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Application News

Gas Chromatography No.G261

Simplified Enrichment Method Using NeedlEx

Ambient odor analysis typically requires enrichment of analytes because odor thresholds are usually very low. Normally, a specialized device is required for enrichment and desorption during introduction into the GC; specialized devices often involve complex operation.
The NeedlEx introduced here can be used to selectively enrich volatile organic compounds in ambient air by desorbing them into the enrichment medium packed inside the injection needle. This technique provides simplified operation, and no special devices are required for either desorption of the enriched compounds or extraction using solvent.
The NeedlEx, containing about 1 mL of aspirated nitrogen gas, is attached to the Luer-lock type gas tight syringe, and the needle is simply inserted into the GC sample injection port as in a typical gas sample injection operation. The compounds are thermally desorbed and then injected with the nitrogen gas into the GC column.

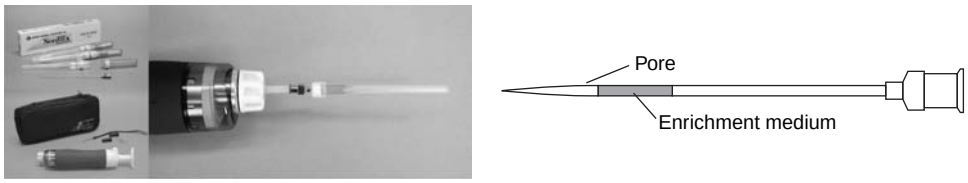

Fig.1 Kitagawa Gas Sampler and NeedlEx

■ Analysis of Organic Solvent

Chromatograms obtained from analysis of 8 compounds, including ethyl acetate, methyl isobutyl ketone, toluene, isobutanol, p-, m-, and o-xylene and styrene were compared. In the left chromatogram, a 1 mL aliquot of a 1 ppm gas sample was injected directly into the GC (Fig. 2). In the right chromatogram, 30 mL of an aspirated sample enriched using the NeedlEX was introduced into the GC (Fig. 2).
When analyzing samples using NeedlEX, the GC injection port temperature (sample desorption temperature) is set to 200 ˚C. The sample is injected into the GC by attaching the NeedlEx to the gas-tight syringe containing 1 mL of aspirated nitrogen gas, and then inserting the syringe needle into the sample injection port. After waiting about 10 seconds for the enriched constituents to be desorbed by the heat of the sample injection port, nitrogen is injected to introduce the constituents into the column. When the aspirated volume is increased from 10 mL to 50 mL, the enrichment volume and peak area values show good linearity (Fig. 3). Since the sampling limit differs according to the analyte, refer to the analytical dynamic ranges (sample concentrations and sampling volumes) listed in the instruction manual provided with the NeedlEx for the maximum sampling volumes.

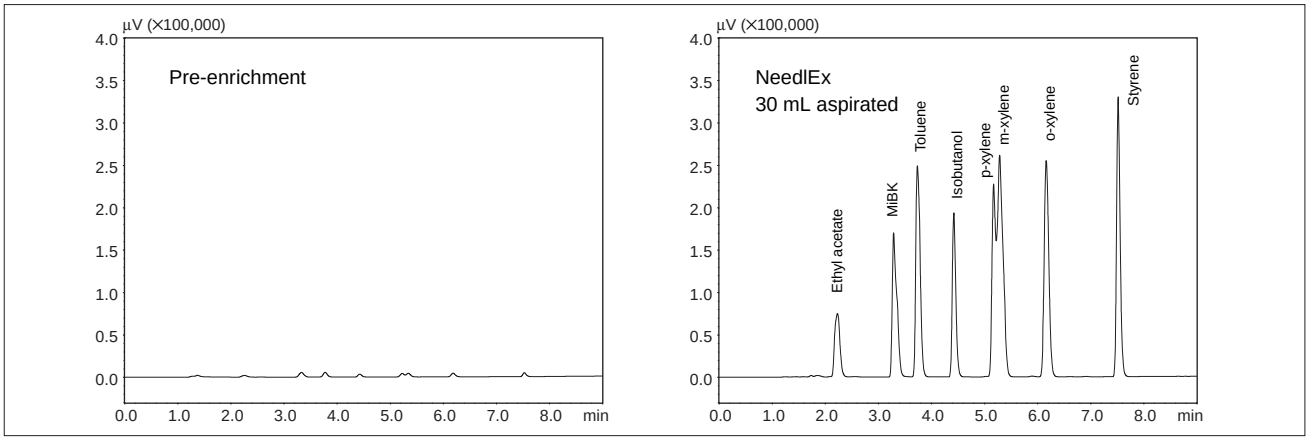

Fig.2 Analysis of Organic Solvents

Table 1 Analytical Conditions for Organic Solvents

Instrument	: GC-2014	Injection Method	: Direct Injection (WBC attachment)
Column	: DB-WAX (60 m × 0.53 mm I.D. df=1.00 μm)	Detector	: 250 ˚C FID-2014
Column Temp.	: 60 ˚C - 5 ˚C/min - 110 ˚C		H_2 : 40 mL/min Air : 400 mL/min
Carrier Gas	: He 15 mL (6 min) - 5 mL/min- 25 mL	Desorption Gas Volume	: 1 mL (N_2)
Injection Port	: 200 ˚C		

