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NATIONAL ENVIRONMENTAL PROTECTION STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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Guideline for the determination of volatile organic compound leaks and uncovered liquid surface emissions

泄漏和敞开液面排放的挥发性有机物 检测技术导则

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Guideline for the determination of volatile organic compound leaks and uncovered liquid surface emissions

1 Scope of application

This Standard specifies the technical requirements for the detection of volatile organic compounds (VOCs) originating from equipment leaks and uncovered liquid surface emissions. It specifies the detection methods, instrument and equipment requirements, quality assurance and control of VOCs from unorganized emission sources such as equipment leaks and uncovered liquid surface. This Guideline is not applicable to the direct determination of mass emission rates of VOCs from leakage and uncovered liquid surface emission sources.

2 Terms and definitions

2.1 leak sources

Various equipment and pipelines that contain VOCs materials and may leak and emit, including valves, flanges and other connections, pumps, compressors, pressure relief devices, open-ended valves or open pipelines, sampling connection systems, exhaust ports of pump and compressor sealing systems, tank breathing ports, inspection port seals, etc.

2.2 open-ended valve

Valves in which one side of the valve seat is in contact with organic gas or volatile organic liquid and the other side is in contact with the atmosphere, but a pressure relief device is not included.

2.3 uncovered liquid surface

The unorganized emission source from the uncovered liquid surface of the collection, transportation and storage equipment of production materials containing VOCs and the uncovered liquid surface of the production process wastewater and waste liquid collection and storage and purification treatment equipment.

2.4 leak definition concentration

The concentration limit specified in relevant emission standards or regulations, measured on the surface of the leakage source or the uncovered liquid surface, indicating the existence of VOCs leakage and requiring measures to be controlled

(based on the measurement reading of an instrument calibrated with a reference compound).

2.5 no detectable emission

When the concentration of VOCs measured on the surface of the source to be measured, after subtracting the background, is lower than 2.5% of the standard concentration limit, it is defined as no detectable emission.

2.6 reference compound

The reference compounds in this Guideline refer to the VOCs compounds specified in relevant emission standards or regulations and used as the calibration benchmark for leakage measurement instruments. For example, if an emission source in a certain emission standard uses methane as the reference compound, the standard concentration limit is 500×10^{-6} mol/mol.

2.7 calibration gas

It refers to VOCs compounds used during calibration to adjust instrument readings to known concentrations. The calibration gas is usually a reference compound standard gas close to the concentration limits of the relevant control standard.

2.8 response factor

It refers to the ratio of the concentration value of a known concentration of VOCs compounds to the instrument reading calibrated with a reference compound of the same concentration value.

2.9 response time

It refers to the time it takes for the instrument to measure the concentration of VOCs from when the instrument reading begins to change to when the instrument finally displays 90% concentration of a stable reading.

2.10 zero gas

It refers to clean air with VOCs content less than 10×10⁻⁶ mol/mol (calculated as methane).

2.11 reference compound standard gas

It refers to the reference compound standard gas whose balance gas is high-purity air, whose concentration is near the relevant control standard concentration limit, and whose relative expanded uncertainty is 2% (k=2).

2.12 non-reference compound gas

It refers to compound standard gases other than reference compounds. It is used to

determine the response ratio of non-reference compounds and reference compounds on the detection instrument. When measuring the non-reference compound gas sample, the measured ratio can be used to convert the instrument response value into the actual concentration value of the non-reference compound.

3 Instruments and equipment

3.1 Portable testing equipment

- **3.1.1** Before use, check or experiment shall be carried out to ensure that the detector responds to the main VOCs components emitted by the emission source to be measured. Instrument detector types include flame ionization detectors, photoionization detectors, infrared absorption detectors, etc., and can also be other types of detectors.
- **3.1.2** The measuring range of the instrument shall be able to meet the measurement requirements of the standard concentration limits in the relevant control standards. Its resolution shall ensure readability within $\pm 2.5\%$ of the leak definition concentration or standard concentration limit in the emission standard.
- **3.1.3** Be equipped with an electric sampling pump that can provide continuous flow. The sampling flow rate measured at the top of the sampling probe with a glass wool plug or filter installed to protect the instrument shall be in the range of 0.10~3.0 L/min.
- **3.1.4** Be equipped with a sampling probe. The outer diameter of the front end of the sampling probe shall ensure that it can enter the narrow gaps of various types of equipment for detection. It generally does not exceed 7 mm.
- **3.1.5** The instrument must be explosion-proof safe and pass explosion-proof safety inspection certification.

3.2 Instrument performance evaluation

Install and start the instrument correctly according to the start-up and initial adjustment requirements in the instrument manual.

