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Heat transfer fluids - Determination of thermal stability

有机热载体热稳定性测定法

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1 Scope

1.1 This standard specifies the test method for the thermal stability of unused heat transfer fluids, including heat transfer fluids which have a maximum use temperature above their boiling point at atmospheric pressure.

1.2 This standard does not evaluate the suitability of heat transfer fluids of organic silicone types.

1.3 This standard is related to some hazardous substances, operations and equipment. It is not intended to advise on all safety issues related to this. Therefore, it shall establish appropriate safety and protective measures prior to the use of this standard, determine the applicability of the restrictions of relevant provisions.

2 Normative references

The provisions in following documents become the provisions of this standard through reference in this standard. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this standard; however, parties who reach an agreement based on this standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

HG/T 3115 Borosilicate glass 3.3 - Properties (HG/T 3115-1998, idt ISO 3585:1991)

SH/T 0558-1993 Standard test method for boiling range distribution of petroleum fractions (Gas chromatography)

3 Terms and definitions

The following terms and definitions apply to this standard.

3.1

Heat transfer fluids

The heat transfer fluid is a general term for organic substances which are used as a heat transfer medium.

4 Summary of method

4.1 The heat transfer fluids are heated at a predetermined temperature. The deterioration rate of the heat transfer fluids is determined to evaluate the thermal stability of the heat transfer fluids. The deterioration rate is the sum of the mass fractions of high-boiling components, low-boiling components, gaseous decomposition products, nonvolatile decomposition products. The mass fraction of the gaseous decomposition products is usually negligible (see 7.5.1).

4.1.1 The unused heat transfer fluid can be sealed in a glass ampoule which is placed in a metal protective tube, or otherwise added into a steel tester and sealed. The metal protective tube or steel tester which is loaded with a glass ampoule is heated to a specified time at the specified test temperature, the test temperature is preferably within the upper limit of the temperature range in which the product to be tested is used.

4.1.2 After heating to the specified time, open the glass ampoule or steel tester. Use the gas chromatography in SH/T 0558-1993 to determine the mass fraction of high-boiling components and low-boiling components. Use a tube distiller to determine the content of the nonvolatile decomposition products.

4.1.3 Compare the test results of the heat transfer fluid before and after it is heated.

5 Meaning and application

5.1 Under the laboratory conditions as specified in this standard, this method records and reflects the effect of heat on the heat transfer fluid as a function of temperature and heating time. The test results provide information on the thermal stability of different types of heat transfer fluids at certain heating temperatures and heating times.

5.2 It cannot extrapolate the information on the performance of heat transfer fluids in industrial heat transfer devices from the test results of this method, because the performance of heat transfer fluids in the device is also affected by other materials, various pollutants, heat accumulation, temperature in the circulatory systems, and so on.

6 Instruments and materials

6.1 Instrument

6.1.1 Heater: It can control the temperature within ±1 °C of the test temperature,

7.2 Heating of specimen

START heating from room temperature. When the temperature rises to 50 °C below the test temperature, CONTROL the heating rate to below 2 °C/min. KEEP the temperature constant during the test, so that the temperature deviation at any point of the heat transfer fluid (including the tester's heating wall) does not exceed ±1 °C of the test temperature. The test time is the duration from point when the set test temperature is reached to the point when the heating is stopped. The test time for each type of specimen is not less than 720 h. The test temperature and the test time are determined according to the requirements of relevant product standards or regulations. Then DETERMINE the deterioration rate of the specimen according to 7.4.

7.3 Opening tester

7.3.1 Overview

After the tester is cooled to room temperature, REMOVE it out from the heater. Then RECORD the appearance of the specimen. FINISH the corresponding weighing.

According to the test temperature and test time, when glass ampoule or steel tester, it shall take appropriate safety measures.

7.3.2 Opening glass ampule

PLACE the glass ampoule in a Dewar. Under freezing of a mixture of acetone or isopropyl alcohol and dry ice (about -70 °C), LOWER its internal pressure. After 5 min ~ 10 min, OPEN the glass ampoule, to let the gas evaporate completely under room temperature. After restoring to room temperature, immediately WEIGH the mass (m_4) of the glass ampoule, accurate to 0.1 mg. When weighing, it shall include all glass pieces and remove the attached condensate. PLACE a portion of the specimen in a gas chromatography bottle for chromatographic analysis. STORE the remaining specimen in a well-sealed glass bottle for other analysis.

