# HJ

NATIONAL ENVIRONMENTAL PROTECTION

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# Ambient air – Determination of aldehyde and ketone compounds – High performance liquid chromatography

环境空气　醛、酮类化合物的测定　高效液相色谱法

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**Warning: Some chemicals used in this Method are harmful to human health, so protective devices shall be worn as required during operation to avoid contact with the skin and clothes. All chemicals shall be fully sealed and independently stored in a cool, shady place to prevent leakage and pollution.**

## 1 Application Scope

This Standard specifies high performance liquid chromatography for the determination of aldehyde and ketone compounds in ambient air.

This Standard applies to the determination of thirteen aldehyde and ketone compounds in ambient air, including formaldehyde, acetaldehyde, acrolein, acetone, propionaldehyde, crotonaldehyde, methacrolein, 2-butanone, butyraldehyde, benzaldehyde, valeraldehyde, m-tolualdehyde and n-hexaldehyde. Other aldehyde and ketone compounds can also be used in the analysis of this Standard provided that they pass the suitability validation.

When the sampling volume is 0.05 $m^3$, the detection limit of this Method is 0.28 μg/$m^3$ ~ 1.69 μg/$m^3$ and the low-limit of determination is 1.12 μg/$m^3$ ~ 6.76 μg/$m^3$. For details see Annex A.

## 2 Normative References

This Standard cites the following documents or clauses therein. For undated reference documents, their valid editions apply to this Standard.

HJ/T 55, *Technical guidelines for fugitive emission monitoring of air pollutants*

HJ/T 194, *Manual methods for ambient air quality monitoring*

## 3 Method principle

Use sampling tubes packed with coating 2,4-Dinitrophenylhydrazine (DNPH) to collect a certain volume of air sample. The aldehyde and ketone compounds in sample react with DNPH coated on silica gel in accordance with Equation (1) through the catalytic action of strong acid, generating stable, coloured hydrazine derivatives. After the elution of acetonitrile, use the ultraviolet (360 nm) or diode array detector of high performance liquid chromatographer for detection. Use the retention time for

Use sealing caps to seal both ends of sampling tubes; use tin foil or aluminum foil to wrap sampling tubes up for storage and transportation at low temperature (< 4°C). If analysis can't be carried out in time, samples shall be stored for not more than 30 days at low temperature (< 4°C).

### 7.3 Preparation of samples

Add acetonitrile to elute sampling tubes; let acetonitrile to flow past sampling tubes naturally with the flow direction opposite to the direction of air flow. Collect the eluent in a 5 mL volumetric flask before adding acetonitrile to scale; use injection syringe (5.7) to absorb the eluent; filter through syringe filter (5.8); and transfer to a 2 mL brown bottle for determination. If the eluent can't be analyzed in time after filtration, it can be stored for 30 days at 4°C.

### 7.4 Preparation of blank samples

#### 7.4.1 Whole-course blanks

Each sampling shall include at least one whole-course blank: bring a sampling tube to site with its both ends open; continue for a sampling cycle without collecting sample in it; seal it in the same way as that of sampling tubes with samples; and bring it to laboratory. Prepare blank samples in accordance with the same procedures of 7.3.

#### 7.4.2 Blank sampling tubes

Take the same batch of sampling tubes in laboratory to prepare blank samples in accordance with the same procedures of 7.3.

## 8 Analytical procedures

### 8.1 Reference chromatographic conditions

Mobile phase: acetonitrile/water. Gradient elution: maintain 60% acetonitrile for 20 min; increase acetonitrile from 60% linearly to 100% within 20 ~ 30 min; then reduce acetonitrile to 60% within 30 ~ 32 min; and maintain for 8 min.

Test wavelength: 360 nm.

Flow rate: 1.0 mL/min.

Sample size: 20 μL.

### 8.2 Calibration

#### 8.2.1 Preparation of standard series

Measure respectively 100 μL, 200 μL, 500 μL, 1 000 μL and 2 000 μL of standard working solution (5.4) to pour into a 10 mL volumetric flask; use acetonitrile to add to

For the details of the results of precision and accuracy see Annex B.

# 11 Quality assurance and quality control

## 11.1 Blank sampling tubes

At least 10% of each batch of sampling tubes shall be taken for blank value testing and the blank value shall meet the following requirements:

Formaldehyde less than 0.15 μg/tube;

Acetaldehyde less than 0.10 μg/tube;

Acetone less than 0.30 μg/tube;

Other matters less than 0.10 μg/tube.

## 11.2 Whole-course blanks

Each batch of samples shall include at least one whole-course blank and the determination result shall be lower than the method detection limit.

## 11.3 Parallel double sample

Each batch of samples shall include 10% of parallel double sample; when the sample quantity is less than 10, at least one parallel double sample shall be determined; and the relative deviation of the results of two parallel determinations shall be less than or equal to 25%.

## 11.4 Sampling flow

Observe from time to time whether the flow of sampler is stable during the sampling period. If the flow at the end of sampling is more than 15% different from the flow at the beginning of sampling, the samples shall be abandoned, and sampling shall be done once again.

## 11.5 Control of breakthrough capacity

The upper limit of aldoketone content in samples taken (in terms of formaldehyde) shall be less than 75% of the DNPH content of sampling tubes. The breakthrough capacity of aldoketones can be calculated in accordance with Equation (3):

$$C_T = C_{DNPH}' \frac{M_{CH_2O}}{M_{DNPH}} \qquad (3)$$

where,

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