

## Illustrated Work Flow ————— Preparation of air pollutant arrestor and extraction of pollutant

Laboratory training

*This text is in line with “Measuring Air Pollution and the Protection of Cultural Properties in the Historic City of Nara, Japan” which ACCU Nara published in 2004 and 2005 on “e-learning” section of its website. (<http://www.nara.accu.or.jp/elearning/index.html>) Please also refer to the 2004 text for the outline, and 2005 text for the detailed analytical process.*

### Triethanol Cylindrical Filter Method

#### Overview of air pollution study

Simple concentration measurement of NO<sub>2</sub>, SO<sub>2</sub> and CL by triethanol cylindrical filter method (hereinafter referred to as “TEA-CF method”). After exposing a cylindrical cellulose filter impregnated with 29.6% triethanol aqueous solution (hereinafter referred to as “TEA aqueous solution”) to the atmosphere for a period of one month, quantitative analysis is conducted by ion-exchange chromatography to cumulatively determine per month average concentration.

Quantitative values are converted to per day weight (μg/day/100cm<sup>2</sup>: relative concentration), and are converted to absolute concentration (ppb) for NO<sub>2</sub> and SO<sub>2</sub>. A 31-day continuous roll autographic recorder is provided at main measurement points to perform temperature offset for NO<sub>2</sub>.

The measurement range for NO<sub>2</sub> is 1.9μg of NO<sub>2</sub>/day/100cm<sup>2</sup> and the range for SO<sub>2</sub> is 2.6μg of SO<sub>3</sub>/day/100cm<sup>2</sup>.

#### Conversion formula in the TEA-CF method

##### *Formula for conversion to relative concentration*

$$\begin{aligned} \text{CL}^- &= 257.15 * (\text{CCL}^- - \text{B}) / n && \text{unit: } \mu\text{gCL}^- / \text{day}/100\text{cm}^2 \\ \text{NO}_2 &= 257.15 * (\text{CNO}_2 - \text{B}) / n && \text{unit: } \mu\text{gNO}_2 / \text{day}/100\text{cm}^2 \\ \text{NO}_3 &= 257.15 * (\text{CNO}_3 - \text{B}) / n && \text{unit: } \mu\text{gNO}_3 / \text{day}/100\text{cm}^2 \\ \text{SO}_3 &= (\text{CSO}_3 - \text{B}) * 214.39 / n && \text{unit: } \mu\text{gSO}_3 / \text{day}/100\text{cm}^2 \end{aligned}$$

CCL<sup>-</sup>, CNO<sub>2</sub>, CNO<sub>3</sub>, CSO<sub>3</sub>: Concentration of analysis results (μg/ml)

B: Concentration of blank

n: Number of days of exposure

### *Temperature offset*

The concentration of NO<sub>2</sub> varies according to temperature. Temperature offset is executed using 20°C as the standard. The difference in reaction is that whereas CL<sup>-</sup> and SO<sub>3</sub> react with alkalis, NO<sub>2</sub> reacts chemically with the TEA impregnated in the sample.

$$\begin{aligned}C'TN &= CTN / TCOR \\ &= CTN / (NRT / NR20) \\ &= CTN * NR20 / NRT \\ &= CTN * (0.12 * 20 + 4.5) / (0.12 * T + 4.5) \quad \text{unit: } \mu\text{gNO}_2/\text{day}/100\text{cm}^2\end{aligned}$$

