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PETROCHEMICAL INDUSTRY STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

ICS 75.080

E 30

NB/SH/T 0174-2015

Replacing NB/SH/T 0174-1992

Petroleum products and hydrocarbon solvents –

Detection of thiols and other sulfur species - Doctor test

石油产品和烃类溶剂中硫醇和其他硫化物的检测 博士试验法

[ISO 5275:2003, MOD]

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Issued on: October 27, 2015

Implemented on: March 01, 2016

Issued by: National Energy Administration

Table of contents

Foreword.....	3
1 Scope	5
2 Normative references	5
3 Method summary.....	6
4 Method application	6
5 Reagents and materials	6
6 Instruments.....	8
7 Samples and sampling	8
8 Test procedures	8
9 Results representation	10
10 Precision.....	10
11 Test report.....	11
Appendix A (Informative) Clause number comparison between this standard and ISO 5275:2003.....	12

Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard replaces SH/T 0174-1992 “Halogen qualitative test of aromatic hydrocarbons and light petroleum products thiols (Doctor test)”. As compared with SH/T 0174-1992, in addition to editorial changes in this standard, the main technical changes are as follows:

- CHANGE the standard name into “Petroleum products and hydrocarbon solvents - Detection of thiols and other sulfur species - Doctor test”.
- This standard applies to hydrocarbon solvents and petroleum fractions (including intermediates and products); SH/T 0174-1992 applies to aromatic hydrocarbon and light petroleum products:
- ADD the Chapter 2 “Normative references”;
- In Chapter 5, DETAIL the solution preparation; and DEFINE that the potassium iodide solution, the acetic acid solution and the starch solution shall be prepared daily;
- In Clause 8.1 “Initial tests”, ADD the specific test procedure in the presence of phenolic interfering substances; whilst in SH/T 0174-1992, the interfering substances are only briefly described in Chapter 6 “Report” in the form of notes;
- SUPPLEMENT the contents of the sample initial test and judgment;
- MODIFY the results report form; EXTEND the number of conditions of results report from two to seven.

This standard, through redrafting methods, modifies and adopts the international standard ISO 5275:2003 “Petroleum products and hydrocarbon solvents - Detection of thiols and other sulfur species - Doctor test”.

This standard has been structurally added an informative appendix (Appendix A) on the clause number comparison between this standard and ISO 5275:2003.

The technical differences between this standard and ISO 5275:2003 AND the causes are as follows:

- CHANGE the contents of paragraph 3 of the scope chapter into the Chapter 4 “Method” of this standard for application;

Petroleum products and hydrocarbon solvents –

Detection of thiols and other sulfur species - Doctor test

WARNING: The application of this standard may involve certain hazardous materials, operations and equipment. However, this standard does not make any recommendations on all security issues related to this. It is the responsibility of the user to establish appropriate safety and protective measures AND to determine the suitability of the relevant regulatory limits prior to the use of this standard.

1 Scope

This standard specifies the test methods of using Doctor reagent to qualitatively determine the thiols, hydrogen sulfide and elemental sulfur.

This standard applies to hydrocarbon solvents and petroleum fractions (including intermediates and products). The initial test of this standard can also be able to detect the presence of peroxide and phenolic substances, BUT it is not applicable to the conditions when the peroxide and phenolic substances are greater than the trace. When the carbon disulfide content is too high (the mass fraction of sulfur content is more than 0.4%), it will cause the water phase darkening, which will interfere with this test.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this Standard.

GB/T 4756 Method for manual sampling of petroleum liquids (GB/T 4756-1998, eqv ISO 3170:1988)

GB/T 6682-2008 Water for analytical laboratory use - Specification and test methods (ISO 3696:1987, MOD)

GB/T 27867 Petroleum liquid - Automatic pipeline sampling (GB/T27867-2011, 1503171:1988 IDT)

3 Method summary

SHAKE the test specimen that is added with sodium plumbite; OBSERVE the mixed solution; from its appearance, JUDGE whether there is thiol, hydrogen sulfide, elemental sulfur or peroxide. Then ADD sulfur powder, SHAKE and OBSERVE the appearance change of the final mixed solution, in order to further determine the presence of thiols.

4 Method application

This standard provides a test method of using the detection threshold of thiols concentration to determine the pass or fail, AND the detection threshold varies depending on the sample to be tested. It is often used as an alternative to thiols quantitative determination method.

5 Reagents and materials

5.1 Overview

Unless otherwise specified, it shall use the analytical pure reagents. Water shall comply with the level 3 requirements in GB/T 6682-2008.

5.2 Lead acetate trihydrate ($\text{CH}_3\text{COO})_2\text{Pb}\cdot 3\text{H}_2\text{O}$

It is analytical pure, used for the preparation of sodium plumbite solution.

