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Technical Report

Increased Analysis Throughput by Overlapped Injection Using the SIL-40 series Autosampler
- Analytical Intelligence Part 6 -

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Abstract:

The use of high-speed analysis methods is now widespread because of improvements in separation efficiency due to the use of ultra-small particles in column packing materials and surface porous particles, and the associated higher pressure resistance and lower size of the instruments. However, to improve the overall analysis throughput, it is necessary to effectively utilize the sample injection time and the times when peaks are not eluted, periods outside the time for the chromatogram to become visible. The minimum injection time of the SIL-40 series autosampler (SIL-40) is 7 seconds or less, which is extremely effective for improving the throughput of the overall analysis. In addition, by performing a simple setting and injecting the sample for the next analysis during the current analysis (overlapped injection), the whole analysis cycle time be can be shortened and throughput increased. Here, we describe how to increase efficiency in size exclusion mode, where improving throughput is normally difficult, by utilizing overlapped injection.

Keywords: automatic pretreatment, overlapped injection, SIL-40 series, GPC

1. Overlapped Injection

The Nexera Series SIL-40 has not only a standard sample injection operation but also an automatic pretreatment function that is capable of a variety of operations, including overlapped injection.

Overlapped injection is a method of injecting the next sample while the immediately previous sample is being analyzed so that contaminant and solvent peaks do not overlap during the period that the target components are being eluted. This enables the analysis time for a continuous series of analyses to be shortened (Fig. 1). The longer the overlap time, the more efficiently the analysis can be performed.
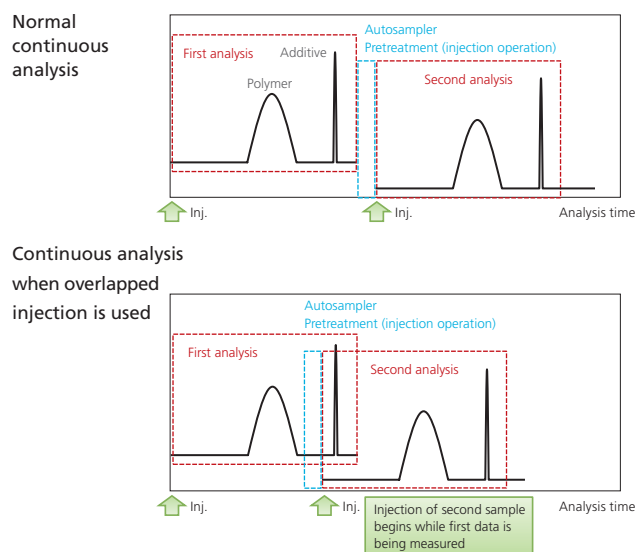

Fig. 1 Comparison of Normal Continuous Analysis and Analysis when Overlapped Injection is Used in Size Exclusion Mode

Overlapped injection achieves improved continuous analysis efficiency in modes such as size exclusion, ligand exchange, and ion exclusion, where adjustment of elution separation is almost impossible and high speed is difficult. However, because the next sample is injected while the previously injected sample is being analyzed, special attention must be paid to the following points when setting up the method.

- The separation mode must be isocratic
- The injection timing must be set up so that the target peaks to be detected in the next analysis do not cover any of the peaks in the previous analysis

2. Overlapped Injection Settings

Overlapped injection is set as shown in Fig. 2. Using the following chromatogram examples, the concepts for overlapping injection setting are introduced.

0 to 10 minutes: period when no components are eluted . . . (1)

10 to 30 minutes: period when components are eluted . . . (2)

Around 20 minutes: period when target components in (2) are not eluted . . . (3)

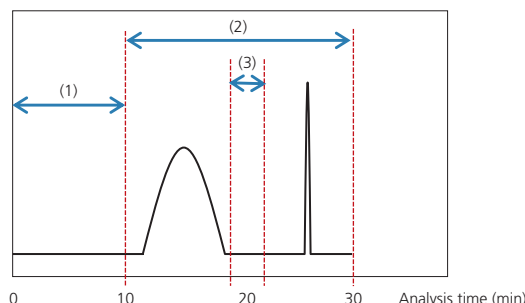Note: It is assumed that all components are eluted within 30 minutes.

Fig. 2 GPC Chromatogram Example

In Fig. 2, because no components are being eluted in period (1), there is no need to record any data, and this time is overlapped with the latter half of (2) of the immediately previous analysis.

