COMPARISON OF SOLVENT CUT LARGE VOLUME (SCLV) INJECTION SYSTEM WITH CONVENTIONAL TECHNIQUE IN DIOXINS ANALYSIS BY HRGC-HRMS

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Introduction

An SCLV injection system has been developed as a quantitative technique on the scale of a few femtograms per microlitre[1,2]. It is advantageous in that it allows a large volume injection without GC injector modification, and it can be carried out under high vacuum because of its narrow column. This system should therefore contribute to improving column noise level and resolution. There has been a few information regarding different types of capillary columns. The aim of this study is to compare the SCLV injection system with the conventional technique for analyzing dioxins by HRGC-HRMS using cyanopropyl phase capillary columns.

Methods and Materials

All HRGC-HRMS analysis was conducted on a 6890 series GC (Agilent Technology, USA) equipped with Autospec-Ultima (Micromass, UK). The SCLV injection system (SGE, Australia) was equipped with a BPX-5 (5 m×0.25 mm×0.25 μm) capillary column (SGE, Australia) as the pre-column and an Rtx-2330 (40 m×0.18 mm×0.10 μm) capillary column (Restek, USA) as the analytical column. Either 4 μL (SCLV injection system) or 1 μL (conventional technique) of waste gas of dioxins extract in nonane was injected. For the conventional technique, an SP-2331 (60 m×0.32 mm×0.20 μm) capillary column (Supelco, USA) was used, and the dioxin extract was analyzed according to Japanese industrial standard[3]. The analytical conditions for the SCLV injection system are shown in Table 1.

Table 1. Analytical conditions for the SCLV injection system

Injector temperature	300ºC
Oven temperature	80ºC→20ºC/min→260ºC (8min)→40ºC/min→180ºC (1min)→ 5ºC/min→260ºC (14min)
Injector pressure	300kPa (5min)→388kPa/min→688kPa (11min)→388kPa/min→ 300kPa (2min)→3.5kPa/min→398kPa (12min)
Purge on time	5 min
Cold trap	3-10min cooling
Solvent cut	5-17min solvent cut valve: off
MS Resolution	>10000
Number of channels	20
Cycle time	780 ms

Results and Discussion

Chromatograms of the dioxin extract were compared between the SCLV injection system and the conventional technique. The chromatograms of TeCDD, TeCDF, PeCDD, and PeCDF are presented in Figures 1-4, respectively; (A) stands for the use of the SCLV injection system, and (B) for the use of the conventional technique. These comparisons demonstrate excellent agreement between the two methods. The peak width of 2,3,7,8-TeCDD in the chromatogram for the SCLV injection system was about 7 seconds, while that for the conventional technique was about 14 seconds. As a result, the resolution of the chromatogram obtained using SCLV injection system was the same as that of the conventional technique. It is considered that the cycle time per scan must be set shorter in SCLV injection system than in the conventional technique.

Table 2 summarizes the ratios of native peak area to $^{13}C_{12}$-labeled internal standard peak area. The data between the two techniques agreed by 98-109 %. All PCDD/F analytes were eluted within about 48 minutes with the SCLV injection system, whereas elution took about 51 minutes with the conventional technique. Though the SCLV injection system needs time for operation of the solvent cut valve and so on, its analysis time was shorter than with the conventional method because of the use of a cold trap and a short, narrow column. When a standard was injected using the SCLV injection system, 0.8pg of 2,3,7,8-TeCDD was detected with S/N=650. This indicates that SCLV injection system has greater sensitivity than the conventional method, with which 2.0pg of 2,3,7,8-TeCDD was detected with S/N=250. From these results, it is concluded that the SCLV injection system is effective for determining low-level dioxins.

Table 2. Comparison of peak-area ratios

	(A) SCLV Injection System	(B) Conventional technique	A/B (%)
2378-TeCDD	0.526	0.500	105
2378-TeCDF	3.29	3.34	99
12378-PeCDD	1.50	1.46	103
12378-PeCDF	5.45	5.39	101
123478-HxCDD	0.676	0.660	102
123478-HxCDF	3.50	3.37	104
1234678-HpCDD	2.02	2.01	101
1234678-HpCDF	4.78	4.86	98
OCDD	1.44	1.36	106
OCDF	1.17	1.08	109

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References

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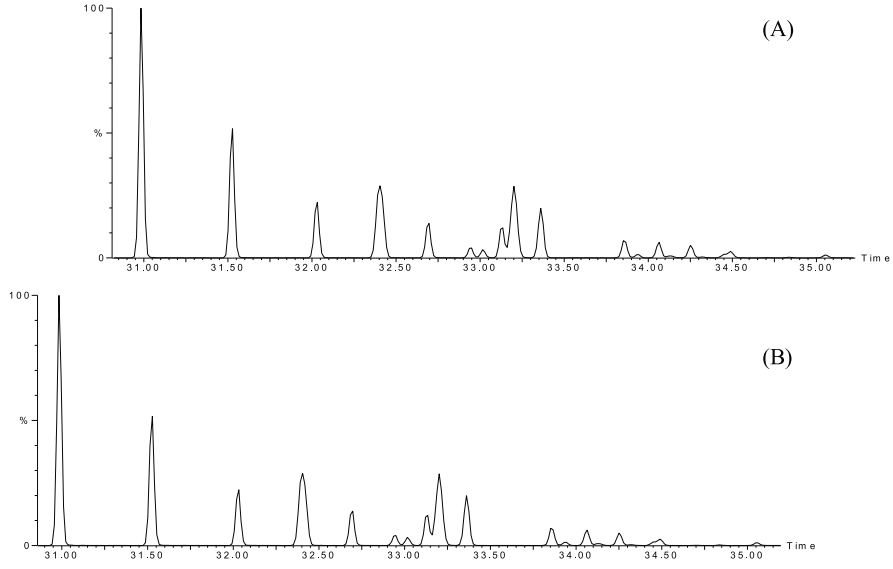

