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Technical Report

Fully Automated Workflow for HPLC Analysis Using Automatic Startup with FlowPilot Function

- Analytical Intelligence Part 3 -

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Abstract:

An appropriate start-up procedure, a warm-up of the LC system and a specific System Suitability Test (SST) are critical steps before any analytical LC session in order to ensure high data quality in terms of reproducibility, accuracy, etc., and to reduce maintenance costs (e.g. by prolonging the lifetime of analytical columns). These procedures are often time-consuming for operators, and, if not performed properly, can lead to the loss of data and the waste of time and resources due to the need for re-analysis. In this report, we explain the ways in which we have improved and completely automated system startup and SST through a combination of different technologies.

Keywords: Intelligent start-up, Intelligent shut-down, System Suitability Test, FlowPilot

1. Automation of Entire Analytical Procedures

The Nexera LC system is equipped with various technologies that allow enhanced automation of all routine operations within the analytical workflow.

Intelligent start-up includes both the FlowPilot function (See section 2). It can be coupled with the warm-up function and scheduled depending on the user requirements. The system can also be evaluated automatically using the automatic SST function (See section 4). Scheduled shutdown automatically turns off the system and switches it to power-saving mode when all analytical operations are complete.

The combination of these functions allows the user to fully automate an entire analytical cycle: Shutdown -> Start-up -> SST -> Analysis -> Results report -> Shutdown (Fig. 1).

2. Intelligent Start-up with FlowPilot

It is well known that pressure shock can affect column performance by reducing column lifetime and leading to channeling, which results in peak-splitting in the corresponding chromatogram.

In order to avoid this issue, operators usually need to start up the system by slowly increasing flow rate, waiting for column pressure to stabilize and finally setting the flow rate for analysis. Nexera solvent delivery units use the FlowPilot function to fully automate all these steps by synchronizing flow ramping and oven temperature stabilization, (Fig. 2):

1. Flow rate is set to 50% of the flow rate for analysis and the oven is turned on.
2. Flow rate remains constant until the column oven reaches the set temperature.
3. Flow rate starts increasing toward the set value once the column oven reaches the set temperature.

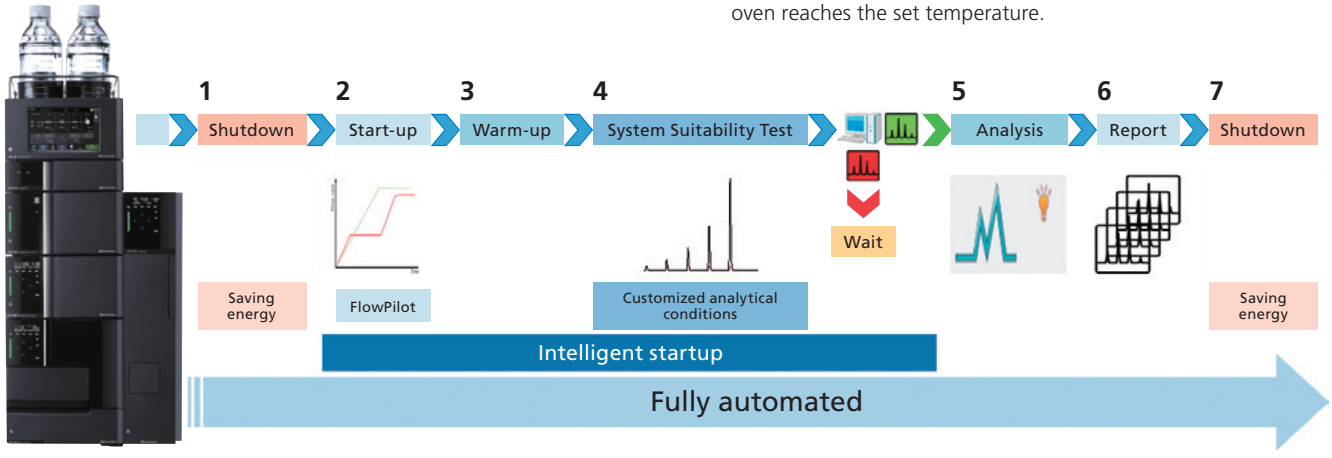

Fig. 1 Workflow diagram showing the fully-automated operation achievable with Nexera LC systems