To avoid any influence by the injection operation on the chromatogram, the injection operation for the second analysis is started at around 20 minutes (Fig. 2 (3))
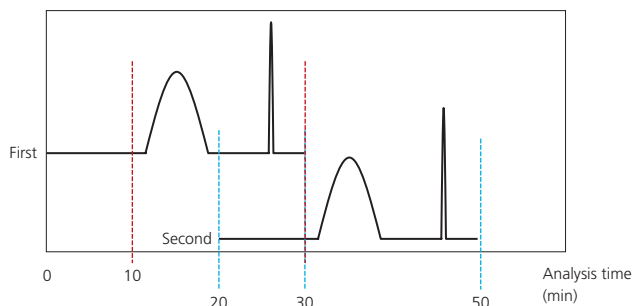

Fig. 3   Example of Successful Overlapped Injection

If overlapped injection is performed with this setting, then in the first analysis data is acquired in the 10 to 30 minute period. In the second analysis, no data is acquired in the period 20 to 30 minutes from the start of the first analysis, but data is acquired in the 30 to 50 minute period (Fig. 3). (The overlap time changes slightly due to the injection operation and data processing.)

On the other hand, if for example the second injection operation starts 15 minutes after the analysis begins, then the elution time of the polymer component in the second analysis and the time of the peak of the additive in the first analysis will overlap, so this setting cannot be used (Fig. 4).
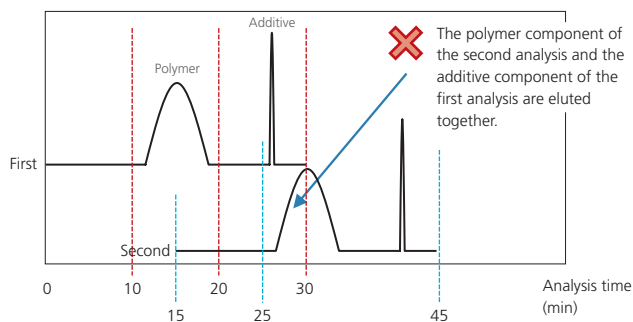

Fig. 4   Example of Unsuccessful Overlapped Injection

3. LabSolutions Setting Method

Overlapped injection can be set up easily from the LabSolutions analysis data system. The times determined in the procedure in Section 2 are entered into a method file as analysis conditions. The Fig. 2 (2) time is entered as [LC End Time] (Fig. 5).
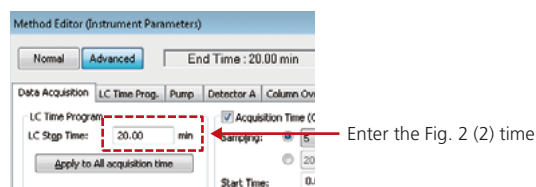

Fig. 5   Data Acquisition Time Setting

Next, select [Overlap] mode in the autosampler's pretreatment program setting window

Set the [Data processing time] to 0.5 min. (initial value), and enter the value of the difference between the time when data is not being acquired (Fig. 2 (1)) and the [Data processing time] (9.5 minutes in this example) as the [Overlap time].

For the [Pretreatment overlap time], enter the same value as the [Overlap time].

If the [Pretreatment overlap time] is too large, the first analysis data acquisition and the second analysis peak elution will overlap.
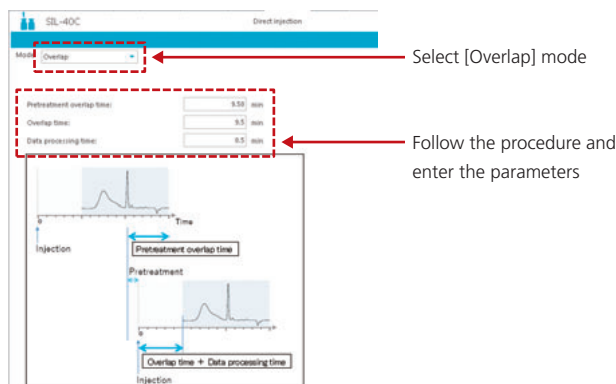

Fig. 6   Overlap Time Setting Input Window

4. Conclusion

- By using overlapped injection, analysis can be performed more efficiently even in analysis separation modes where throughput improvements are normally difficult, such as size exclusion, ligand exchange, and ion exclusion modes.
- The settings for overlapped injection can be easily set up from the LabSolutions control software.

References

Application News No. L537 (Increased Throughput with Nexera™ GPC system: Overlapped Injection and Simultaneous Determination of Polymer Additives)

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