date; the filled mass in kg; and the number of this

document together with the purity of the tetrafluoromethane.

The packaging container is marked with the words for electronic grade tetrafluoromethane. Packaging marks follow GB 190, colour marks GB/T 7144, and labels GB 15258 and GB/T 16804.

Cylinders follow GB/T 5099 (all parts) and GB/T 33145. Filling, transport and storage follow GB/T 14193, TSG R0005, TSG 07 and TSG 23, with the safety management requirements of the national regulations on hazardous chemicals and on special equipment. The maximum filling quantity is the product of the filling factor in kg/L and the marked internal volume of the cylinder in L; a note gives the filling factor as 0.7 kg/L for a cylinder of 20.0 MPa nominal pressure.

Cylinders with a treated internal surface are preferred, and the valve outlet connection is preferably DISS 716 or CGA 580. Leakage and contamination of the valve outlet are to be prevented, and the product is stored in a cool, dry, ventilated warehouse away from heat sources and out of direct sunlight.

A Annex A (normative) Safety information

Basic information: chemical formula CF_4 , English name tetrafluoromethane; relative molecular mass 88.005 calculated on the 2018 international relative atomic masses; CAS number 75-73-0; UN dangerous goods number 1982.

Physical properties: boiling point $-127.8\text{ }^{\circ}\text{C}$, melting point $-183.6\text{ }^{\circ}\text{C}$, critical temperature $-45.65\text{ }^{\circ}\text{C}$, critical pressure 3740 kPa, critical density 0.6285 g/cm³, relative vapour density 3.04 with air taken as 1, and vapour density 3.688 kg/m³ at 15 °C and 1 atm.

Hazard statement: tetrafluoromethane is a colourless, odourless, high-pressure liquefied gas; rapid evaporation of the liquid can cause frostbite. Its hazard classification for specific target organ toxicity after single exposure is category 3, it has a narcotic effect at high concentration, and exposure can bring on headache, nausea and vomiting.

Handling: the production process is closed, the plant is fully ventilated, operators are specially trained and follow the operating procedures strictly, and heat sources and hot surfaces are kept away.

Emergency response: in a fire the gas supply is cut off, fire fighters wear self-contained breathing apparatus and work from upwind, and containers are moved from the fire area to open ground where possible; dry powder, carbon dioxide and water are the preferred extinguishing media. A person who has inhaled the gas is moved quickly to fresh air and the airway kept clear; oxygen is given for breathing difficulty, and cardiopulmonary resuscitation is started at once and medical help sought if breathing or heartbeat has stopped.

Leakage: the source is cut off where possible, a warning area is marked out according to