TCOR : Offset coefficient using 20°C as the standard

C'TN : NO<sub>2</sub> concentration by TEA-CF method of temperature offset

CTN : NO<sub>2</sub> concentration by TEA-CF method

NR : NO<sub>2</sub> adsorption of TEA-CF 100 cm<sup>2</sup> per day

T : Temperature at each point

### *Formula for conversion to absolute concentration*

$$\text{NO}_2 ; \text{CAS} = 0.118 * C'TN + 3.3 \text{ (ppb, 1 ppm = 1000ppb)}$$

CAS : NO<sub>2</sub> concentration (ppb) by automatic measurement instrument

C'TN : NO<sub>2</sub> concentration by TEA-CF method of temperature offset

$$\text{SO}_2 ; \text{CAS} = 0.043 * C'TS + 2.8$$

CAS : SO<sub>2</sub> concentration (ppb) by automatic measurement instrument

C'TS : SO<sub>3</sub> concentration by TEA-CF method

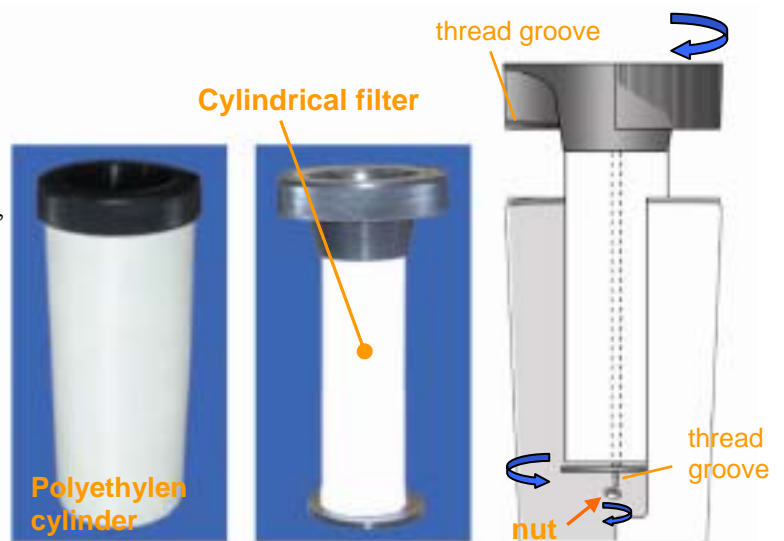
### *Features of TEA*

- Colorless liquid with minimal ammonia odor.
- Slightly viscous.
- Dissolves in water or ethanol.
- Heavier than water.
- Adsorbs to acidic gas.
- Comparatively low toxicity towards the human body.

## 1 Sample preparation

>Required items<

- 2, 2', 2'' nitro triethanol  
N(CH<sub>2</sub>CH<sub>2</sub>OH)<sub>3</sub> = 149.19
- Cylindrical filter (φ3.3 mm × 100 mm,  
103.7 cm<sup>2</sup> surface area, neutral)
- Ultra pure water
- Polyethylene cylinder
- beaker
- tweezers
- flat-bottom flask (1000ml)
- acid-free filter paper



1. The filters must not be touched with the bare hands. Ion exists in various places such as in the air, in people's bodies (surface of the skin, breath) and surface of apparatus. You must therefore be careful not to allow ion components to get mixed in when creating, extracting and preparing samples.



2. Dilute the nitro triethanol (TEA) 3 times.  
300ml of TEA + 600 ml of ultra pure water  
= 900 ml of TEA solution  
Shake well, and mix TEA and ultra pure water.



3. Immerse the cylindrical filter in the TEA solution for about 30 minutes.



4. Remove the cylindrical filter from the TEA solution, stand it on the spread out acid-free filter paper and remove the excess water. In addition to the samples for exposure, blanks for comparison or offset are also needed.



5. Mount the filter on the cylinder.

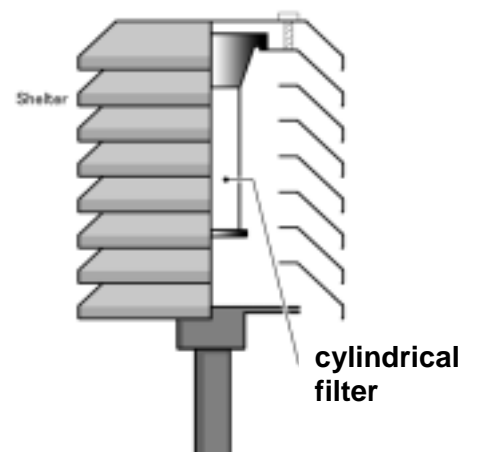


## 2 Exchange and expose the cylindrical filter

>Required items<

- Portable thermograph
- Precision Hygro-Termograph
- Datalogger
- Notebook PC

1. The filters must not be touched with the bare hands or shelter. Filters are exposed in the shelter placed 1.8m high from the ground, which is calibrated to retain 20% aeration.



2. Retrieve data from the thermograph. Since TEA-CF method requires compensated temperature to attain the concentration of NO<sub>x</sub>, the averaged temperature data derived in this step is used as the compensated temperature data. It also plays an important role as data for maintenance and control, as it gives a long-term observation of the cultural properties in the given area from the hygrothermal data in their surrounding.

When using a precision hygro-thermograph as shown in the photograph, replace the indicator paper and check for the remaining ink and battery.

*Note: Since the needle of this type of device is very sensitive to vibration, place it where there is least vibration.*



When using a datalogger, retrieve the digital data. Check for the remaining battery as well.

*Note: Datalogger here refers to a device with the function of accumulating, analyzing and indicating the data from various sensors such as voltage and electric current. It can also be integral with sensor, or a simplified measurement device. It is usually the case in the cultural property field, that datalogger refers to the type of devices with measurement and memory functions.*



