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Development of Ball SAW Gas Chromatograph with Preconcentrator for Analysis of Multiple Hazardous Gases
多種類の有害ガス分析のための濃縮器を備えたボール SAW ガスクロマトグラフの開発

Takamitsu Iwaya[1†], Shingo Akao[1], Kazushi Yamanaka[1], Tatsuhiro Okano[1], Nobuo Takeda[1], Yusuke Tsukahara[1], Toru Oizumi[1], Hideyuki Fukushi[1], Maki Sugawara[1], Toshihiro Tsuji[1], Tomoki Tanaka[1], Ryoko Hiraoka[1], Akinobu Takeda[1], Asuka Shima[2], Satoshi Matsumoto[2], Haruna Sugahara[2], Takeshi Hoshino[2], and Tetsuya Sakashita[2]
([1]Ball Wave Inc.; [2]JAXA)
岩谷隆光 [1†]，赤尾慎吾 [1]，山中一司 [1]，岡野達広 [1]，竹田宣生 [1]，塚原祐輔 [1]，大泉透 [1]，福士秀幸 [1]，菅原真希 [1]，辻俊宏 [1]，田中智樹 [1]，平岡領子 [1]，武田昭信 [1]，島明日香 [2]，松本聡 [2]，菅原春菜 [2]，星野健 [2]，坂下哲也 [2] ([1] ボールウェーブ, [2] 宇宙航空研究開発機構)

1. Introduction

On-site analysis of multiple volatile organic compounds (VOC) has diverse needs. For example, monitoring hazardous gases for the safety of workers in factories and laboratories, early detection of diseases by biogas analysis in the medical field, and food quality control by analyzing odorants of foods are expected. Gas chromatograph (GC) is useful for such analysis, but conventional GCs are too large for on-site analysis. We have developed a portable ball surface acoustic wave (SAW) GC [1-3] using ball SAW sensor [4,5] where the SAW makes multiple roundtrips on a spherical piezoelectric crystal. In addition, as a result of the development of a preconcentrator (PC) that traps VOCs and injects them to GC by thermal desorption, it became possible to analyze VOCs at ppbv levels [6].

In this study, as a part of the examination of the usage of the portable ball SAW GC equipped with a PC, which was developed for the analysis of space related gases, we analyzed multiple hazardous gases in the working environment on the earth.

2. Ball SAW GC with preconcentrator

Fig. 1 shows a schematic diagram of a ball SAW GC equipped with a PC that uses the forward flush (FF) method [3] for the short time analysis of multiple gases. This system consists of a PC [6] which concentrates and injects sample gas, two columns with different retention force (CL1, CL2), and two ball SAW sensors (BS1, BS2). First, gases A to G are injected at the state where CL1-BS1-CL2-BS2 are serially connected (Fig. 1(a)). While gases D to G are retained in CL1, gases A to C, which have weaker retention to CL1, are detected by BS1, nondestructively. Next, the valves are switched to connect CL1-BS1 and CL2-BS2 in parallel (Fig. 1(b)). Gases D to G remaining in CL1 are separated by CL1 and detected by BS1, and gases A to C are separated by CL2 and detected by BS2. In the following experiments, for the preliminary verification of the FF method, two types of columns CL1 or CL2 was used separately.

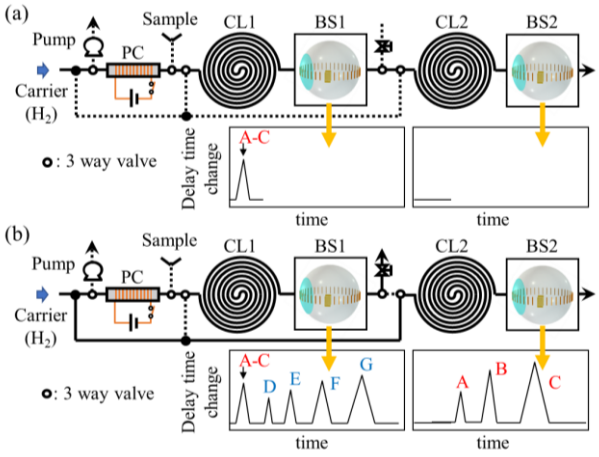

Fig. 1 Schematic diagram of a ball SAW GC equipped with a PC using forward flush method. (a) Serial connection. (b) Parallel connection.

3. Experiment

We analyzed multiple hazardous gases using the ball SAW GC. Sample was prepared by injecting 9 kinds of hazardous gases as 0.4 µl liquids into a gas bag with 1 L of nitrogen and vaporizing them in the bag. **Table 1** shows the concentrations of each component and their allowable concentrations in the working environment indicated by the Japan Society of Occupational Health [7]. The concentration of each sample gas was approximately 1/3 to 3 times the allowable concentration. In addition, the sample gas contained water vapor that was mixed in during preparation.

