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Determination of hydrocarbon impurities of propylene for industrial use - Gas chromatographic method

工业用丙烯中烃类杂质的测定 气相色谱法

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Determination of hydrocarbon impurities of propylene for industrial use - Gas chromatographic method

WARNING: This document does not intend to explain all safety issues related to its use. Users are responsible for taking appropriate safety and health measures to ensure compliance with relevant national laws and regulations.

1 Scope

This document specifies the gas chromatography method for the determination of industrial propylene purity and hydrocarbon impurities, including methane, ethane, ethylene, propane, cyclopropane, isobutane, n-butane, propadiene, acetylene, trans-2-butene, 1-butene, isobutylene, cis-2-butene, 1,3-butadiene, methylacetylene, etc.

This document is applicable to the determination of industrial propylene with a purity greater than 98% (volume fraction) and a hydrocarbon impurity content not less than 1 mL/m^3.

2 Normative references

The provisions of the following documents constitute the essential clauses of this document through normative references in this text. Among them, for referenced documents with dates, only the versions corresponding to the dates are applicable to this document; for referenced documents without dates, the latest versions (including all amendments) are applicable to this document.

GB/T 3393 Ethylene and propylene for industrial use - Determination of trace hydrogen - Gas chromatographic method

GB/T 3394 Determination of trace carbon monoxide, carbon dioxide and acetylene of ethylene and propylene for industrial use - Gas chromatographic method

GB/T 3396 Determination of trace oxygen in ethylene and propylene for industrial use - Electrochemical method

GB/T 3723 Sampling of chemical products for industrial use - Safety in sampling

GB/T 3727 Ethylene or propylene for industrial use - Determination of trace water

GB/T 8170 Rules of rounding off for numerical values & expression and judgment of limiting values

GB/T 12701 Ethylene and propylene for industrial use - Determination of trace oxygenates - Gas chromatographic method

GB/T 13290 Propylene and butadiene for industrial use - Sampling in the liquid phase

GB/T 19186 Propylene for industrial use - Determination of oligomers - Gas chromatographic method

3 Terms and definitions

There are no terms or definitions that require definition in this document.

4 Principle

4.1 Corrected area normalization method

Under the conditions specified in this document, the sample is injected into the chromatograph for analysis. The peak areas of each impurity and main component are measured, and the content of each impurity component is calculated by the corrected area normalization method. Impurities such as hydrogen, oxygen, carbon monoxide, carbon dioxide, water, oligomers and oxygen-containing compounds are determined using the corresponding standard methods, and the results are normalized with the results determined in this document.

4.2 External standard method

Under the conditions specified in this document, the sample to be tested and the external standard are injected into the chromatograph for analysis. The peak areas of the impurity components to be tested in the sample and the standard are determined, and the content of each impurity is calculated from the peak area ratio of the corresponding impurities in the sample and the standard. The purity of propylene can be obtained by subtracting the total amount of hydrocarbon impurities and the total amount of impurities such as hydrogen, oxygen, carbon monoxide, carbon dioxide, water, oligomers and oxygen-containing compounds determined by other standard methods from 100%.

5 Reagents and materials

5.1 Carrier gas and auxiliary gas

High-purity nitrogen or high-purity helium, with a purity of not less than 99.999% (volume fraction), dried and purified by silica gel and 5A molecular sieve.

5.2 Combustion gas

Hydrogen, with a purity of not less than 99.99% (volume fraction), is dried and purified by silica gel and 5A molecular sieve.

5.3 Combustion-supporting gas

Air, oil-free, dried and purified by silica gel and 5A molecular sieve.

5.4 Standard samples

With propylene as the background, the gaseous or liquid standard sample contains hydrocarbon components such as methane, ethane, ethylene, propane, cyclopropane, isobutane, n-butane, propadiene, acetylene, trans-2-butene, 1-butene, isobutylene, cis-2-butene, 1,3-butadiene, and methylacetylene. The gaseous or liquid standard sample with known hydrocarbon component content can be purchased from the market or prepared by weighing. The content of hydrocarbon components in the standard sample shall be similar to that of the sample to be tested. If prepared by oneself, the propylene background sample used in the preparation shall be checked in advance under the conditions specified in this document. There shall be no chromatographic peak eluting at the component to be tested; otherwise, it shall be corrected.

If a liquid standard is used, a standard cylinder with a movable piston can be used and filled with inert gas so that the pressure in the cylinder can be maintained at 1380 kPa or higher than the standard vapor pressure at room temperature to ensure the stability of methane, ethane, and ethylene components in the liquid standard. If the methane, ethane, and ethylene components cannot be kept stable, a liquid standard that does not contain these three impurities can also be selected.

6 Instruments

6.1 Gas chromatograph

A gas chromatograph equipped with a gas injection valve or a liquid injection valve and a hydrogen flame ionization detector (FID). The peak height generated by the instrument for the lowest measured impurity concentration specified in this document shall be at least twice the noise, and the dynamic linear range of the instrument shall meet the quantitative requirements.

6.2 Chromatographic columns

The chromatographic columns and typical operating conditions recommended in this document are shown in Table 1, and the typical chromatogram is shown in Figure 1. Other chromatographic columns or operating conditions that can achieve the same separation efficiency may also be used.

A stainless-steel sintered sand core with a pore size of 2 μm~4 μm can be used as a metal filter to filter out possible mechanical impurities in the sample and protect the injection valve. A stainless-steel capillary (or damper valve) of appropriate length is installed at the injection valve outlet to avoid sample vaporization, distortion, and effect on injection repeatability.

6.4.2 Gas injection valve (quantitative tube volume: 0.5 mL)

Liquid samples can be vaporized by flash vaporization devices, or other suitable sample vaporization methods can be used. The vaporization device shall ensure that the liquid sample is completely vaporized, so that the gas composition is consistent with the composition of the liquid sample, and the representativeness of the sample does not change. When injecting samples, the sampling cylinder is in a vertical state, and the propylene sample flows out of the sampling cylinder in a liquid state and enters the vaporization device. The vaporized sample is injected through a six-way gas injection valve.

7 Sampling

Sampling shall be carried out according to the safety and technical requirements specified in GB/T 3723 and GB/T 13290.

8 Measurement steps

8.1 Setting operating conditions

According to the instrument operating instructions, install and age the chromatographic column in the chromatograph. Then adjust the instrument to the operating conditions shown in Table 1, and start the measurement after the instrument stabilizes.

8.2 Corrected area normalization method

8.2.1 Determination of relative volume correction factor

Use a gas injection device or a liquid injection device to inject the standard sample (5.4) into the gas chromatograph under the conditions specified in Table 1. If a liquid injection device is used, operate the liquid standard sample according to 6.4.1; if a gas injection device is used, operate the liquid standard sample according to 6.4.2, or directly connect the gas injection valve to inject the gas standard sample. Repeat the measurement twice and measure the peak area of each hydrocarbon component.

The volume correction factor (f_i) of each hydrocarbon component relative to propylene is calculated according to formula (1).

The difference between the two determination results of the correction factor f_i of each component shall not be greater than 5% of its average value. The average value is taken as the relative volume correction factor of the component, and three significant figures shall be retained.

If a liquid sampling device is used to analyze the sample, and stable liquid standards of methane, ethane, and ethylene are not available, the relative volume correction factors of these three components can be replaced by the relative volume correction factors measured by gaseous standards, or the theoretical relative volume correction factors can

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