about 70mm

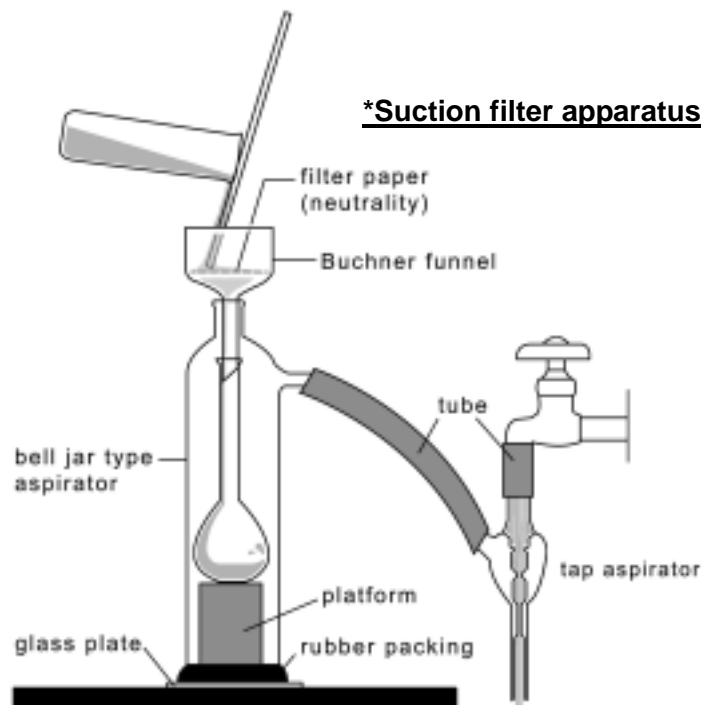
datalogger



### 3 Extraction of Pollutants

#### Required items

- Ultra pure water
- Tweezers
- Measuring cylinder
- Beaker (100ml)
- Tall beaker
- Hot plate
- Glass rod
- Suction filter apparatus\*
- Pipette (15 ml)
- Flat-bottom flask (200 ml)
- Measuring pipette (1 ml)
- Test tube (25 ml)
- Hydrogen peroxide (highest quality)  
( $H_2O_2 = 34.01$ )
- Filter paper



1. Divide the recovered TEA-CF into the proper size (about 8 – 16 divisions), place into a tall beaker. The TEA-CF must not be touched with bare hands.



2. Measure 100 ml ultra pure water in a measuring cylinder.





3. Put the cylindrical filters in the tall beakers and pour the solution into them.



4. Cover up the tall beakers with cellophane wrap or aluminum foil against intrusion of dust. Place the tall beaker on a hot plate heated to approximately 120°C and heat for about an hour.



5. After heating, crush the filter paper finely with a glass rod.

6. Place a flat-bottom flask (200ml) in a suction filter apparatus (hydraulic type in this case).



7. When the extraction process is completed, bring the volume of the filtered liquid to 200 ml by adding the required amount of ultra pure water and agitate well. This serves as the test solution.

*Note: Extraction of eluted substances could be made without such a device, but it would take considerable time under the atmospheric pressure.*

#### 4 Pre-treatment for Analysis

Before the extracted fluid is measured in ion-exchange chromatograph, a pre-treatment is necessary.

1. Put a stopper in the flask, turn it upside-down and shake. This serves as the test solution.

*Note: This is done for even dispersion of eluted substances in the solution.*



2. Take a 15ml-sample from each and inject the samples in test tubes.



3. Add 0.3 ml of highest quality hydrogen peroxide. Put a stopper in the flask, turn it upside-down and shake.





4. Bring the volume of the solution to 20 ml by adding the required amount of ultra pure water and agitate well (put a stopper in the flask, turn it upside-down and shake). This serves as the sample solution for analysis.



>Reference<

Wait until the reagent is stabilized, and start measuring after 24 hours. The analysis of the said four substances takes about 13 minutes per sample.



anion mixed standard solution



standard solution

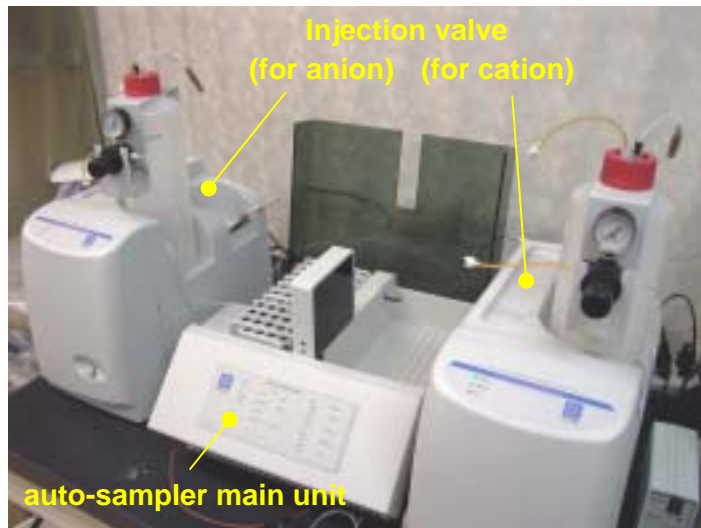


eluting solution

	Cl <sup>-</sup>	NO <sub>2</sub> <sup>-</sup>	NO <sub>3</sub> <sup>-</sup>	SO <sub>4</sub> <sup>2-</sup>
ST1	.5	.75	1.5	2
ST2	1	1.5	3	4
ST3	2	3	6	8
ST4	5	7.5	15	20

Unit: mg/l (ppm)

*Set the vials in the ion-exchange chromatograph with an automatic sampler.*



*Now Loading...*

Process steps provided by  
**Conservation Science Laboratory, Department of Cultural Properties,  
Faculty of Letters, Nara University**