Figure 1. Chromatograms of TeCDD (A) SCLV injection system (B) conventional technique

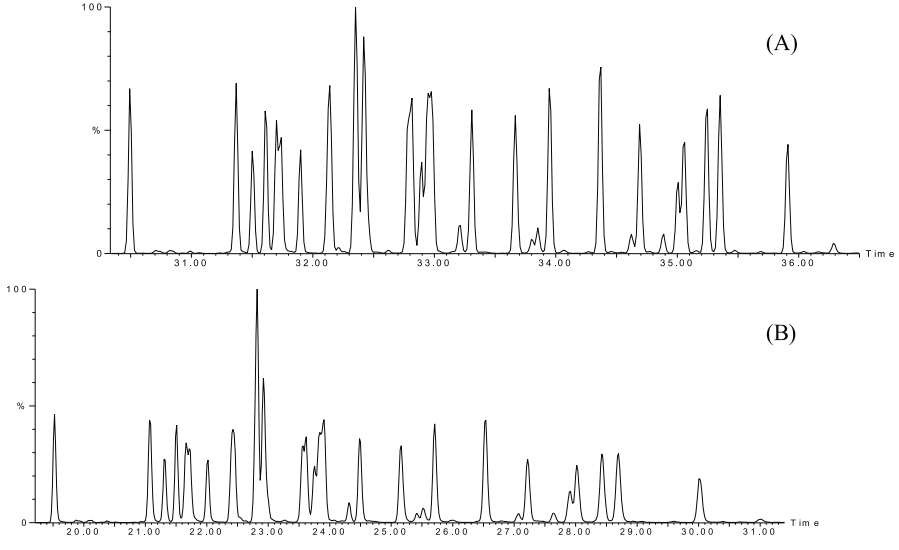

Figure 2. Chromatograms of TeCDF (A) SCLV injection system (B) conventional technique

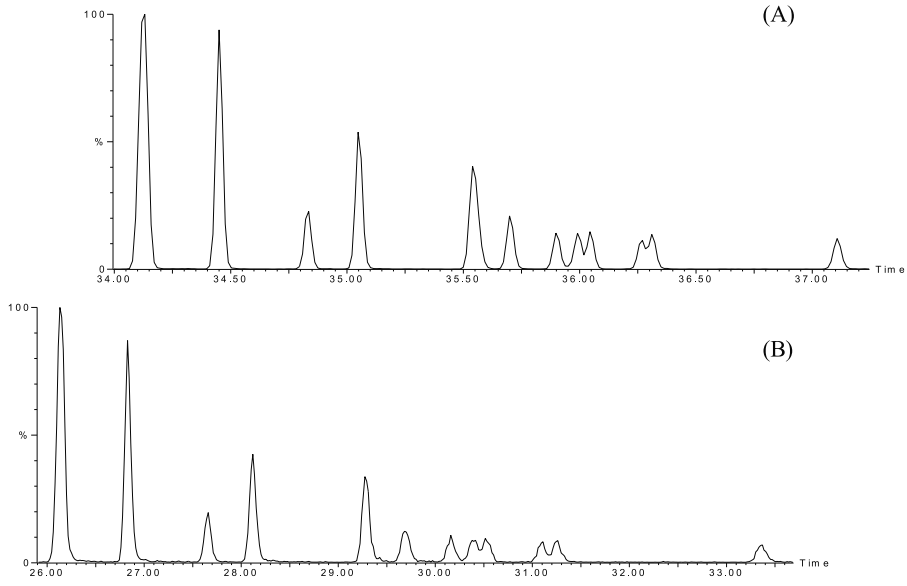

Figure 3. Chromatograms of PeCDD (A) SCLV injection system (B) conventional technique

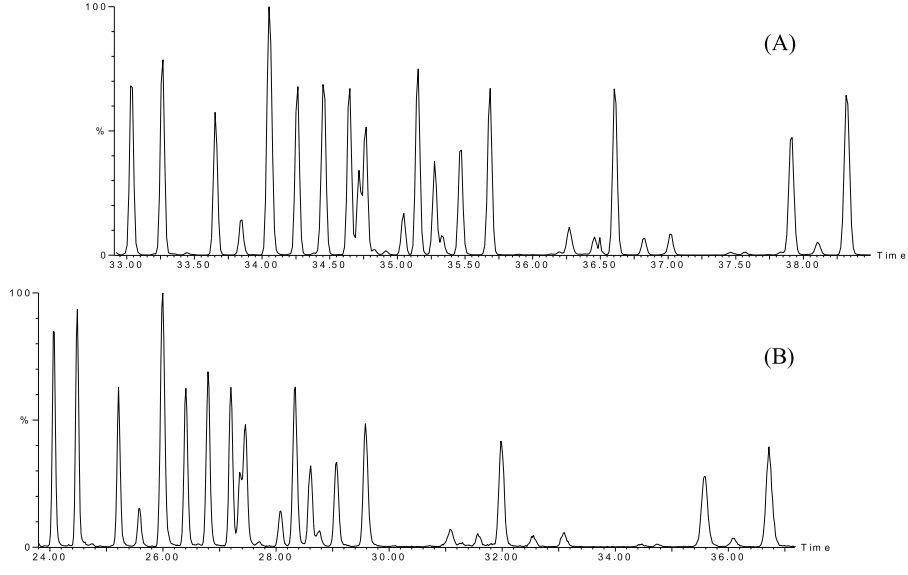

Figure 4. Chromatograms of PeCDF (A) SCLV injection system (B) conventional technique