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**Determination of explosion limits of combustible vapors and
gases at elevated temperature and pressure**

高温高压条件下可燃气体（蒸气）爆炸极限测定方法

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Determination of explosion limits of combustible vapors and gases at elevated temperature and pressure

Warning – After igniting the mixture of combustible gas (vapor) and air according to the method specified in this document, even if no propagation of flames is formed, it cannot be completely considered that the mixture will not explode.

Warning – The personnel who uses this document shall have hands-on experience in formal laboratory work. This document does not address all possible safety issues. It is the responsibility of the user to take appropriate safety and health measures and to ensure compliance with the conditions which are set by the relevant national regulations.

1 Scope

This document describes a method for the determination of explosion limits of the combustible mixture of combustible gases (vapors) and air at a given initial temperature and initial pressure.

This document applies to the determination of explosion limits of combustible gases (vapors) under the conditions of initial temperature below 200 °C, initial pressure below 1.0 MPa (absolute pressure), and maximum explosion pressure not greater than 15.0 MPa (absolute pressure).

This document does not apply to the determination of explosion limits of chemically unstable gases or mixed gases in which components may react with each other.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the version corresponding to that date is applicable to this document; for undated references, the latest version (including all amendments) is applicable to this document.

GB/T 12474, Method of test for explosion limits of combustible gases in air

GB/T 16425, Determination for minimum explosive concentration of dust clouds

GB/T 21844, Standard test method for concentration limits of flammability of chemicals (vapors and gases)

4 Test principle

Under the given initial temperature and initial pressure conditions, pre-mix a certain volume fraction of combustible gas (vapor) and air in the explosion reaction vessel; use electric spark or electric heating wire to ignite; judge whether an explosion occurs by the rising value of the explosion pressure (not less than 5% of the given initial pressure); systematically change the concentration of the combustible gas (vapor) until the minimum or maximum concentration where the explosion occurs is measured.

5 Test device

5.1 General

The test device for explosion limits is mainly composed of an explosion reaction vessel, an ignition device, a pressure measurement unit, a gas distribution unit, a temperature control unit, etc. See Appendix A for a schematic diagram of the test device.

5.2 Explosion reaction vessel

5.2.1 The explosion reaction vessel shall be a spheroidal shape or a cylindrical test vessel with an aspect ratio of 1, which is resistant to high temperature and high pressure. The volume shall not be less than 1 L and should be 20 L, 12 L or 5 L; the shape specifications shall be in accordance with the relevant provisions for test device in GB/T 16425 and GB/T 21844 respectively.

5.2.2 The expected operating temperature of the explosion reaction vessel under initial pressure conditions shall not be less than 200 °C, and the explosion-resistant pressure shall be able to withstand a maximum explosion pressure of not less than 15.0 MPa (absolute pressure) without permanent deformation.

5.2.3 The maximum explosion pressure of combustible gases (vapors) at elevated temperature and pressure shall not be higher than the maximum working pressure of the explosion reaction vessel.

5.2.4 For the determination of explosion limits of low-flammable substances such as ammonia, halogenated substances and certain amines, a 20 L spherical stainless steel explosion tank or an explosion reaction vessel with a larger diameter should be used.

5.2.5 An oil-free stirring device shall be equipped in the explosion reaction vessel.

5.2.6 The connecting pipe fittings between the explosion reaction vessel and the high-pressure gas cylinder shall be made of 316 stainless steel pipes with a pressure not lower than 4.0 MPa (absolute pressure). All pipelines and joints connected to the explosion reaction vessel shall be able to withstand a pressure not lower than 15.0 MPa (absolute pressure).

5.3 Ignition device

5.3.1 The mixture of combustible gas (vapor) and air can be ignited by electric spark, or by electric fuse or high-temperature hot wire.

5.3.2 The ignition energy of the ignition device shall be sufficient to ignite the combustible gas mixture to be tested, and the pressure increase of the ignition source to the mixed gas shall not be greater than 0.5% of the initial pressure.

5.4 Pressure measurement unit

5.4.1 Pressure sensors with low-pressure range and high-pressure range, which are installed in the explosion reaction vessel, shall be able to withstand the continuous high temperature of 200 °C during the test. The response frequency of the pressure sensor with high-pressure range shall not be lower than 10 kHz; it should be installed flush. The distance between the top of the sensor probe and the inner wall of the container for non-flush installation shall not exceed 10 cm.

5.4.2 The pressure sensor with low-pressure range is used to measure the partial pressure of different component gases in the gas distribution process. The lower limit of the range of the pressure sensor with low-pressure range shall reach 0.07 kPa (absolute pressure), and the reading error shall not exceed 1%, and shall be able to meet the gas distribution accuracy requirements. The range of the pressure sensor with high-pressure range shall be able to meet the requirements for measuring the maximum explosion pressure, and shall be able to detect and determine whether an explosion has occurred.

5.5 Gas distribution unit

5.5.1 The gas distribution unit shall adopt an oil-free vacuum pump, and shall realize vacuuming the explosion reaction vessel to below 0.002 MPa (absolute pressure).

