Detection and Identification of Trace components in the Air Using smartGC-infiTOF

Keywords：Environment, gas analysis, trace impurities

Overview

- Using our GC-MS “smartGC-infiTOF,” unknown impurities in an air sample were unexpectedly detected during trace gas analysis and were subsequently identified.
- The identity of the unknown impurity, which could not be determined using the EI mass spectrum or retention time, was elucidated using the high-resolution and high mass-accuracy capabilities of the “infiTOF.”
- Data shows our “smartGC-infiTOF” could be useful for measuring trace impurities in gaseous samples.

Introduction

For trace impurity analysis of gas samples, conventional detection methods use GC and GC-MS. When unknown impurities are detected, identification is usually attempted in the following manner:

1) Search for a matching retention time in a database that compares parameters such as column type and length, flow rate, and temperature.
2) Measure a standard gas sample of the suspected substance under the same conditions and compare the retention time and mass spectrum against the unknown substance.
3) Estimate the identity using the mass spectral fragment pattern.

Here, we report on using the high-resolution/high mass accuracy of the infiTOF to identify the unknown impurity measured in an air sample by our smartGC-infiTOF system.

Experimental

We installed a packed column into our smartGC and connected to our infiTOF inlet. One mL samples were introduced into and separated using the packed column and then introduced into the infiTOF ion source. Samples were measured in both low (1 lap) and high (20 laps) resolution (detailed measurement conditions are listed in Table 1).

Table. 1 Analysis conditions for air samples

Method Parameters	
Instrument	smartGC (MS-SG-02) infiTOF (MS-UHV-Pro)
Column	1/8inch -SUS tube "ShincarbonST" packing Size: 1.6 mm i.d. X 1.5 m
Column Temp.	150 °C
Carrier Gas	He
Flow Rate	20 cc/min
Ionization Energy	23 eV
infiTOF # of Laps	Low res: 1; High res: 20

Results and Discussion

Fig. 1(a) shows the mass chromatogram for Kr in the air sample when measured in low-resolution mode and Fig. 1(b) shows the mass chromatogram for the unexpected unknown impurity observed at *m/z* 64. Compared to Kr, the unknown elutes earlier, has a wider peak width, and poorer S/N.

We used the high-resolution mode of the infiTOF to measure *m/z* 64 in order to attempt to identify the unknown compound using the high mass accuracy of the instrument.

Fig. 2(a) is the mass spectrum near *m/z* 64 when measured in low-resolution (1 lap) and Fig. 2(b) shows the result of high-resolution measurement (20 laps). With high resolution, the peak width decreased and the mass accuracy improved dramatically compared to the low-resolution measurement. The precise mass of the peak near *m/z* 64 was assigned as *m/z* 63.9622.

