

SFC

Pressure Stability

of YMC-Actus

Semi Preparative Columns



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1. Introduction

Customers reported reduced stability of chiral and achiral 50 mm ID SFC columns from different suppliers. Under SFC conditions, they observed that the inlet frit is typically distorted after about 500 runs or less. As a consequence silica leakage occurred alongside the frits/column inlet fitting.

In order to confirm the durability of YMC columns for SFC the following was performed:

- Pressure testing under LC conditions for 80 hours at the maximum column pressure (240 ml/min, 200 bar)
- SFC pressure testing over 1,000 injections with performance monitoring every 100th injection under regular column usage.

2. Column tested

YMC-Actus SIL (Silica), 12 nm, 5 µm, 250 x 50 mm
SL12S05-2553AX

3. Summary

In order to monitor column stability under customer's usual SFC working conditions not only pressure of 120 bar (at column head) was applied. Also, 1,000 injections were applied every 1 minute with a pressure drop of 40 bar. The column performance was checked after every 100 injections.

The inlet frit was inherently stable under SFC conditions (195 mL/min, 120 bar at the column head). The column maintained its initial performance throughout the study.

These findings demonstrate that the column is stable not only in LC mode (corresponding to 480-2400 injections with 10 or 2 min runs, respectively), but also in SFC mode for a long term period.

4. Outline of the study

- Initial column performance test
 - ↳ Repeated injections (1,000 injections)
 - ↳ Column performance test (after every 100 injections)
 - ↳ Final column performance test
 - ↳ Frit observation

5. Results

5.1. Chromatographic conditions for repeated injections

Table 1: Chromatographic conditions

Mobile Phase	CO ₂ / methanol (135 / 60)
Flow Rate	195 mL/min
Pressure	120 bar (at column head)
Detection	254 nm
Temperature	30°C
Backpressure	100 bar
Sample	Methanol
Injection Volume	1 mL (inject every 1 min)

5.2. Chromatographic conditions for the standard test

Table 2: Chromatographic conditions

Mobile Phase	CO ₂ / methanol (135 / 60)
Flow Rate	195 mL/min
Pressure	120 bar (at column head)
Detection	254 nm
Temperature	30°C
Backpressure	100 bar
Sample	1. Toluene (5 µL/mL), 2. Caffeine (500 µg/mL)
Injection Volume	1 mL
Pressure drop	40 bar

5.3. Column performance test

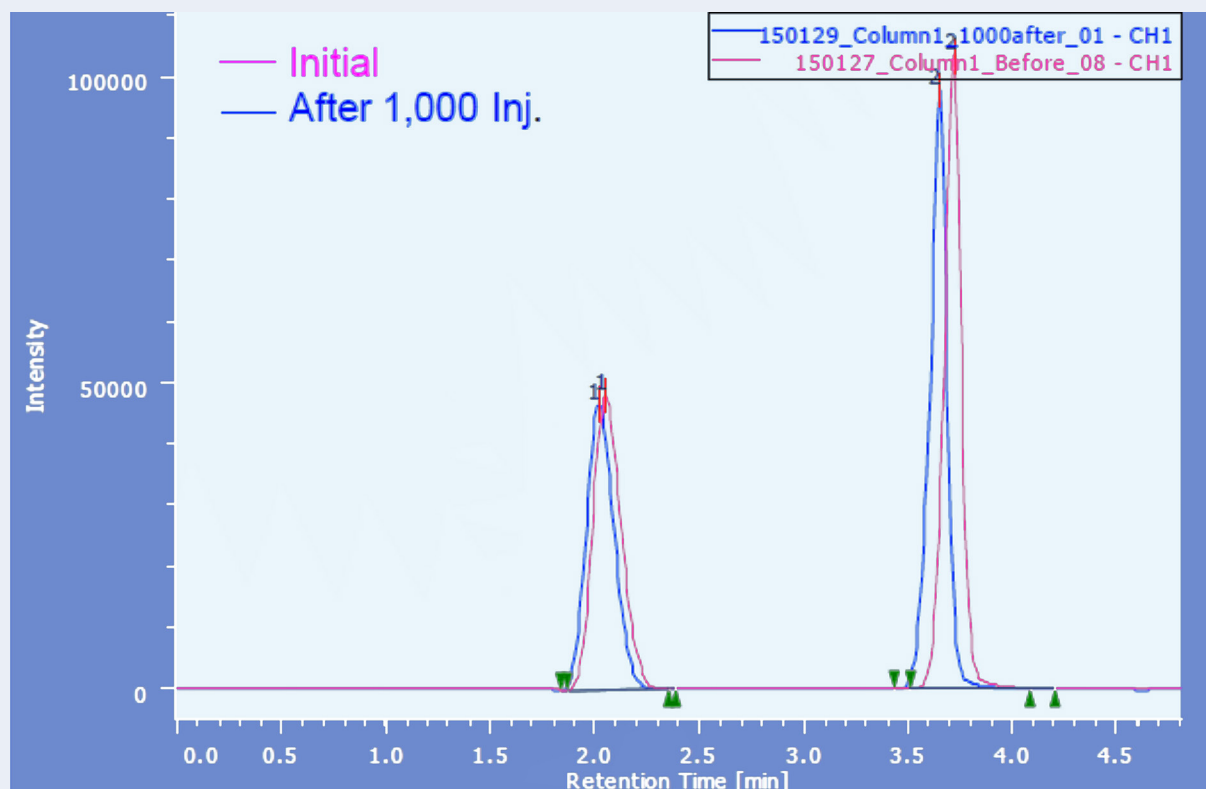


Figure 1: Initial test chromatogram and after 1,000 injections

No significant change in performance could be observed after 1,000 repeated injections (corresponding to about 17 h; fig. 1). Furthermore, no significant changes in retention time, theoretical plate count and tailing factor could be determined over the complete test period (fig. 2-4).

