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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 11141-2014

Replacing GB/T 11141-1989

Light olefins for industrial use - Determination of trace sulfur

工业用轻质烯烃中微量硫的测定

Issued on: July 8, 2014 **Implemented on: December 1, 2014**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine;
Standardization Administration of the PRC

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Foreword

This standard was drafted in accordance with GB/T 1.1-2009 given rules. This standard replaces GB/T 11141-1989 "light olefins in the determination of trace sulfur oxide Microcoulometry." The main changes in the standards and GB/T 11141-1989, compared as follows:

--- Modify the standard name;

--- Increased UV fluorescence method (see Chapter 3);

--- Oxidation Microcoulometry electrolyte in accordance with the requirements of the method of preparation to instrument manual preparation (see 426, 1989, version 4.6.);

--- Oxidation Microcoulometry the water bath temperature to 60 °C ~ 70 °C, remove the water bath apparatus Figure (see 3.3.5.1,1989 Version 6.3.2);

--- Oxidation Microcoulometry added flash vaporization device (see 4.3.4). The standard proposed by China Petroleum and Chemical Corporation. This standard by the National Standardization Technical Committee Petrochemical Chemical Technical Committee (SAC/TC63/SC4) centralized. This standard was drafted. China Petroleum & Chemical Corporation Shanghai Research Institute of Petrochemical. The main drafters of this standard. Li Cheng Wei, Xu Jing early, Zhang Yuhong. This standard replaces the standards previously issued as follows:

--- GB/T 11141-1989. Light olefins for industrial use - Determination of trace sulfur

1 Scope

China's national method for the trace sulfur in industrial light olefins - the ethylene, propylene and butenes that feed the polymer and petrochemical industry, in which sulfur is the impurity that poisons catalysts. It specifies the determination of trace sulfur in light olefins, C2 to C4, by ultraviolet fluorescence and by oxidative microcoulometry. The ultraviolet fluorescence method applies over a sulfur content of 0.2 mg/kg to 100 mg/kg; the oxidative microcoulometric method covers its own stated range. Two methods for the same

determination is not redundancy: they have different ranges, different tolerance of the matrix and different equipment costs, and a laboratory chooses between them on what it already has and on the concentration it expects. The principle in both cases is combustion. The sample is burned in oxygen so that every sulfur compound present - mercaptan, sulfide, carbonyl sulfide, whatever it happens to be - becomes sulfur dioxide, and it is that total which is measured, in the one method by the fluorescence emitted when the sulfur dioxide is excited by ultraviolet light, in the other by the current needed to titrate it coulometrically. Measuring the total rather than the individual species is the right answer for this application, because a polymerisation catalyst is poisoned by sulfur in any form. The standard sets the apparatus, the calibration against standards prepared in a matching matrix, the sample introduction - which for a liquefied gas under pressure is where most of the error enters - the procedure, the calculation and the precision. Issued on 8 July 2014 and in force since 1 December 2014, it replaces GB/T 11141-1989.

This standard specifies the light olefins (C2 ~ C4) in the determination of trace sulfur UV fluorescence and oxidation Microcoulometry. This standard UV fluorescence method is suitable for the determination of sulfur content of 0.2mg/kg ~ 100mg/kg of light olefins, Oxidative Microcoulometry Measured at 0.5mg/kg ~ 100mg/kg of light olefins suitable for sulfur content. This standard is not intended to illustrate its use of all safety-related issues. The user's responsibility to take appropriate safety and health measures Facilities, to ensure compliance with the provisions of the relevant national regulations.

2 Normative references

The following documents for the application of this document is essential. For dated references, only the dated version suitable for use herein Member. For undated references, the latest edition (including any amendments) applies to this document.

GB/T 3723 Sampling of chemical products for industrial use General Safety (GB/T 3723-1999, idt ISO 3165. 1976)

GB/T 6682 analytical laboratory use specifications and test methods (GB/T 6682-2008, ISO 3696. 1987, MOD)

GB/T 8170 repair value expressed about the rules and limit values and judgment

GB/T 13289 Industrial liquid and gaseous ethylene sampling method (GB/T 13289-2014, ISO 7382. 1986, NEQ)

GB/T 13290 Industrial liquid propylene and butadiene sampling method (GB/T 13290-2014,

ISO 8563. 1987, NEQ) SH/T 1142 Industrial cracking carbon four liquid sampling

3 Ultraviolet fluorescence method

3.1 Principle of the method After the sample into the gaseous or vaporized liquid sample combustion tube by the carrier gas is mixed with oxygen and burned, most of which trace sulfur conversion for two Sulfur oxides (sulfur trioxide generated small portion), the sample gas generated by combustion in the removal of water after UV irradiation, the ultraviolet light absorbing sulfur dioxide Energy into the excited state of sulfur dioxide (SO2 *), fluorescent emission when the excited state returns to sulfur dioxide during steady state and Detected by a photomultiplier tube, the resulting signal value calculated from the sulfur content in the sample. Warning. excessive UV exposure is harmful to health, the test must avoid ultraviolet light and a secondary or scattered radiation to the direct rays of the body Various parts of the body, especially the eyes hazard.

3.2 Reagents and materials

3.2.1 thiophene or dibutyl sulfide. purity of not less than 99% (mass fraction) for the preparation of sulfur standard stock solution.

3.2.2 iso-octane or n-heptane. for the preparation of sulfur standard stock solution solvent. Sulfur content shall not be higher than 0.2ng/μL, if necessary, to deal with the Blank correction sulfur content of the solvent.

3.2.3 Carrier gas. argon, purity of not less than 99.99% (volume fraction).

3.2.4 Reaction gas. oxygen purity of not less than 99.99% (volume fraction).

3.2.5 sulfur standard stock solution. 1000ng/μL. Weigh about 0.46g dibutyl sulfide (or 0.26g thiophene), accurate to 0.0001g, Add 100mL flask, with iso-octane or n-heptane is diluted to the mark line. The sulfur content in the solution according to equation (1) calculations.