5.3 Sodium hydroxide (NaOH)

It is analytical pure, used for the preparation of sodium hydroxide solution of mass fraction 10%.

5.4 Sodium plumbite solution (Doctor reagent)

5.4.1 DISSOLVE 25 g of lead acetate trihydrate into 200 mL of water; FILTER it and ADD the filtrate into 100 mL of water solution which is dissolved with 60 g of sodium hydroxide. In the boiling water bath, HEAT this mixed solution for 30 min $\pm$ 5 min; COOL it down and ADD water to dilute it to 1000 mL.

5.4.2 STORE this solution in a closed vessel. If it is not clear, it shall be filtered before use.

5.5 Cadmium chloride (CdCl_2)

6 Instruments

6.1 Mixing cylinder: glass, with plug, the capacity is 50 mL, used to mix solution in the test process.

6.2 Measuring cylinder: glass, the capacity is 5 mL and 10 mL respectively, used for measuring the reagent.

6.3 Separatory funnel: glass, with a plug, and the capacity is 50 mL.

7 Samples and sampling

7.1 Unless otherwise specified, the sampling shall follow the provisions of GB/T 4756 or GB/T 27867; AND the samples to be analyzed shall be representative.

7.2 The sample shall be thoroughly shaken before the test so that the sample is thoroughly mixed; at room temperature if the sample vapor pressure exceeds 30 kPa, it shall always release the pressure in order to ensure safety.

8 Test procedures

8.1 Initial tests

8.1.1 Phenolic substances - If it is suspected that the tested sample contains phenolic substances which are used as an oxidation inhibitor AND it may interfere with the aqueous phase color of the test results, it may use the mixing cylinder (6.1) to take 10 mL of sample AND add 5 mL of sodium hydroxide solution (5.3) of mass fraction in 10%, AND violently SHAKE the measuring cylinder for 15 s. Then OBSERVE its color developing conditions. If there is no significant color developed, FOLLOW the provisions of 8.1.2 to continue tests. If significant color is developed, STOP the test.

Note: any developed color that is deeper than the light yellow is a significant color. If a yellow color is developed, modification may be required based on the fourth type of color developing conditions in Table 1.

8.1.2 Sulfide and peroxide - PLACE the 10 mL of sample and 5 mL of sodium plumbite solution (5.4) in a mixing cylinder (6.1); vigorously SHAKE the mixing cylinder for 15 s. Then OBSERVE the mixed solution, and FOLLOW the provisions in Table 1 to conduct judgment.

shall not be too much, just cover the interface between the sample and the sodium plumbite mixed solution. Violently SHAKE the mixing cylinder for 15 s, and LET it standing for $60\text{ s} \pm 5\text{ s}$. OBSERVE whether the mixed solution has brown or black precipitate. If a precipitate is formed, it is considered that there is thiol higher than the thiol detection threshold of this standard.

9 Results representation

9.1 If the test cannot proceed due the presence of interfering substances as determined in accordance with the procedures of 8.1.1, it shall report as "Invalid test - there is an interfering substance".

9.2 If there is peroxide as determined in accordance with the procedures of 8.2, it shall report as "Invalid test - there is peroxide".

9.3 If the sample has black precipitant as soon as the sample is mixed with the sodium plumbite solution after the test as specified in 8.1.2, it shall report as "Fail (positive) - there is hydrogen sulfide". If there is black or brown precipitate after adding sulfur in accordance with Clause 8.4 after eluting the sulfide, it shall report as "Positive (fail) - there is hydrogen sulfide and/or thiol".

9.4 If the mixed solution of sample and sodium plumbite becomes milky white after oscillating as specified in 8.1.2, and then the color gradually becomes darker, it shall report as "Positive (fail) - there is thiol and/or elemental sulfur".

9.5 If the mixed solution of sample and sodium plumbite becomes milky white after oscillating as specified in 8.1.2, AND the brown or black precipitate is formed in the mixed solution after adding sulfur as specified in 8.4, it shall report as "Positive (fail) - there is thiol".

9.6 If the mixed solution of sample and sodium plumbite has no precipitate, no color change or changes into light yellow after oscillation as specified in 8.1.2, AND no precipitate is formed in the mixed solution after adding sulfur as specified in 8.4, it shall report as "Negative (pass); if there is precipitate formed in the mixed solution, it shall report as "Positive (fail) - there is thiol".

10 Precision

Since this method is to judge the presence of sulfur in the sample through comparing the active sulfur content in single sample AND the detection threshold as specified in this method at the specified test conditions, it is impossible to determine the precision.

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