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Sulfuryl fluoride

硫酰氟

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Sulfuryl fluoride

1 Scope

This Standard specifies the requirements, test methods, acceptance, quality assurance period and signs, labels, packaging, storage and transportation for sulfuryl fluoride.

This Standard applies to sulfuryl fluoride consisting of sulfuryl fluoride and impurities generated in its production.

NOTE: See Annex A for other names, structural formulas and basic physicochemical parameters of sulfuryl fluoride.

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 190, *Packing symbol of dangerous goods*

GB/T 1604, *Commodity pesticide regulations for acceptance*

GB/T 1605-2001, *Sampling method for commodity pesticides*

GB 3796, *General rule for packing of pesticides*

GB/T 6681, *General rules for sampling gaseous chemical products*

GB/T 6682-2008, *Water for analytical laboratory use - Specification and test methods*

GB/T 8170-2008, *Rules of rounding off for numerical values and expression and judgement of limiting values*

GB 15603, *Rule for storage of chemical dangers*

GB 17916, *Specifications for storage and preservation of toxic goods*

3 Requirements

3.1 Appearance

It is colorless and odorless gas at normal temperature and pressure. It is a colorless

homogeneous liquid under pressure or freezing.

3.2 Technical indicators

Sulfuryl fluoride shall also meet the requirements of Table 1.

4 Test methods

WARNING -- Persons using this Standard shall have practical experience in laboratory work. Determination of pH value shall be carried out in a fume hood. This Standard does not address all safety issues. It is the user's responsibility to take appropriate safety and health measures and to ensure compliance with the relevant state regulations.

4.1 General provisions

Unless other requirements are specified, the reagents and water used in this Standard refer to the analytically pure reagents and grade three water specified in GB/T 6682-2008. The determination of the inspection results shall be carried out according to 4.3.3 in GB/T 8170-2008.

4.2 Sampling

Conduct according to GB/T 6681 and 5.3.4 in GB/T 1605-2001. Use random sampling method to take samples from any steel cylinder for each batch of products. The sampling volume shall not be less than 300mL (the cylinder pressure shall not be less than 5.0MPa, and the filling factor shall be 1.0kg/L).

4.3 Identification test

Infrared spectroscopy -- There shall be no obvious difference between the infrared absorption spectra of the specimen and the sulfuryl fluoride standard gas in the range of $1800\text{cm}^{-1}\sim 400\text{cm}^{-1}$. The infrared spectrum of sulfuryl fluoride standard gas is shown in Figure 1.

100 (or other chromatographic columns with the same column efficiency).

Sample injector: 5mL.

4.4.4 Preparation of chromatographic column

4.4.4.1 Application of fixative

Weigh 0.5g of polychlorotrifluoroethylene wax. Use ether or acetone to dissolve in the beaker. Pour 25g of carrier and mix well. Dry under infrared light for later use.

4.4.4.2 Packing of chromatographic column

Connect a small funnel to the outlet of the washed and dried chromatographic column. Fill the column with the prepared packing in batches. At the same time, tap the column wall continuously until the packing is 1.5cm away from the column outlet. Move the funnel to the inlet of the column. Plug a small dollop of silanized glass wool at the outlet end. Connect it to vacuum pump via rubber hose. Turn on the vacuum pump. Continue to slowly add the packing. Keep tapping on the column wall. Pack it evenly. The packing is completed. Stuff a small dollop of glass wool at the inlet end. Squeeze properly to keep stuffing from moving. (Or purchase other chromatographic columns with the same column efficiency).

4.4.4.3 Aging of chromatographic column

Connect the column inlet end to the vaporizer. The outlet end is temporarily not connected to the detector. Pass the carrier gas at a flow rate of 15mL/min. Rise the temperature to 150°C in several stages. At this temperature, conduct at least 24h aging.

4.4.5 Operating conditions of gas chromatography

Temperature (°C): 100 for column temperature, 150 for gasification chamber, 150 for detector chamber.

Carrier gas flow rate (mL/min): 30.

Injection volume (mL): 2.

Retention time (min): 0.8 for air, 1.3 for carbon dioxide, 1.7 for sulfuryl fluoride, 4.3 for chlorine, 4.8 for water, 6.9 for sulfur dioxide, 8.9 for sulfuryl chloride fluoride.

Stop time (min): 20.

The above operating conditions of gas chromatography are typical operating parameters. According to the characteristics of different instruments, the given operating parameters can be properly adjusted, so as to obtain the best results. A typical sulfuryl fluoride gas chromatogram is shown in Figure 2.

ΣA_i - The sum of the peak areas of all component chromatographic peaks (excluding the air peak).

4.4.8 Tolerance

The difference between the two parallel determination results of sulfonyl fluoride volume fraction shall not be greater than 0.2%. Take the arithmetic mean as the determination result.

4.5 Determination of pH value

4.5.1 Test instruments

Porous gas washing bottle: 250mL.

Balance: the accuracy is 0.01g.

Acidity meter: the division value of pH is 0.01.

4.5.2 Determination steps

Add 100mL of carbon dioxide-free distilled water to the gas washing bottle. Connect the washing bottle to the sampling cylinder with a rubber tube. Then place the cylinder on the balance. Control the flow rate of the absorbed gas to be about 300mL/min, that is, about 1g/min. Inject 1g of sulfonyl fluoride gas. Then use the pH meter to measure the pH of the solution in the absorption bottle.

4.5.3 Tolerance

The difference between the results of three parallel determinations of pH value shall not be greater than 0.2. Take the arithmetic mean value as the determination result.

5 Acceptance and quality assurance period

5.1 Acceptance

It shall comply with the provisions of GB/T 1604.

5.2 Quality assurance period

Under the specified storage and transportation conditions, the quality assurance period of sulfonyl fluoride is 2 years from the production date. During the quality assurance period, all indicators shall meet the standard requirements.

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