ICS 71.100.20
CCS G 86

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 43977-2024

Electronic gas - Octafluorocyclobutane

电子气体 八氟环丁烷

Issued on: April 25, 2024 **Implemented on: November 1, 2024**

Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC

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3 Terms and definitions

3 states that the document has no terms or definitions that need to be established.

4 Technical requirements

4 requires octafluorocyclobutane to meet Table 1. The table has two columns, item and requirement, and nine rows, and the pairing of the two columns is unambiguous in the source.

Purity of octafluorocyclobutane (C4F8) as a volume fraction, in units of 10 to the power minus 2: not less than 99.999.

Oxygen plus argon (O2 + Ar), nitrogen (N2), carbon monoxide (CO), carbon dioxide (CO2), hexafluoropropylene (C3F6) and water (H2O) contents are all fixed as volume fractions in units of 10 to the power minus 6, with limits below 1 for oxygen plus argon, below 2 for nitrogen, below 0.5 for carbon monoxide, below 0.5 for carbon dioxide, below 5 for hexafluoropropylene and below 2 for water.

Acidity, calculated as hydrogen fluoride, is fixed as a mass fraction in units of 10 to the power minus 6 and is to stay below 0.1. The total impurity content, again as a volume fraction in units of 10 to the power minus 6, is not to exceed 10.

5 Test methods

5 opens with a warning that some of the test procedures described can give rise to hazardous situations and that the user is to take suitable safety and health measures.

5.1 fixes the general provisions: unless otherwise stated the reagents are analytically pure and the water is grade three water as defined in GB/T 6682; standard solutions, preparations and products are prepared according to GB/T 601 and GB/T 603.

5.2 covers oxygen plus argon, nitrogen, carbon monoxide, carbon dioxide and hexafluoropropylene. They are determined by the cut (heart-cut) injection technique of GB/T 28726. Other equivalent methods are allowed, but where the result is disputed the method of this document is the referee method.

5.2.2 and 5.2.3 describe two pre-separation columns and two chromatographic columns, all of stainless steel tubing of about 3 mm internal diameter: pre-separation column I is about 2 m long and packed with a modified carbon molecular sieve (CST) of 0.15 mm to 0.18 mm particle size, pre-separation column II about 5 m long packed with a polymer (Porapak Q) of 0.18 mm to 0.25 mm; chromatographic column I is about 3 m long packed with 5A molecular sieve of 0.15 mm to 0.18 mm and serves for oxygen plus argon, nitrogen and carbon monoxide, chromatographic column II about 7 m long packed with the polymer of 0.18 mm to 0.25 mm and serves for carbon dioxide and hexafluoropropylene. Equivalent columns may replace any of them.

5.2.4 sets the component content of the standard sample at a volume fraction of 1 to 5 in units of 10 to the power minus 6, with helium as the balance gas.

5.3 refers the water content to GB/T 5832.3; other equivalent methods are allowed, with GB/T 5832.3 as the referee method in case of dispute.

5.4 gives the acidity method. The sample is vaporized and bubbled through porous gas washing bottles containing water, the acidic species are absorbed, and the solution is titrated with sodium hydroxide standard titration solution using bromocresol green as indicator. The reagents are sodium hydroxide standard titration solution at a concentration of 0.01 mol/L and bromocresol green indicator solution at 5 g/L.

5.4.3 lists the apparatus: 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 microburette of 2 mL or 5 mL with a smallest scale division of 0.01 mL, and an electronic balance with a maximum weighing capacity of not less than 3000 g and an accuracy of 0.1 g.

5.4.4 sets out the procedure: the apparatus is assembled as shown in Figure 1, 100 mL of water is placed in each of four gas washing bottles, the initial mass of the sampling cylinder with its control valve is weighed, about 100 g of sample is bubbled continuously through the four bottles over 15 min to 20 min, and the final mass is weighed. The solutions of the third and fourth bottles are transferred to two conical flasks and given one to two drops of indicator; the colour in the flask corresponding to the third bottle is not to be lighter than that corresponding to the fourth, otherwise the test is repeated. The solutions of the first and second bottles are then combined, given two to four drops of indicator and titrated to a blue

end point, while the combined solutions of the third and fourth bottles serve as the blank.

