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Autosampler of Ultimately Reduced Carryover for HPLC Equipped with an Ultrasonic Cleaning Device

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Ultrasonic cleaning is a rapid way to eliminate carryover peaks in HPLC, based on a cleaning mechanism different from that used in conventional autosamplers. Intensities of carryover peaks were remarkably decreased with the technique.

As detection sensitivity in high performance liquid chromatography (HPLC) has remarkably increased, mainly due to the recent interfacing technologies with mass spectrometry, carryover, or a memory effect, from previous sample runs began being often discussed (1–3). It is becoming a serious problem, especially in the field of pharmacokinetics and clinical analysis, where concentrations of unknown samples often range widely. In this study, an attempt to install an ultrasonic cleaning device to an autosampler for HPLC is described.

Experimental Conditions

An HPLC system was constructed with components of NANOSPACE SI-2 series (Shiseido, Tokyo, Japan), including an HTS autosampler Z 3133 equipped with an ultrasonic cleaning device (Figure 1). EzChrom Elite software for Shiseido (Tokyo, Japan) was used as an instrument controller and data system. Chlorhexidine (Sigma-Aldrich, Japan) was used to evaluate a level of carryover. Chlorhexidine was run with conditions; column, CAPCELL PAK C18 MG 5 μm (Shiseido), 2 mm i.d. × 150 mm, mobile phase: 100 mM $NaClO_4$ and 10 mM $NH_4H_2PO_4$ (pH 2.6) in H_2O/CH_3CN (55/45), flow rate: 0.2 mL/min, column oven temperature: 40 °C, injection volume: 2 μL; detection: UV at 260 nm.

Results and Discussion

Figure 2 shows carryover peaks of chlorhexidine, obtained with and without an ultrasonic cleaning process involved. The use of ultrasonic cleaning lowered the carryover value to 1/80th. These carryover peaks were observed after running a large amount of the compound (1200 μg/mL × 2mL, corresponding to 2.4 μg). The percentage values in the figure were calculated by dividing amounts determined with a calibration curve by the value of 2.4 μg. The ultrasonic cleaning was carried out as follows. After collecting a sample solution from a sample vial, the needle was first dipped into the pre-wash port, and thereafter, dipped into the ultrasonic cleaning port with the vibration applied for 10 s. The carryover value of 0.0007% corresponded to less than a half the amount the authors previously reported with a different cleaning strategy (4). Besides the performance to minimize a carryover level, a remarkably shortened time seems to be an additional advantage in the technique.

References

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(3) J.W. Dolan, *LCGC*, **19**(10), 1050 (2001).
(4) Shirota O, *Proceedings of Pittcon 2003*; 2530–2610.

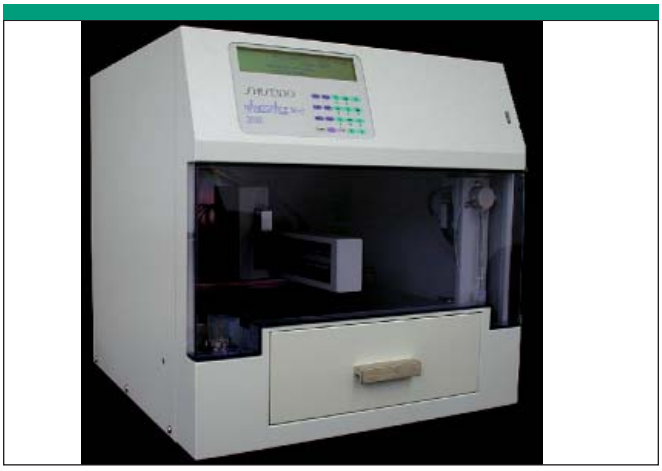

Figure 1: HTS autosampler Z 3133 equipped with ultrasonic cleaning device.

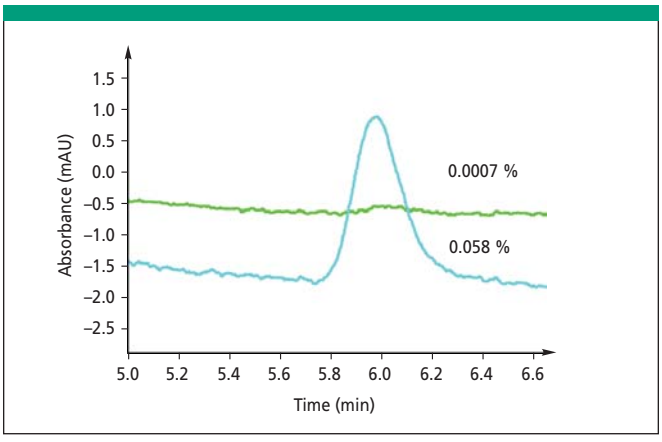

Figure 2: Effect of ultrasonic cleaning on carryover of chlorhexidine. Carryover peak (top) with ultrasonic cleaning and (bottom) without it.

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