Product information



YMC-Actus
SFC Semi Prep.
Column Hardware

SFC Pressure Stability of YMC-Actus Semi Prep. Columns

Date: 10/08/2015

Author: Daniel Eßer

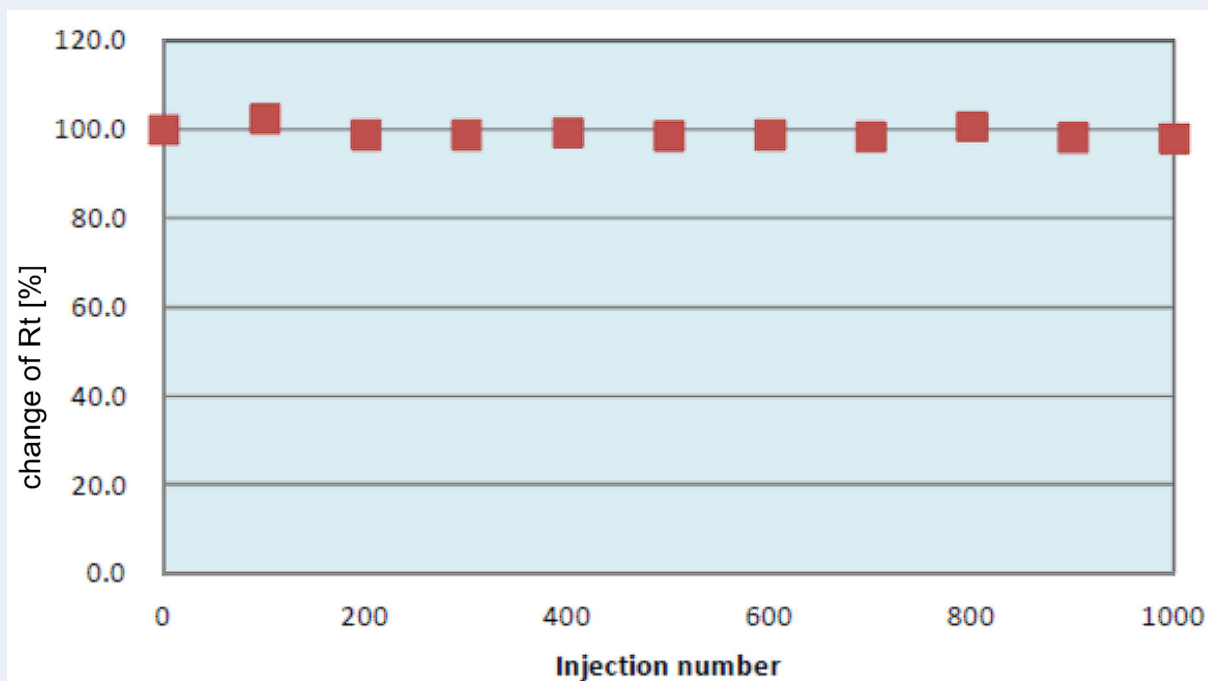


Figure 2: Retention time

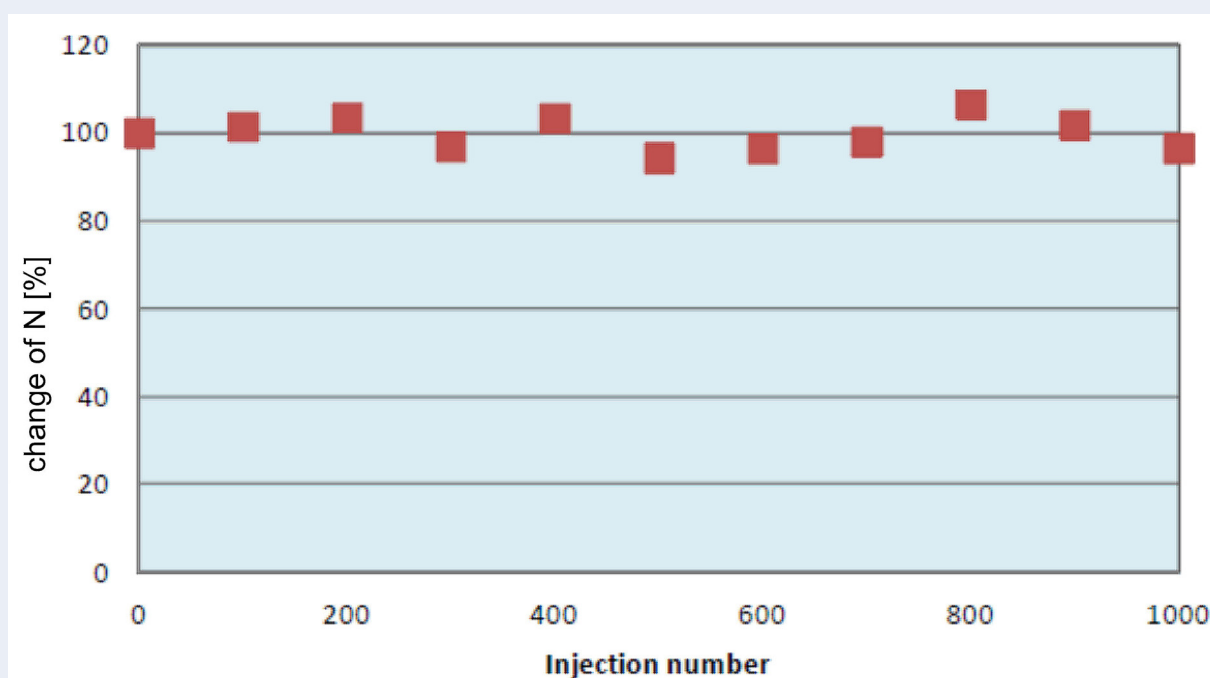


Figure 3: Theoretical plate number

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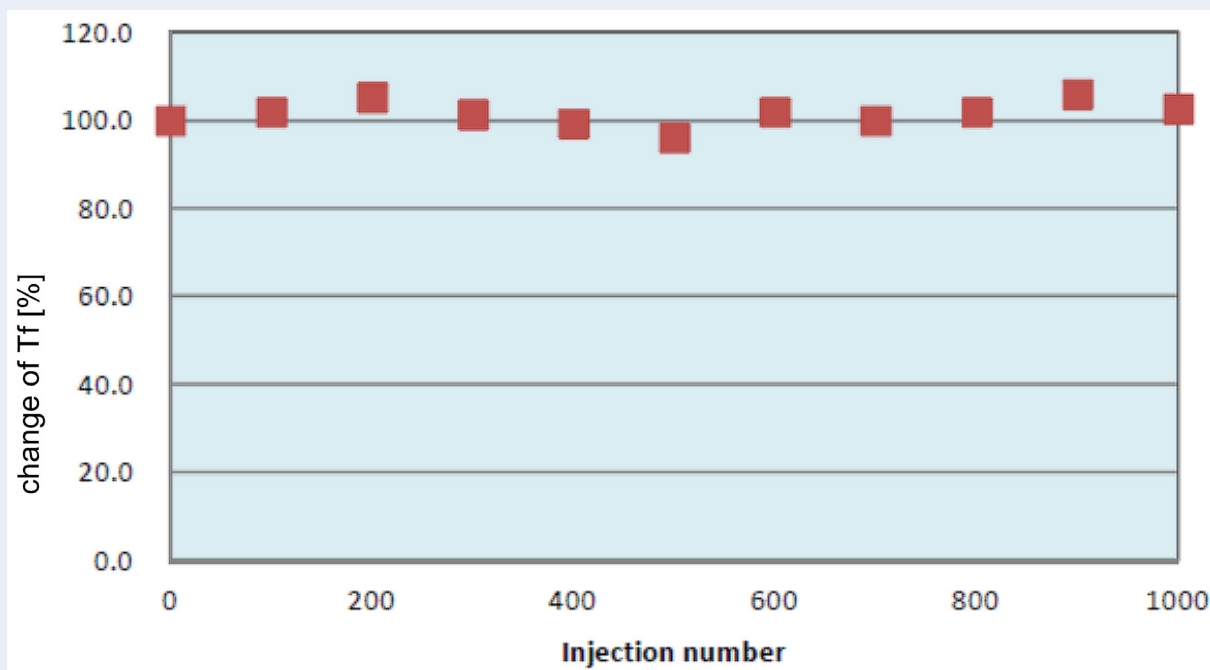


Figure 4: Tailing factor

The pressure at the column head was 120 bar throughout the test. Therefore, the total pressure was 210 bar. At injections the pressure drop was 40 bar.

5.4. Visual inspection after column evaluation



Figure 5: Open column with inlet frit after 1,000 injections

On opening the column after 1,000 injections, neither frit distortion nor gel leakage was observed.

6. Conclusions

LC mode

- ✓ A YMC-Actus semi preparative column was first evaluated at column maximum pressure under LC conditions for 80 hours.
- ✓ Inlet frit remains stable at 240 mL/min and 200 bar.
- ✓ No change in column performance was observed.

SFC mode

- ✓ A second evaluation under SFC conditions was performed for 1,000 injections with column performance test every 100 injection.
- ✓ The column performed fully reproducible throughout the whole test at a flow rate of 195 mL/min and pressure of 120 bar (at column head).
- ✓ Inlet frit remains unaffected and no leakage or distortion was observed.

➡ **Stability/reproducibility performance of YMC-Actus hardware is confirmed for SFC applications.**