5.4.5 gives formula (1) for the acidity as a mass fraction. The printed equation is broken by the extraction and is not reproduced here; its legend reads: the acidity calculated as hydrogen fluoride, as a mass fraction in units of 10 to the power minus 6; the concentration of the sodium hydroxide standard titration solution in moles per litre; the volume of that solution consumed by the sample and by the blank, both in millilitres; the molar mass of hydrogen fluoride in grams per mole, taken as 20.01 g/mol; and the initial and final masses in grams.

5.4.6 takes the arithmetic mean of parallel determinations as the result and allows a relative deviation between two parallel determinations of not more than 40 %. 5.4.7 allows other equivalent methods, with the method of the document as referee.

5.5 obtains the total impurity content by formula (2) as the sum of the oxygen plus argon, nitrogen, carbon monoxide, carbon dioxide, hexafluoropropylene and water contents, each a volume fraction in units of 10 to the power minus 6. 5.6 obtains the purity by formula (3) from 100 and the total impurity content, the purity being a volume fraction in units of 10 to the power minus 2.

6 Inspection rules

6.1.1 defines the batch: the cylinder-filled product from one production line running continuously and steadily forms one batch, as does the product of one storage tank, one tube trailer or one tank truck.

6.1.2 requires numerical rounding to follow the rounded value comparison method of GB/T 8170.

6.1.3 covers cylinder product. Oxygen plus argon, nitrogen and water are checked on every cylinder, and a cylinder failing Table 1 on any of them is rejected. The remaining impurities are checked on cylinders drawn at random in the numbers given in Table 2; that table has two columns and three rows and reconciles: for a batch of 1 to 10 cylinders at least 1 is sampled, for 11 to 20 cylinders at least 2, and for more than 20 cylinders at least 5. If the result meets Table 1 the batch passes; if it does not, a doubled random sample is drawn from the same batch, and if that sample passes the batch is accepted apart from the failing cylinder, while a second failure rejects the batch.

6.1.4 requires storage-tank and tank-truck product to be inspected and accepted one by one against Table 1.

6.2 refers sampling safety to GB/T 3723. 6.3 requires tail gas from the determinations to be treated so as to prevent environmental pollution.

7 Marking, packaging, transport, storage and safety information

7.1.1 requires a quality certificate to leave the works with the product, carrying 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 kilograms; and the number of this document together with the purity of the octafluorocyclobutane.

7.1.2 requires the words for electronic grade octafluorocyclobutane to be painted on the packaging container. 7.1.3 refers the packaging mark to GB 190, the colour mark to GB/T 7144 and the label to GB 15258 and GB/T 16804.

7.2.1 requires the cylinders to comply with GB/T 5099 (all parts), GB/T 5100 and GB/T 33145. 7.2.2 refers filling, transport and storage to GB/T 14193, TSG R0005, TSG 07 and TSG 23, and points to the national regulations on hazardous chemicals and on special equipment for the safety management requirements.

7.2.3 gives formula (4) for the maximum filling: the mass of octafluorocyclobutane in the cylinder in kilograms is the filling coefficient, 1.31 kg/L, multiplied by the internal volume marked on the cylinder in litres.

7.2.4 recommends cylinders whose internal surface has been treated and names DISS 716 or CGA 660 for the valve outlet connection. 7.2.5 requires leakage and contamination of the valve opening to be prevented. 7.2.6 requires storage in a cool, dry, ventilated warehouse away from heat sources and out of direct sunlight.

7.3 refers the safety information to Annex A.

A Annex A (normative) Safety information

A.1 gives the basic data: chemical formula C4F8; the Chinese name of the substance and the English name Octafluorocyclobutane; relative molecular mass 200.032, calculated from the 2018 international relative atomic masses; CAS number 115-25-3; UN dangerous goods number 1976.

A.1.4 lists the physical properties: boiling point -6.4 °C, melting point -40.2 °C, critical temperature 115.0 °C, critical pressure 2777 kPa, critical density 0.616 g per cubic centimetre, vapour pressure at 20 °C 270 kPa, vapour density at 0 °C 9.338 kg per cubic metre, liquid density at 15 °C 1.48 g per cubic centimetre.

A.2 describes the hazard: octafluorocyclobutane is a colourless low-pressure liquefied gas whose rapid evaporation in the liquid state can cause frostbite; the vapour of the liquefied gas is at first heavier than air and can spread along the ground; heating to high temperature can generate irritating, corrosive and toxic gases.

A.3 requires closed operation and full ventilation of the plant, trained operators and strict observance of the operating procedure, and keeping away from heat sources and hot surfaces.