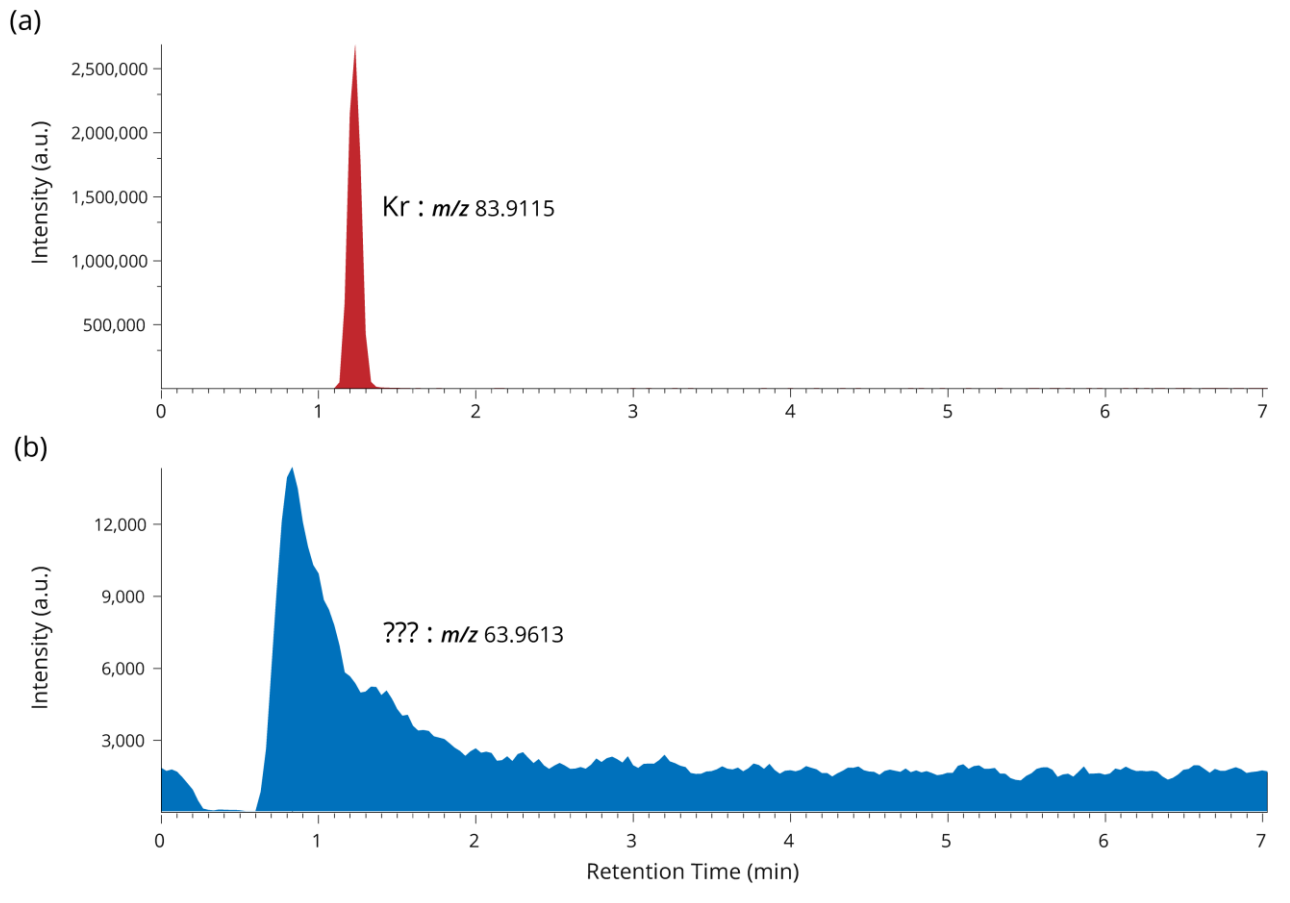

Fig. 1 Low-resolution mass chromatograms for (a) Kr and (b) unknown substance near *m/z* 64

The assigned mass was used in a software-based search for possible substances based on composition. Search criteria were limited to substances containing C/H/O/N/S/P and a mass of 62.9622 ± 0.003 (see Table 2). Two possible types of compounds were returned by the search: SO_2 and P_2H_2 (see Table 3). The unknown impurity was identified as SO_2 by taking into account binding mode, probably of existence, mass error, etc.

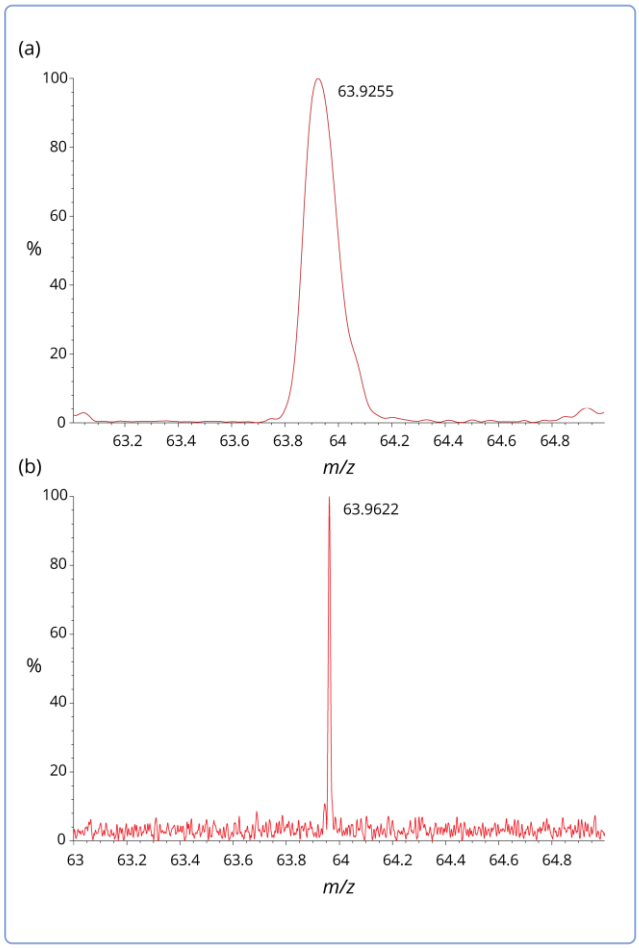

Fig.2 Mass spectra near *m/z* 64 using (a) low-resolution and (b) high-resolution modes

Conclusion

We conducted trace analysis of gases in air using our smartGC-infiTOF system and reported a method for identifying unknown impurities using the high-resolution and high mass-accuracy capabilities of the infiTOF. The identity of the unknown impurity could not be determined by retention time or mass spectral fragmentation patterns, however, by comparing the accurate mass from the infiTOF data to a software database with chemical composition abilities, the identity of the unknown impurity was elucidated.

Based on our results, the smartGC + infiTOF system could be useful for determining the identity of unexpected unknown impurities in gaseous samples using low- and high-resolution modes and accurate mass data.

Table 2 Parameters used in compound search by composition

Selected Isotopes	$H_{0\text{-}10}C_{0\text{-}5}O_{0\text{-}2}N_{0\text{-}4}S_{0\text{-}2}P_{0\text{-}2}$
Error Limit [u]	0.003
Measured Mass	63.96224

Table 3 Results of compound search by composition

Formula	SO_2	P_2H_2
Calculated Mass	63.96190	63.96318
Error [u]	0.00034	-0.00094

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