3.2.1 Determination of response factor

The response factor of various VOCs emitted by the emission source under test shall be determined before use of the instrument. The response factor can be measured directly or obtained from reference materials. After the response factor of the instrument is determined, it is not necessary to repeat the measurement. The determination of the response factor can ensure that the instrument has sufficient response to the VOCs that need to be detected. When a known emission source emits a single VOC, the detection value can be converted into the concentration of the compound through the response factor of the compound.

instrument's sampling pump or sampling flow rate is adjusted and the instrument's response time changes, the response time must be remeasured before use.

- **3.2.3.1** Introduce the zero gas from the sampling probe port. After the instrument reading is stable, quickly switch to the calibration gas. Record the time required for the instrument to reach 90% of the final stable display reading. Repeat this step 3 times. The average value is taken as the response time of the instrument.
- **3.2.3.2** The instrument response time shall not exceed 30 s. When measuring the response time, the sampling pump, dilution probe (if any), sampling probe and filtering device shall be in place.

3.3 Wind direction anemometer

A wind direction anemometer of which the wind speed resolution is ≤ 0.1 m/s, the wind direction resolution is $\le 3^{\circ}$, and starting wind speed is ≤ 0.5 m/s.

4 Testing technical requirements

4.1 Instrument calibration

After the instrument is warmed up and zero gas calibrated, introduce the reference compound standard gas for calibration into the instrument sampling probe. After the instrument reading is stable, adjust the instrument reading according to the standard value. If the instrument reading cannot be adjusted to a suitable concentration value, it indicates that the instrument is faulty and shall be eliminated before use. Calibration can be a single point concentration calibration. The single point concentration value shall be close to the standard concentration limit. Multi-point calibration is also possible. Standard concentration limits shall be within the range of calibration concentrations.

4.2 Sampling testing

4.2.1 Testing of leak sources

Place the sampling probe at the relevant part of the equipment or device where leakage emissions may occur. Move along its periphery at a speed less than 10 cm/s. Also keep an eye on the instrument readings. If you notice an increase in readings, slow down the sampling probe movement until the maximum reading is measured. Stop at maximum reading. The residence time is approximately 2 times the instrument response time. Record the maximum reading. The sampling and testing regulations for leakage sources are as follows.

4.2.1.1 Valves

The most likely place for a valve to leak is the seal between the valve stem and valve body. Place the sampling probe at the valve stem stuffing box gland. Sample by moving assumed that no detectable emissions are present. If there are emission points on the pipeline where leakage may occur, background and source sampling shall be performed to determine whether no detectable emission is present.

4.2.4 Leak detection procedures for other equipment

4.2.4.1 Use auxiliary detection methods such as infrared thermal imaging cameras, Fourier transform infrared imaging spectrometers, and leakage ultrasonic detectors

On the premise that the on-site conditions meet the testing requirements of the instrument used, there is no interference, and the optical imaging instrument responds to the VOCs emitted by the emission source to be measured, USE infrared thermal imaging cameras, Fourier transform infrared imaging spectrometers, leakage ultrasonic detectors, etc. to detect and scan the area of the equipment under test to quickly locate possible leakage emissions. This type of method can be used to help locate the source of leaking emissions at higher locations that are inaccessible to inspectors. After using the above auxiliary detection method, the source to be measured that can be reached by the detection personnel with the help of the extended sampling probe still needs to be tested according to the requirements in 4.2.1 or 4.2.2.

4.2.4.2 Method of spraying soap solution to generate foam at the connections of equipment where leakage may occur

This method is suitable for situations where there are no continuously moving parts, the surface temperature of the equipment is not higher than the boiling point of the solution or not lower than the freezing point of the solution, there is no excessively large open area in contact with the air that would prevent soap bubbles from being produced, and there are no significant traces of liquid leakage. If the above applicable conditions cannot be met, testing shall be carried out according to the requirements in 4.2.1 or 4.2.2.

4.2.4.3 Spray soap solution on all possible leak points

Special leak detection soap solutions can be used OR a solution prepared with a certain concentration of detergent and water can be used. Use a pressure sprayer or squeeze bottle to spray the solution. Observe possible leak points for soap bubbles to form. If no suds appear, it can be assumed that there is no discharge or leak. If soap bubbles appear, testing shall be carried out according to the requirements in 4.2.1 or 4.2.2.

4.3 Recording and reporting requirements

The environmental conditions of the testing site, relevant meteorological conditions, instrument and equipment calibration results, relative error results of indication values, etc. shall be recorded in a standardized manner. Together with the test results, they serve as the content of the test report.

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