

SN

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

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**Determination of hexavalent chromium in foods for export
(English Translation)**

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1 Scope

This Part of SN/T 3077 specifies the determination method of flash point by modified continuously closed cup tester.

Note: For the substance being tested, the flash point is not a constant physical and chemical constant. It is related to the instrument design, instrument use conditions, test execution steps.

Therefore, the flash point can only be defined according to the standard test method. There is no guarantee that the results measured by different methods or different test instruments have a universally valid correlation.

This Part applies to the determination of the flash point of fuel oil, lubricating oil, solvent oil, and other liquids; the measurement range is 35 °C ~ 225 °C.

Note: This Part can determine samples with flash points below 35 °C or above 225 °C, but the precision has not been established.

If the flash point test method specified in the product specification required by the user is

not this method, neither this method nor any other method can replace the specified method without comparative data and approval by the specification setter.

This Part involves certain hazardous materials, operations, and equipment, but does not provide recommendations for all safety issues related to this. Therefore, it is necessary to establish appropriate safety and protective measures and establish appropriate operating procedures before using the standard. Special hazard warnings are shown in

7.2, 8.5, 10.1.2 warnings.

2 Normative references

The following documents are essential for the application of this document. For all dated references, only the dated version applies to this document. For all undated references, the latest version (including all amendments) applies to this document.

ASTM D93 Standard test methods for flash point by Pensky-Martens closed cup tester

ASTM D4057 Standard practice for manual sampling of petroleum and petroleum products

ASTM D4177 Standard practice for automatic sampling of petroleum and petroleum products

ASTM D6450 Standard test method for flash point by continuously closed cup (CCCFP) tester

ASTM D6708 Standard practice for statistical assessment and improvement of expected agreement between two test methods that purport to measure the same property of a material

ASTM E300 Standard practice for sampling industrial chemicals

3 Terms and definitions

The following terms and definitions apply to this document.

3.1

Flash point

The lowest temperature (corrected to 101.3 kPa pressure) at which the vapor of the specimen ignites under the specified test conditions. In a closed test chamber, the specimen is confirmed to have flashed when the instantaneous pressure increase caused by the hot flame is not less than 20 kPa.

3.2

Dynamic

The state when the vapor on the specimen and the specimen have not reached

temperature equilibrium when the fire source is introduced.

4 Method overview

4.1

Set the test temperature to be not less than 18 °C lower than the expected flash point.

Inject 2.0 mL $\pm$ 0.2 mL of the specimen into the sample cup. The temperature of the specimen and the sample cup is not less than (the same below) 18 °C lower than the expected flash point; it can be cooled if necessary. Raise the sample cup and press it tightly with the lid to achieve airtightness. At this time, the total volume of the test chamber is 7.0 mL $\pm$ 0.3 mL.

4.2

After the test chamber is closed, the temperature difference between the specimen and the lid shall be within 1 °C; it is heated at a uniform rate. A high-voltage arc of a certain energy is discharged in the test chamber at specified intervals. After each ignition, an indefinite amount of air (see Table 1) is introduced into the test chamber, to provide the required oxygen for the next flash test. The pressure in the test chamber is voltage arcs, a connecting pipe connected to the lid for pressure detection and air introduction. It also requires an electronic device to control the temperature of the test chamber and a digital display that can display the specimen temperature. The apparatus and its main components are shown in Figures A.1 and A.2:

- Test chamber: The test chamber consists of a sample cup and a temperature- controlled lid with a total volume of 7.0 mL $\pm$ 0.3 mL. The metal contact between the lid and the sample cup is to ensure good heat conduction; the incomplete seal is to ensure that the test chamber is at ambient atmospheric pressure during the test.

Its dimensions are shown in Figure A.2. When the temperature rises, the pressure in the test chamber will be detected. Excessive sealing will cause the pressure increase caused by the temperature and vapor pressure of the specimen to be higher than the atmospheric pressure. Poor heat conduction will cause a large difference in temperature between the specimen and the lid.

- Sample cup: The sample cup can be made of nickel-plated aluminum or other materials with similar thermal conductivity. The sample cup is a cup that forms a seal after closing and can accept air injection test chamber function. Its total volume is 7.0 mL $\pm$ 0.3 mL and can accommodate 2.0 mL $\pm$ 0.2 mL of specimen. Its dimensions and requirements are shown in Figure A.2.

- Specimen temperature sensor: The specimen temperature sensor (see Figure A.1) is a stainless steel thermocouple with a diameter of 1 mm (NiCr-Ni or similar) and a response time of $t(90) = 3$ s. Its insertion depth into the specimen shall be not less than 2 mm; the

temperature sensing sensitivity is 0.1 °C; the accuracy is ± 0.2 °C;

it is best to connect a temperature display.

- Magnetic stirring: The instrument must be able to stir the sample. The sample cup is equipped with a rotating magnet, which can rotate a small stirring magnet placed in the sample cup containing the specimen. The diameter of the stirring magnet is

3.0 mm $\pm$ 0.2 mm and the length is 12 mm $\pm$ 1 mm. The speed of the driving magnet is 250 r/min $\sim$ 270 r/min.

- Air introduction device: The instrument must be able to introduce air immediately after each flash test. A small membrane air compressor generates a short air pulse and introduces air into the pressure sensor through the T-type inlet in the connecting pipe. The amount of air introduced depends on the sample temperature (see Table

1), 0.5 mL $\sim$ 2.0 mL.

- Temperature regulation device: During the test, the temperature of the test chamber is regulated by electric heating and cooling of the lid. The temperature control accuracy is ± 0.2 °C.

- High-voltage arc: Use high-voltage arc to ignite flammable vapors. The energy of each arc shall be 1.3 J $\pm$ 0.3 J (1.3 ws $\pm$ 0.3 ws)/arc; the arc time shall be within 19 ms $\pm$ 2 ms.

Note: When the specimen contains low-flash point substances or the flash point of the specimen is lower than the initial temperature, saturated vapor may be generated in the test chamber. Therefore, when the instrument is set up, it shall be able to enable that, after the test chamber is closed and before reaching the initial temperature, the arc shall be pre-set to an interval of 5 °C, when the temperature of the lid and the sample cup is in equilibrium.

- Flash point pressure test sensor: The flash pressure test sensor shall be connected to the connecting pipe in the lid, which has an operating range of 80 kPa $\sim$ 177 kPa, a minimum resolution of 0.1 kPa, a minimum accuracy of ± 0.5 kPa. It can measure an instantaneous pressure increase of at least 20 kPa above atmospheric pressure within 100 ms.

- Pipette or syringe: 2.0 mL $\pm$ 0.2 mL of specimen can be transferred using a pipette or syringe that meets the accuracy requirements.

7.1 Reagents and purity

Purity requirements for chemicals used refer to Table B.1. Unless otherwise specified, analytical grade reagents shall be used. Other grades of reagents may be used when the purity of the reagents used does not affect the accuracy of the measurement.