

Evaluation of Effectiveness of Dedicated High Durability Column for Use with Py-Screener™ Ver.2

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User Benefits

- ◆ Integrated Guard extends column life and reduces the maintenance burden and running costs.
- ◆ Integrated Guard eliminates the worries of leakage, and adsorption and decomposition of components at the connection site.
- ◆ Even if peak tailing occurs, the peak shape can be improved by partially cutting off the guard column.

■ Introduction

The RoHS Directive in the EU designates phthalate esters and brominated flame retardants (polybrominated biphenyls (PBBs), polybrominated diphenyl ethers (PBDEs)) as regulated compounds and restricts their use in components of electrical and electronic products. Py-Screener Ver. 2 is a simultaneous screening system for phthalate esters, PBBs, and PBDEs which enables simple inspection merely by cutting a small piece of the test sample with a cutter and placing it in a sample cup. Since the release of the former product, Py-Screener Ver. 1, the Py-Screener series has been used by many customers, such as manufacturers of electrical and electronic equipment.

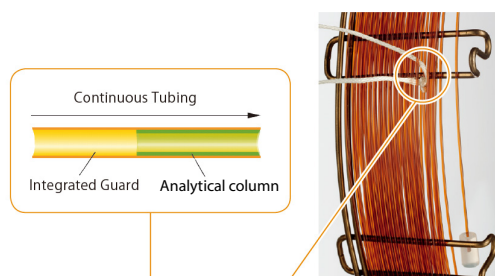
On the other hand, some customers who conduct tests frequently expressed a desire for longer life of the analysis column because use of a column in which peak tailing occurs due to column deterioration may possibly affect the quantitation accuracy and repeatability of inspections.

To solve this problem, Py-Screener Ver. 2 supports inspections with a newly-developed high durability column (SH-1MS with guard column, P/N: 227-36346-01, Shimadzu Corporation). The column has excellent resistance to deterioration and reduces the maintenance burden and running costs.

This article introduces the durability of the column and the effect of maintenance when peak tailing occurs.

■ Structure and Features of High Durability Column

Fig. 1 shows the structure of the high durability column. The column includes a 2 m guard column on the GC injection side and can limit column deterioration by preventing contaminants from reaching the analytical column. Normally, a guard column is connected to the analytical column using a connecting component such as a press-tight connector or a micro union, but this column has a structure (Integrated Guard) in which the guard column and the analytical column are continuous. This means that this column needs no connectors. Therefore, there is no need to worry about leaks and adsorption due to the use of connectors.



Possible to distinguish the guard column section and analytical column (indicated by string)

Fig. 1 Structure of High Durability Column

■ Measurement Conditions and Evaluation Method

Table 1 shows the analysis conditions. Using a GCMS system in which a high durability column was installed, RoHS inspections of more than 700 samples of various parts, which were included in electrical and electronic products, were conducted by the simultaneous inspection method for phthalate esters and brominated flame retardants included in Py-Screener Ver. 2.

100 mg/kg phthalate ester polymer standard materials (P/N: 225-31003-91, Shimadzu Corporation) were inspected periodically, and the tailing factor of DBP (dibutyl phthalate) was calculated. (The tailing factor is an index to evaluate peak tailing. Larger tailing factors indicate peak tailing is larger, showing the deterioration of the column is worse.) The changes in the tailing factor of DBP with an increasing number of inspections were investigated, and the durability of the system was evaluated.

As an additional experiment, the column was intentionally contaminated to cause tailing, and the improvement of peak tailing by cutting off part of the guard column was investigated.

Table 1 Analysis Conditions

Instrument configuration	
GCMS	: GCMS-QP2020 NX
PY	: EGA/PY-3030D, AS-1020E (Frontier Laboratories Ltd.)
Software	: Py-Screener Ver. 2
Column	: SH-1MS with guard column (length: 17 m (including guard column: 2 m), inner diameter: 0.25 mm, film thickness: 0.10 μm)
PY conditions	
Pyrolyzer temp.	: 200 °C → 20 °C/min → 300 °C → 5 °C/min → 340 °C (1 min)
Interface temp.	: 300 °C
Carrier gas	: He
GC conditions	
Injection port temp.	: 300 °C
Column oven temp.	: 80 °C → 20 °C/min → 320 °C (4 min)
Carrier gas	: He
Control mode	: Constant linear velocity
Injection mode	: Split (1 : 50)
Linear velocity	: 52.1 cm/s
Purge flow rate	: 3 mL/min
MS conditions	
Interface temp.	: 320 °C
Ion source temp.	: 230 °C
Ionization method	: EI
Measurement mode	: Simultaneous Scan/SIM
Scan event time	: 0.15 s
Scan range	: m/z 50 – 1000
SIM event time	: 0.3 s

■ Evaluation of Durability

Fig. 2 shows the change of the tailing factor of DBP (100 mg/kg) as the number of inspections increased. Even after more than 700 inspections, the tailing factor was below the threshold value of 2.5, which indicates a condition in which peak tailing is occurring. Based on this result, no deterioration of the column was seen after more than 700 inspections. Since peak tailing occurs after around 300 to 400 in inspections using a conventional column without a guard column, durability was found to be at least 2 times greater than that of the conventional column.