5.5.2 The gas distribution accuracy shall not be lower than 0.1% volume fraction.

5.6 Temperature control unit

The ambient temperature in the explosion reaction vessel shall be measured by a k-type thermocouple. The controllable temperature range is from room temperature to 200 °C, and the temperature control accuracy is ± 2 °C.

6 Test steps

Warning – It is strictly forbidden to disassemble the explosion reaction vessel under pressure.

6.1 Test preparation

± 2 °C of the initial temperature set for the test. Turn on the oil-free stirring device and stir for 5 minutes to mix the combustible gas and air in the explosion reaction vessel evenly. When measuring a variety of combustible gas mixtures with different concentrations, fill them into the explosion reaction vessel in sequence from low to high concentrations of combustible gases to be measured to the set partial pressure value.

6.2.5 Close the protective valve on the pipeline connecting the pressure sensor with low-pressure range and the explosion reaction vessel.

6.2.6 Turn on the pressure sensor with high-pressure range to record the pressure inside the explosion reaction vessel.

6.2.7 After mixing and stirring, let stand for 2 minutes before igniting. Use the pressure sensor with high-pressure range to measure and record the pressure change with time in the explosion reaction vessel after ignition. Compare the change of the maximum explosion pressure before and after ignition, and analyze and judge whether an explosion occurs according to 7.1.

6.2.8 After the ignition test, open the exhaust valve in the explosion reaction vessel, to release the test exhaust gas to the outside, and use inert gas to purge the explosion reaction vessel and corresponding pipelines, for no less than 5 purging times.

6.2.9 Repeat the test steps in 6.2.1 ~ 6.2.8, and use the progressive method to determine the explosion limit value. When determining the lower (upper) explosion limit, if no explosion occurs at a certain concentration, increase (decrease) the concentration of combustible gas (vapor) until the minimum (maximum) concentration that can cause explosion is measured; if an explosion occurs at a certain concentration, decrease (increase) the concentration of combustible gas (vapor) until the maximum (minimum) concentration at which the explosion cannot occur is measured. When the determination is close to the lower explosion limit, the change amount of the sample to be tested shall not be greater than 10% of the last injection volume each time; when the determination is close to the upper explosion limit, the change amount shall not be greater than 2% of the last injection volume each time. The unstable temperature and large fluctuation of pressure or temperature after filling a certain gas indicates that a reaction may have occurred before ignition, where the test shall be stopped immediately. Halogen reactions can cause a pressure drop, and oxidation reactions can cause a pressure rise.

6.2.10 Test to obtain the minimum concentration L_1 of combustible gas (vapor) for explosion and the maximum concentration L_2 for non-explosion near the lower explosion limit, where L_2 is lower than L_1 , and the difference between L_2 and L_1 is not more than 0.2% of the volume fraction. Repeat the test 3 times at the L_2 concentration to ensure that no explosion will occur at this concentration.

6.2.11 Test to obtain the maximum concentration U_1 of combustible gas (vapor) for explosion and the minimum concentration U_2 for non-explosion near the upper explosion limit, where U_2 is higher than U_1 , and the difference between U_2 and U_1 is

not more than 10% of U_1 . Repeat the test under the concentration of U_2 3 times to ensure that no explosion occurs at this concentration.

6.2.12 After the test is over, clean up the carbon deposits or other residues in the explosion reaction vessel in time.

6.3 Determination of explosion limits at elevated temperature and pressure

6.3.1 According to the determination method specified in GB/T 21844, gradually increase the temperature to determine the explosion limits of the combustible gas (vapor) under normal pressure and high temperature, where the temperature increase range shall not be higher than 50 °C. When the maximum explosion pressure of the mixed gas is close to the maximum working pressure of the explosion reaction vessel during the test, the determination test shall be terminated immediately.

6.3.2 Preheat the explosion reaction vessel and related components to the initial temperature set for the test and keep the temperature constant. If the sample to be tested is liquid vapor, the liquid container shall be preheated to the initial temperature set for the test, and the connecting pipeline shall be heated and protected; also, remote control of sample injection shall be realized.

6.3.3 After preheating the test device and related components to the set initial temperature, carry out the test and measurement according to the steps in 6.2.1 ~ 6.2.11.

7 Test data processing

7.1 When the explosion pressure increment after the combustible gas (vapor) is ignited is not less than 5% of the given initial pressure, it is determined that the mixed gas has propagation of flames, that is, an explosion has occurred. The calculation method is shown in Formula (1).

$$\frac{P_2}{P_1} \geq 1.05 \quad \dots\dots\dots (1)$$

Where:

P_1 – the value of the initial absolute pressure, in megapascals (MPa);

P_2 – the value of the maximum absolute pressure of explosion after ignition, in megapascals (MPa).

Note: A pressure increment of 5% is equivalent to an increase of 0.005 MPa per unit atmospheric pressure.

7.2 The lower explosion limit of combustible gas (vapor) is calculated according to Formula (2), and the upper explosion limit is calculated according to Formula (3).

and causes a sudden change in pressure, the test shall be stopped immediately and corresponding protective measures shall be taken.

8.5 An explosion-proof barrier shall be set up between the test device and the operator to prevent casualties caused by the rupture of the explosion reaction vessel.

