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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 17606-2009

Replacing GB/T 17606-1998

**Determination of sulfur in crude-oil by energy-dispersive X-ray
fluorescence spectrometry**

原油中硫含量的测定 能量色散X-射线荧光光谱法

(English Translation)

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Foreword

This revised standard adopts ASTM D4294:2003 "Petroleum and Petroleum Products Standard Test Method for Determination of sulfur emitted energy dispersive X-Ray fluorescence spectroscopy" (in English).

This standard is based on ASTM D4294, redrafted 2003. This standard ASTM D4294:2003 main differences are as follows.

- ASTM D4294:2003 name for the "oil and petroleum products in the Standard Test Method for Determination of sulfur emitted energy dispersive X-Ray fluorescence spectrometry, "this standard entitled" Determination of sulfur content in crude oil energy dispersive X-ray fluorescence spectrometry";
- ASTM D4294:2003 for petroleum and petroleum products, this standard applies to crude oil;
- This standard applies to moisture content does not exceed 0.5% of the crude oil sample;
- This standard does not adopt ASTM D4294:2003 specifically described with respect to petroleum products.

This standard replaces GB/T 17606-1998 "Determination of sulfur content in crude oil energy dispersive X-ray fluorescence spectrometry." this standard GB/T 17606-1998 and main technical differences are as follows.

- The standard "range 1", the mass fraction of sulfur content measurement range from GB/T 17606-1998 in "0.05% 5.00%" is replaced by "0.0150% to 5.00%";
- This standard adds "4 interference";
- This standard GB/T 17606-1998 in "Reagent 4" to "6 reagents and materials";
- GB/T 17606-1998 specified with nitrogen to make the optical path, the optical path of the

standard is no longer requested;

--- GB/T 17606-1998 in calibration standards into three groups of 14, 10 of the standard into two groups;

--- GB/T 17606-1998 There are no loading problems like heavy oil and heavy oil will be described, the present standard adds this part;

--- GB/T no quality control of the narrative 17606-1998, the present increase in the standard "13 Quality Control";

--- The standard by ASTM D4294:2003 Dui GB/T 17606-1998 in "9 Precision" in the formula has been modified.

The standard proposed by China National Petroleum Corporation.

This standard by the National Standardization Technical Committee of Oil and Gas.

This standard was drafted. China Petroleum Daqing Oilfield Engineering Co., Ltd. Analysis and Testing Center, China Petrochemical Chemical Research Institute of Petroleum, China Petroleum Dalian Petrochemical Company Environmental Quality Inspection Center, in northwest China Petroleum Geology Center and petrochemical laboratories.

1 Scope

1.1

standard applies to the determination of total sulfur content of crude oil, sulfur content measured in the range of 0.0150% to 5.00%.

1.2

This standard applies to moisture content does not exceed 0.5% of the crude oil sample, if the crude oil sample moisture content exceeds 0.5%, to in the case of the sample without destroying the integrity of dehydration. The water content of crude oil according to GB/T 8929-2006 for testing.

At a concentration of 1.3 units of International System of Units defined as a standard, the preferred sulfur content per unit of mass fraction.

1.4

This standard does not list all of the security problems, users should establish some appropriate security measures and practical management system to ensure that Safety, prevention knowledge, see Chapter 5.

2 Normative references

The following documents contain provisions which, through reference in this standard and become the standard terms. For dated references, subsequent Amendments (not including

errata content) or revisions do not apply to this standard, however, encourage the parties to the agreement are based on research Whether the latest versions of these documents. For undated reference documents, the latest versions apply to this standard.

GB 4075-2003 Sealed radioactive sources - General requirements and classification GB/T 4756-1998 Petroleum liquid manual sampling method GB/T 8929-2006 Determination of water content of crude oil distillation

3 Methods Summary

The sample is placed in X- ray beam emitted by the source, the measurement of sulfur characteristic X- ray spectral intensity and spectral intensity cumulative pre Good spectral intensity of the standard sample is prepared in the first comparison, and sulfur content of the samples should be within a known calibration standard of the sulfur content in the range to give Samples with sulfur content mass fraction expressed.

4 Interference

4.1

When the crude oil sample other elements emit X- rays, producing a spectral interference, the detector can not detect the correct emission of sulfur emitted X-Line, resulting in a spectral superposition. The aqueous sample, alkyl lead, silicon, phosphorus, calcium, potassium, and halides, sulfur content exceeds one-tenth or Several hundred milligrams per kilogram, it will produce spectral interferences. The device according to the manufacturer's operating instructions to calibrate the instrument to eliminate interference.

4.2

Changes in the concentration of elements in the sample matrix can change directly affects absorption of X- rays, thus changing the measurement result of each element fruit. This interference usually occurs in X- ray fluorescence analysis, but not spectral interference.

4.3

Some instruments equipped with noise reduction software, can automatically check and reduce interference.

Crude oil sample 4.4 components are quite different with the white oil of 8.1, the matrix material composition can be selected with the same or similar components Sample analysis.

5 Instruments

Energy dispersive X- ray fluorescence spectrometer, as long as the structural integrity of the design of energy-dispersive X- ray fluorescence spectrum analyzer can make Use, the instrument should have at least the following components.

5.1

X- ray emission source, its energy is higher than 2.5keV.

Warning. In addition to other precautions, the use of radioactive sources, should be in accordance with the requirements of GB 4075-2003 well shielded to avoid