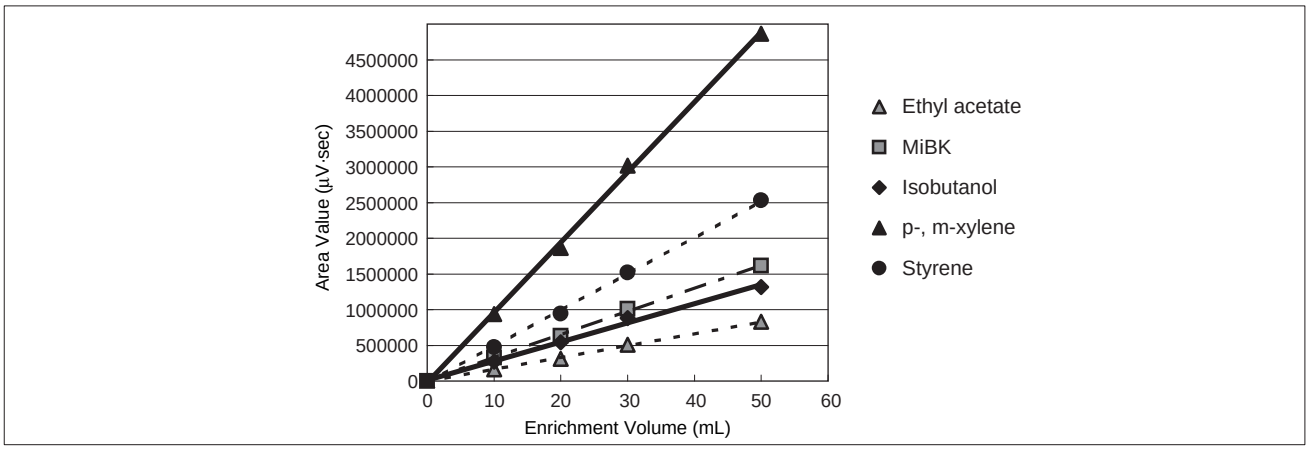

Fig.3 Linearity of Enrichment Volume

■ Analysis of Trimethylamine

Chromatograms obtained when directly injecting 1 ppm trimethylamine / 1 mL nitrogen standard gas, and when using the NeedlEx with a 100 mL enrichment volume were compared (Fig. 4).
In analysis of trimethylamine, the injection port temperature is set to 250 ˚C for desorption of the enriched sample. The desorption wait time is the same as that used in the analysis of organic solvents, about 10 sec, and 1 mL of nitrogen gas is similarly used for expelling the enriched components.
It is also possible to enrich monomethylamine and dimethylamine with NeedlEx for trimethylamine enrichment.

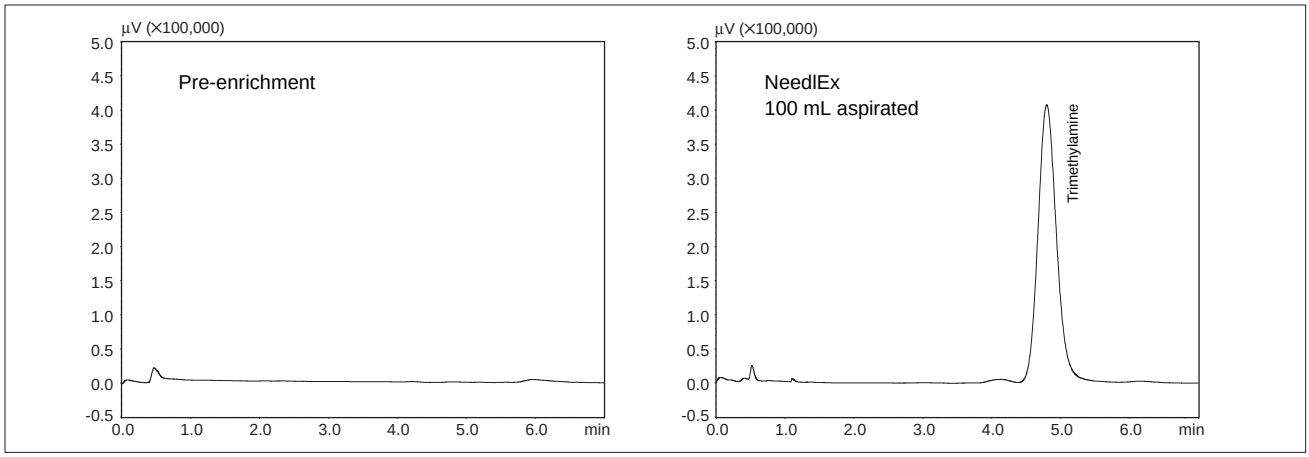

Fig.4 Analysis of Trimethylamine

Table 2 Analytical Conditions for Trimethylamine

Instrument	: GC-2014	Injection Port	: 250 ˚C
Column	: Thermon-3000 + KOH (2+2) % Sunpak-N 3.2 mm × 2 m	Detector	: 250 ˚C FID-2014 H_2 : 40 mL/min Air : 400 mL/min
Column Temp.	: 120 ˚C	Desorption Gas Volume	: 1 mL (N_2)
Carrier Gas	: He 40 mL/min		

■ NeedlEx Series

P/N	Product Description	Qty
GLC501-110	NeedlEx for organic solvent enrichment	3 pc
GLC501-120	NeedlEx for trimethylamine enrichment	3 pc
GLC501-130	NeedlEx for fatty acid enrichment	3 pc

Accessories

P/N	Product Description
501-210	Kitagawa gas sampler AP-20 (50, 100 mL sampling volumes)
501-220	Kitagawa gas sampler AP-20N (10, 50, 100 mL sampling volumes)
008025	SGE Luer-lock type gas-tight syringe 1 mL

Handled by Shimadzu GLC Ltd.

NOTES:
✻This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.

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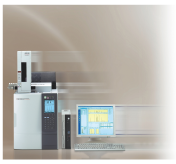

GC-2014

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