

Ball SAW Gas Chromatograph for Atmospheric Monitoring in Crewed Space Environment Using Air Carrier Gas

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1. Introduction

In a closed environment such as spacecraft, monitoring of trace volatile compounds in the atmosphere is necessary to maintain the health of the crews and to prevent malfunctions of onboard equipment. On the International Space Station (ISS), (1) compounds that are frequently detected at relatively high concentration (e.g., acetone and ethanol), (2) compounds harmful to the human body at low concentrations (e.g., benzene), and (3) compounds that may adversely affect the life support systems (e.g., siloxanes) are being analyzed.¹⁾

Gas chromatograph (GC) is effective in analyzing such a wide variety of gases. Currently, two compact GCs with the weight of approximately 3 kg, each equipped with one gas separation column of a different type, are used for atmospheric monitoring on the ISS.²⁾ In this situation, we have developed a 1 liter sized GC with the weight of approximately 1 kg using ball surface acoustic wave (SAW) sensors³⁾ that utilizes multiple roundtrips of the SAW on a spherical element. This GC system implemented the forward flush method⁴⁾ that uses two different columns to analyze a wide range of gases that are difficult to separate with a single column. It had succeeded in separating and detecting multiple gases that correspond to target compounds (1) and (2).⁵⁾ On the other hand, siloxanes, which corresponds to (3), have never been analyzed by the Ball SAW GC.

In addition, since this ball SAW GC uses a hydrogen storage canister as a carrier gas supply, the canister needs to be replaced periodically. Therefore, we are developing a new prototype GC that uses recirculated air as the carrier gas in order to eliminate the need for canister replacement. In this paper, as a basic experiment for GC development using air carrier gas, analysis of a siloxane mixture and verification using nitrogen as a carrier gas to simulate an air carrier were conducted using a single-column ball SAW GC.

2. Design of ball SAW GC equipped with recirculation system

Fig.1 shows the flow path configuration of a ball SAW GC system (part of which is in conception) that uses recirculated air as the carrier gas. The system is equipped with two different stationary phase columns (CL1, CL2) and two ball SAW sensors (BS1,

BS2). The sample gas is pumped and trapped in a pre-concentrator (PC), then injected into CL1 when the PC is heated in serial connection as shown in Fig. 1(a). Next, the valve is switched to the parallel connection shown in Fig. 1(b), and the compounds not separated in CL1 are separated in CL2 and detected in BS2. The compounds remaining in CL1 are separated in CL1 and detected in BS1. After the detector, the carrier gas is pumped through the adsorbent to remove detected compounds and impurities and is recirculated.

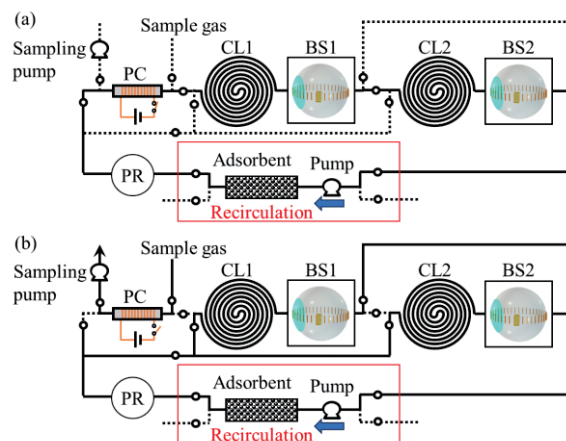


Fig. 1 Schematic diagram of a portable GC using recirculated air as the carrier gas.

(a) Serial connection. (b) Parallel connection.

3. Analysis of siloxane mixture

A single column ball SAW GC was used to analyze a mixture of cyclic and chain siloxane gases. The solid sample at room temperature was dissolved in acetone, while the liquid sample at room temperature was directly injected into a gasbag and vaporized to prepare the sample gas mixture. The set concentrations of each component and the spacecraft maximum allowable concentrations (SMACs)⁶⁾ for a 180-day stay in a spacecraft are shown in **Table 1**.

The gas mixture was collected in a concentrator (PC) packed with Tenax TA by a GC pump at 20 ml. The column was a 30 m long metal capillary column (UltraALLOY®) with a stationary phase of 5% diphenyl-95% poly-dimethyl siloxane (PDMS). The column temperature was held at 50 °C for 2 min and then increased to 180 °C at 10 °C/min. The ball SAW sensor was a quartz ball (150 MHz) with PDMS deposited as the sensitive film. Note that hydrogen carrier gas was used in this experiment.

