9.7 Measurement methods for polycyclic aromatic hydrocarbons in suspended particles [1)2)]

9.7.1 Introduction

Airborne suspended particles are collected on a filter using either a high volume air sampler or low volume air sampler and, after sampling with benzene ethanol, they are transferred to a solution of acetonitrile where, if a high performance liquid chromatography (HPLC) is being used, they are possible to analyze polycyclic aromatic hydrocarbons such as benzo (a) pyrene, benzo (k) fluorantene, benzo (ghi) peryleme.

(1) Sample collecting methods Same as for 9.6.2.

(2) Test operation

① Sampling operation

There are several different types of extraction operation: Ultrasonic extraction using benzene-ethanol, Soxhlet extraction using dichloromethane, and supercritical extraction using either CO_2 or N_2O. Here, the simplest method of extraction, ultrasonic extraction, is explained.

<Ultrasonic extraction>

A fixed volume is removed from the sample filter, and after being finely chopped up, is placed in a centrifugal sediment tube with a 10 mℓ two-way stopper.

1 mℓ of ethanol and 3 mℓ* of benzene is then added, and the whole subjected to ultrasound for 15 minutes in an ultrasound scanner, and the organic components extracted. Following centrifugal sedimentation for 15 minutes at 3,000 rpm, the top 2 mℓ of the solution is transferred to another centrifugal sedimentation tube, 3 mℓ of 5% sodium hydroxide is added and, following mixing for approximately one minute in a lavomixer, centrifugal sedimentation is carried out for 15 minutes at 3,000 rpm. This 1 mℓ organic layer is then transferred to another test tube, and after drying, is redissolved in a fixed volume of acetonitryl, which is used as the sample solution for analysis.

*; Dichloromethane can also be used, but because its specific gravity is high, it is necessary to pay attention and consider suitability when extracting the lower layer following washing in alkali.

② Setting HPLC analysis conditions, and adjusting the equipment

The HPLC analysis conditions should be set appropriately with reference to the following:

Separation column	: ODS, 5 μ m
	Internal diameter 4.6mm φ, length 250 mm
Mobile phase	: Acetonitrile/water = 85/15
Flow amount	: 1.0 mℓ/min.
Sample injection volume	: 10 μ ℓ
Column temperature	: 40℃
Detector	: Fluorescence detector (excitation wavelength: 365 nm, emission wavelength: 410 nm)

③ Sample solution measurements

Approximately 20 μ ℓ of the sample solution, which has been adjusted in ①, is extracted using a microsyringe, and infused into the HPLC, the chromatograph is recorded, and about of the peak for the benzo (a) pyrene (hereinafter called BaP), either the peak surface area or the peak height is determined. The BaP concentration in the sample solution is determined from the calibration curve drawn up beforehand.

④ Drawing up the calibration curve

a) The BaP first standard solution (10 μ g/mℓ) is diluted with acetonitryl so that its concentration is 1 to 10 ng/mℓ, and a control series is compiled for the calibration curve. The control series has five or more steps, including zero.

b) Using the ③ operation, either the peak area or peak height is determined equivalent to each BaP concentration.

c) The calibration curve is drawn up from the relation between the peak surface area or height, and the BaP concentration.

⑤ Blank test

For the sample and the filter from the same lot, the sample solution is adjusted by means of the ①, the ③ operation is carried out, and the blank value (Ab; ng) is determined.

⑥ Sensitivity test

The ③ operation is carried out with regard to the standard solution around the middle of the calibration curve from the control concentration series, and fluctuations in the sensitivity are checked. This operation is carried out at least once for every 10 sample measurements.

(3) Calculating the concentration

From the results of ③ and ⑤ in (2), the airborne BaP concentration is calculated using next Eq. (1).

$$C = \frac{(A_s - A_b) \times ve \times E \times S \times 1,000}{v \times (\frac{4}{3}) \times vc \times V \times s \times \frac{293}{273+t} \times \frac{P}{101.3}} \quad \cdots\cdots (1)$$

Here,

C = Airborne BaP concentration at 0℃ (μ g/m^3)

A_s = BaP in the sample solution injected into HPLC

A_b = BaP blank (ng)

S = Filter area that collected the specimen (cm^2)

s = Filter area that was used for the measurement (cm^2)

E = Extracted solution volume (mℓ, normally 4 mℓ)

v = Volume of solution injected into HPLC (μ ℓ)

ve = Final sample solution volume (mℓ, normally 1 mℓ)

vc = Solution volume extracted after alkaline clean up (mℓ, normally 1 mℓ)

V = Air suction volume (ℓ)

t = Mean air temperature during sampling (℃)

P = Mean atmospheric pressure during sampling (kPa)