The sample gas was collected at 8 ml/min for 30 seconds into a PC filled with activated carbon. As CL1 and CL2, the fused-silica capillary columns with a length of 3 m and 30 m were used, coated with polydimethylsiloxane (PDMS) for non-polar gases and polyethylene glycol (PEG) for polar gas,

respectively. The column temperature was maintained at 40 °C for 10 minutes and raised at 10 °C/min using a desktop GC oven. We used two quartz ball SAW sensors (150 MHz) coated with poly-vinylpyridine (P4VP) which has high sensitivities to polar gases and polyepichlorohydrin (PECH) which has sensitivities to non-polar gases.

Table. 1 Target and maximum allowable concentrations for components of sample gases.

	Gas	Concentration of sample gas (ppmv)	Allowable concentration (ppmv) [7]
1	Acetone	132	200
2	Ethyl acetate	99	200
3	Methanol	240	200
4	Methyl ethyl ketone	106	200
5	2-Propanol	126	400
6	Dichloromethane	152	50
7	Butanol	106	50
8	Toluene	92	50
9	(o, m, p-)Xylene	79	50

4. Result and discussion

Fig. 2 shows chromatograms of nine mixed gases separated by column CL1 and detected by the two ball SAW sensors. While gases 1-6 shown in Table 1 were not separated from the background gas nitrogen and water, butanol (7), toluene (8), and xylene (9) three isomers were clearly detected as separated peaks. The difference in response magnitude was due to that in the interaction between the gas molecules with the sensitive film materials.

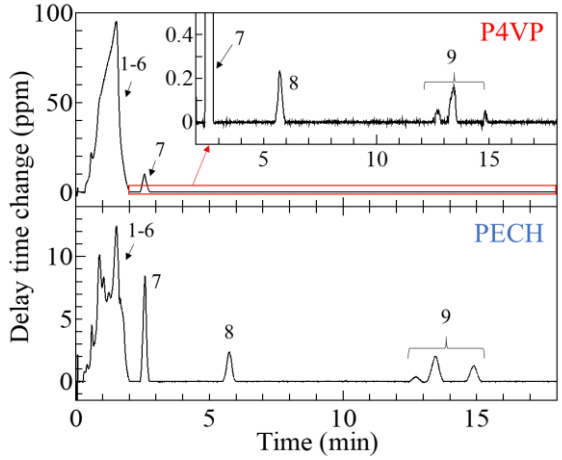

Fig. 2 Chromatogram of multiple hazardous gases separated by column CL1.

Fig. 3 shows the chromatograms of six mixed gases that were not separated in Fig. 2 separated using column CL2. The gases 1 to 6 were separated from the background nitrogen and water and detected by both P4VP and PECH coated sensors.

Since each gas was clearly detected by only 30 seconds of collection, it is assumed that these gases can be sufficiently detected even if their concentrations were lower than allowable concentrations. If the FF method using these columns CL1 and CL2 is applied, the chromatograms of Fig. 2 and Fig. 3 can be obtained in one measurement, and it can be expected to realize the short-time analysis of 9 kinds of gases.

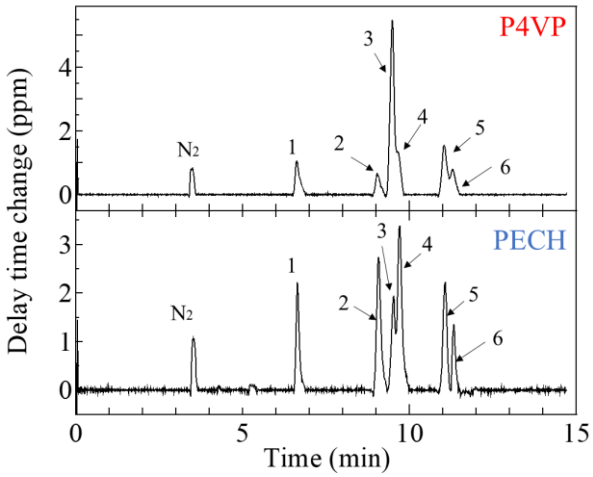

Fig. 3 Chromatogram of multiple hazardous gases separated by column CL2.

5. Conclusion

We succeeded in separating and detecting 9 kinds of hazardous gas in the working environment by ball SAW GC equipped with PC. Therefore, we found that the ball SAW GC can be useful for on-site analysis of hazardous gases in the working environment.

Acknowledgment

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iwaya@ballwave.jp