8.6 The ignition device shall be provided with functions such as interlock and remote control, and it shall be ensured that the operator can only start the ignition when he is in the safe area outside the explosion-proof barrier.

8.7 Where the test device is equipped with a constant temperature box, the box shall be installed with an explosion-proof pressure relief sheet that opens outwards, and filled with inert gas to prevent explosion caused by accidental leakage of the mixed gas to be measured.

8.8 After the test, the exhaust gas shall be discharged outdoors in time and treated according to safety and environmental protection requirements.

8.9 The test operator shall carry out the test in strict accordance with the operating procedures, and shall use the necessary protective equipment to avoid harm to the human body caused by the mixed gas to be tested or explosive products.

8.10 The laboratory shall prepare necessary safety emergency materials.

9 Calibration of the test device

9.1 The pressure sensor and temperature sensor of the test device shall be regularly verified according to the requirements of the instructions, and the verification period shall not exceed 3 months.

9.2 Before the formal test of the test device, use ethylene with a purity of not less than 99.9% to calibrate. Under the initial conditions of 50 °C and 0.3 MPa (absolute pressure), the lower explosion limit is 2.6%, and the upper explosion limit is 40.0%. If the test results meet the following requirements, the device is considered to be operating normally:

- a) For repeated test results measured in the same laboratory, the error shall not be greater than 5%;
- b) For repeated test results measured in different laboratories, the error shall not exceed 10%.

10 Test report

The test report shall include the following contents:

- 6 – pressure relief door;
- 7 – explosion reaction vessel;
- 8 – stirring blade;
- 9 – magnetic stirrer;
- 10 – explosion pressure sensor;
- 11 – ignition electrode;
- 12 – observation window.

Figure A.1 – Schematic diagram of 5 L/12 L long neck high temperature and high pressure explosion limit test device

A.1.2 Heating chamber

The heating chamber uses hot air circulation to maintain a constant temperature. The temperature ranges from room temperature to 200 °C.

A.1.3 5 L explosion reaction vessel

Place the 5 L explosion reaction vessel in the heating chamber, and test the high temperature and high pressure explosion limit of combustible gas or vapor in the explosion reaction vessel. For the size and specification of the vessel, see GB/T 21844.

A.1.4 Pressure sensor

The test device (inside) is equipped with a pressure sensor 1 and a pressure sensor 2 for gas distribution, and a pressure sensor 3 for testing the explosion pressure rise value, wherein the test range of the pressure sensor 1 is 0 MPa ~ 0.1 MPa (absolute pressure), the test range of pressure sensor 2 is 0 MPa ~ 1.0 MPa (absolute pressure), and the test range of pressure sensor 3 is 0 MPa ~ 15.0 MPa (absolute pressure).

A.1.5 Temperature sensor

The test device is provided with two temperature sensors, which are the temperature sensor of the sample to be tested and the temperature sensor of the heating chamber. The temperature sensor of the sample to be tested is used to test the temperature of the gas or vapor introduced into the explosion reaction vessel and the temperature measurement range is from room temperature to 800 °C. The temperature sensor of the heating chamber is used to test the temperature in the heating chamber, and the temperature measurement range is from room temperature to 300 °C.

A.1.6 Magnetic stirrer

The magnetic stirrer is set in the 5 L explosion reaction vessel, to mix the combustible gas or vapor to be tested with air in the vessel evenly.

The temperature control jacket is arranged outside the explosion reaction vessel, the explosion reaction vessel is heated by heat conduction oil, and the temperature control range is from room temperature to 200 °C.

A.2.4 Ignition electrode

The ignition electrode is set in the explosion reaction container, and is used to ignite the sample gas or vapor to be tested in the vessel.

A.2.5 Pressure sensor

The test device is provided with two pressure sensors, which are respectively the gas distribution pressure sensor and the explosion pressure sensor, wherein the test range of the gas distribution pressure sensor is 0 MPa ~ 1.0 MPa (absolute pressure), and the test range of the explosion pressure sensor is 0 MPa ~ 15.0 MPa (absolute pressure).

A.2.6 Temperature sensor

The test device is provided with two temperature sensors, which are the temperature sensor of the sample to be tested and the jacket temperature sensor. The temperature sensor of the sample to be tested is used to test the temperature of the gas or vapor introduced into the explosion reaction vessel, and the temperature range is from room temperature to 800 °C. The jacket temperature sensor is used to test the temperature inside the jacket, and the temperature measurement range is from room temperature to 300 °C.

A.2.7 Magnetic stirrer

The magnetic stirrer is set in the 12 L explosion reaction vessel, to mix the sample gas or vapor to be tested with air in the vessel evenly.

A.3 20 L spherical high temperature and high pressure explosion limit test device

Figure A.3 is a schematic diagram of the 20 L high temperature and high pressure explosion limit test device. The device is composed of a 20 L spherical explosion reaction vessel, an ignition electrode, a temperature control jacket, a pressure sensor, a temperature sensor, a magnetic stirrer and other parts. The size and specifications of the device shall be in accordance with the device requirements of GB/T 16425.

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