(Note: Column deterioration may vary depending on the type of inspection sample.)

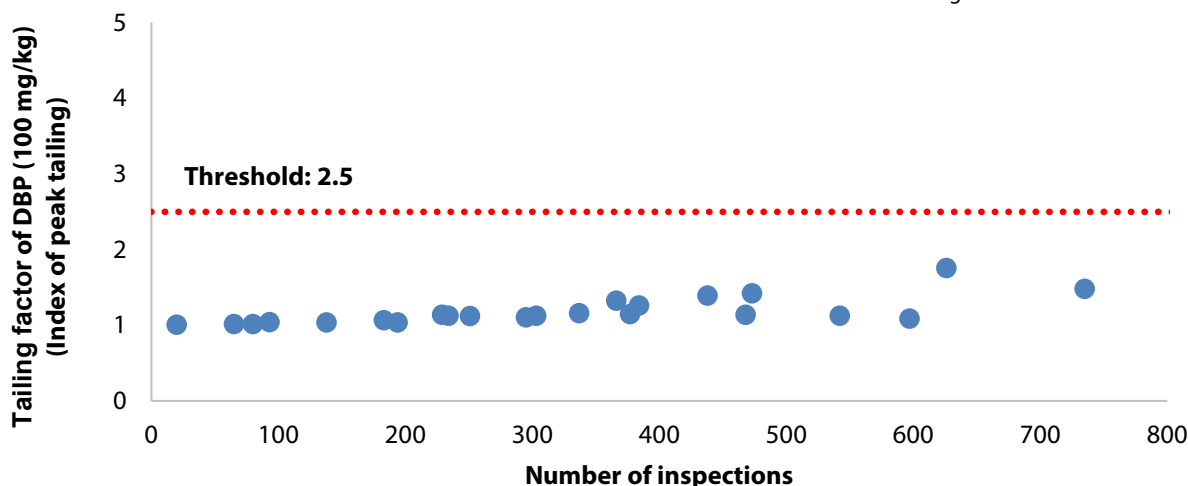


Fig. 2 Result of Evaluation of Durability in RoHS Inspections Using High Durability Column

■ Effect of Column Maintenance

After intentionally inducing the peak tailing of SH-1MS column, effective methods to maintain the column were explored. When 50 cm of the guard column of the SH-1MS column in which peak tailing is occurring by causing excessive contamination was cut off, the tailing factor of DBP (100 mg/kg) reduced from 3.72 to 1.38 and peak tailing was substantially eliminated (Fig. 3). It is believed that the pollutants accumulated in the guard column caused the peak tailing. By cutting off a part of the guard column, the pollutants can be removed and the performance of the column can be restored. Although the optimum cutting-off length will vary depending on the condition of the column, cutting off about 50 cm to 1 m of the guard column is considered effective for maintaining this column.

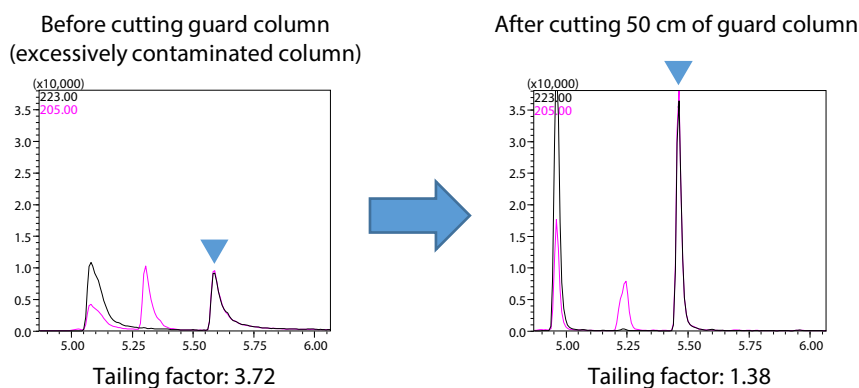


Fig. 3 Change of Peak Shape and Tailing Factor of DBP (100 mg/kg) by Cutting 50 cm of Guard Column

■ Conclusion

The high durability column for use with Py-Screener Ver. 2 is an integrated-type column in which the guard column and analytical column are integrated. This Integrated Guard is a structure that eliminates the need for guard column connection work and avoids leaks and adsorption of the target compound at the connection site. The results of the durability evaluation showed that this column can be used without maintenance after more than 700 RoHS inspections, achieving high durability at least about 2 times greater than that of the conventional column. Because the frequency of maintenance and the burden on workers are also reduced, equipment downtime for maintenance

can also be decreased.

This column is an effective solution for customers who must conduct inspections with a high frequency. In particular, even if peak tailing occurs due to continuous use, the condition of the column can be improved simply by cutting off part of the guard column. Since one column includes a 2 m guard column section, maintenance can be carried out 4 or more times, assuming 50 cm of the guard column is cut off at the time of maintenance. Moreover, each column has a long useful life, which also contributes to reduced running costs.

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