Analytical result is shown in **Fig. 2**. Each component was successfully separated and detected with good S/N, indicating that it is possible to detect concentrations sufficiently lower than SMACs.

Table. 1 Each concentration of siloxane mixture.

Components	Concentration (ppmv)	180 days SMACs (ppmv)
Hexamethylcyclotrisiloxane (D3)	1.06	1
Octamethylcyclotetrasiloxane (D4)	0.39	1
Decamethylcyclopentasiloxane (D5)	0.12	1
Dodecamethylcyclohexasiloxane (D6)	0.05	-
Hexamethyldisiloxane (M2)	5.31	-
Octamethyltrisiloxane (M3)	0.31	-
Decamethyltetrasiloxane (M4)	0.12	-
Dodecamethylpentasiloxane (M5)	0.10	-

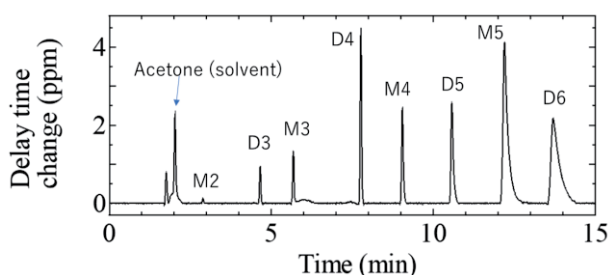


Fig. 2 Chromatogram of siloxane mixture.

4. Experiments simulating air carrier gas

The same system as in Chapter 3 was used to verify the differences between hydrogen carrier gas and nitrogen carrier gas which simulates air carrier gas. The sample gases were mixtures of five volatile organic compounds (VOCs) shown in **Table 2**. For analysis using hydrogen carrier gas, a hydrogen storage canister was used to supply the carrier gas. 10 ml of sample gas was collected, and the other conditions were the same as in the experiments in Chapter 3. In the experiment using nitrogen carrier gas, the carrier gas was supplied by a nitrogen cylinder, and the carrier gas pressure was adjusted so that the retention time was the same as in the hydrogen carrier gas experiment.

The results of the experiment are shown in **Fig. 3**. The width of each peak in the chromatogram of the nitrogen carrier gas shown in Fig. 3(b) was larger than that of the chromatogram using the hydrogen carrier gas in Fig. 3(a). This is due to the effect that the column separation performance deteriorates more markedly with respect to the carrier gas flow rate in nitrogen because the diffusion coefficient of the compound is smaller in nitrogen. The full width of half maximum of each peak increased up to 1.9 times for heptane relative to the chromatogram of the hydrogen carrier gas, but there was no problem with adjacent peaks overlapping each other. The S/N was more than 10, indicating that the analysis was possible with sufficiently lower detection limit concentrations than SMACs.

Table. 2 Each concentration of VOC mixture.

Components	Concentration (ppmv)	180 days SMACs (ppmv)
n-Heptane (C7)	50	3
Toluene (T)	30	4
n-Octane (C8)	20	3
m-Xylene (m-X)	20	8.5
n-Nonane (C9)	10	3

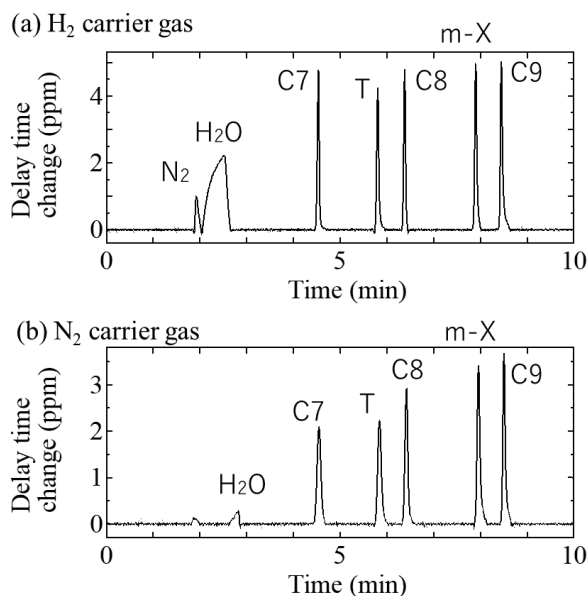


Fig. 3 Comparison of carrier gas. (a)Hydrogen. (b)Nitrogen.

5. Conclusion

From these experiments, it was confirmed that siloxanes can be analyzed using the ball SAW GC and that an air carrier gas can be used, although its peak widths were larger than that of the hydrogen carrier gas. These results indicate the feasibility of a portable GC using air as a carrier gas for crewed space environment monitoring.

Acknowledgment

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Referents

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