7.3.3 Opening steel tester

PLACE the steel tester in a Dewar. Under freezing of a mixture of acetone or isopropyl alcohol and dry ice (about -70 °C), LOWER its internal pressure. After 5 min ~ 10 min, OPEN the sealed cap of the steel tester, to let the gas evaporate completely under room temperature. After restoring to room temperature, REMOVE the attached condensate. WEIGH the mass (m_4) of the steel tester, accurate to 0.1 mg. PLACE a portion of the specimen in a gas chromatography bottle for chromatographic analysis. STORE the remaining specimen in a well-sealed glass bottle for other analysis.

7.4 Analysis

7.4.1 Based on the mass difference of the glass ampoule or steel tester before and after being opened, DETERMINE the content of the gaseous decomposition product.

7.4.2 USE the gas chromatography of SH/T 0558-1993 to perform simulate distillation to determine the boiling point range of the specimen before and after heating. USE a capillary column. Before determination, it shall first determine the boiling point range of the reference oil. The boiling point of the determined reference oil shall meet the repeatability requirements of the method in SH/T 0558-1993. When using the method of SH/T 0558-1993 to determine the specimen, only REPORT the results of the boiling point distribution of the fraction before the final boiling point of 538 °C. For the fraction above 538 °C in the specimen, the content of the nonvolatile decomposition product shall be calculated according to 7.5.2.

7.4.3 WEIGH the mass of the hollow tail ball of the tube distiller (m_5), accurate to 0.1 mg. Then ADD about 4 g of the heated specimen into the tail ball. Accurately WEIGH the mass of tail ball plus specimen (m_6), accurate to 0.1 mg. ROTATE the ball tube in the rotary tube distiller. USE a vacuum pump to create vacuum. At the end of distillation, the pressure shall reach to below 10 Pa ± 0.2 Pa. Slowly HEAT the tube distiller to make its temperature reach to 250 °C, so that the heat transfer fluid does not undergo further thermal decomposition or boiling hysteresis during the distillation process. CONTINUE distillation until the mass of residues is constant. WEIGH mass of the tail ball (including the residue) after the test (m_7), accurate to 0.1 mg. It may use the gas chromatography to assess whether the distillation process is complete. The portion which remains volatile in the residue shall be less than 0.1% of the total mass of the heat transfer fluid.

7.5 Evaluation

7.5.1 Calculation of the content of gaseous decomposition product of specimen

Use the formula (1) to calculate the mass fraction of the gaseous decomposition product content of specimen, %:

$$G = (m_3 - m_1)/(m_2 - m_1) \times 100 \qquad \cdots\cdots(1)$$

Where:

m_1 - The mass of the empty glass ampoule or steel tester, in grams (g);

m_2 - The mass of glass ampoule or steel tester which contains unheated specimen, in grams (g);

chromatography, it must use the formula (3) and the formula (4) to correct the uncorrected content of low-boiling components (mass fraction) N' (%) and the uncorrected content of high-boiling components (mass fraction) H' (%):

$$N = N' \times (100 - G - U)/100 \qquad \cdots\cdots(3)$$

Where:

N - The corrected content of low-boiling components of the specimen (mass fraction), %;

N' - The content of low-boiling components as determined through the simulated distillation curve of the specimen (mass fraction), %;

G - The content of gaseous decomposition products of the specimen (mass fraction), %;

U - The content of nonvolatile decomposition products of the specimen (mass fraction), %.

$$H = H' \times (100 - G - U)/100 \qquad \cdots\cdots(4)$$

Where:

H - The corrected content of high-boiling components of the specimen (mass fraction), %;

H' - The content of high-boiling components as determined through the simulated distillation curve of the specimen (mass fraction), %;

G - The content of gaseous decomposition products of the specimen (mass fraction), %;

U - The content of nonvolatile decomposition products of the specimen (mass fraction), %.

7.5.4 Calculation of deterioration rate of specimen

The deterioration rate (mass fraction) of specimen Z (%) is calculated according to formula (5):

$$Z = G + N + H + U \qquad \cdots\cdots(5)$$

Where:

G - The content of gaseous decomposition products of the specimen (mass fraction), %;

N - The corrected content of low-boiling components of specimen (mass

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