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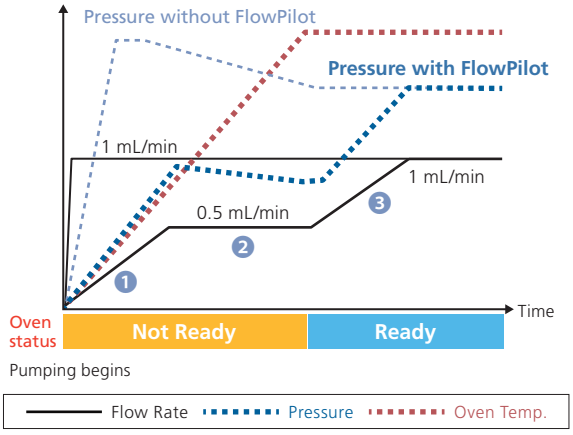

Fig. 2 Diagram of system pressure profile during start-up with the FlowPilot function

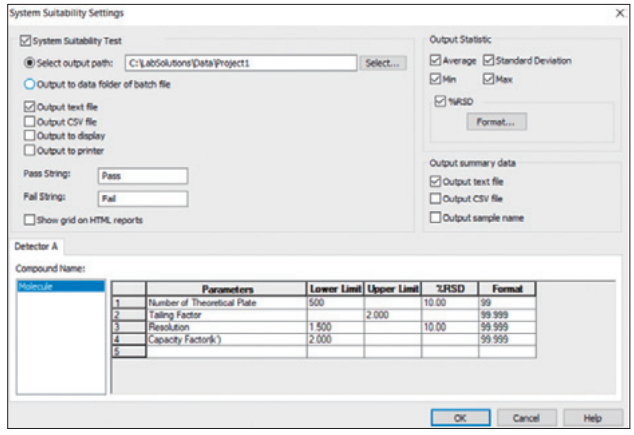

Fig. 3 System Suitability Test - example of 4 user-selected "pass/fail" criteria

3. System Suitability Test

SST are used to verify that the chromatography system is adequate for the intended analysis. The tests are based on the concept that the equipment, electronics, analytical operations, and samples analyzed constitute an integrated system that can be evaluated as such.

SST is mandatory in USP, FDA, and EP standards to check and ensure the ongoing performance of analytical systems. Nevertheless, several different parameters can be evaluated depending on the system and the analytical conditions. For this reason, there is a growing demand for a degree of flexibility in the set-up of SST parameters and possibilities for their customization in modern LC systems.

4. Fully-automated SST

SST parameters are embedded in the analytical method file. This means that users can easily create an SST with specific analytical conditions, in which selected parameters are evaluated (e.g. number of theoretical plates, tailing factor, resolution, capacity factor *k* ; see Fig. 3). After creating the SST, it is possible to choose when to run the SST during a batch analysis (at the beginning, after analysis of some samples or at the end of the batch).

Once the SST is complete, a "pass" or "fail" result is issued depending on the previously-selected criteria, and this result will then trigger specific actions based on user preferences (see Fig. 4).

Fig. 4 shows an example where the user has selected a batch composed of 4 samples for calibration and 7 unknown samples. By customizing SST parameters, it is possible to inject the SST sample after warm-up; in the case of a "pass" result, the analysis of the batch will continue with subsequent samples (both calibration and unknown samples).

In the case of a "fail" result, a blank is injected and the SST is repeated. A second "fail" will trigger the suspension of the batch processing and the instrument will be automatically put into standby mode. If the user has selected automatic shutdown, the instrument will be put into power-saving mode at the end of the batch.

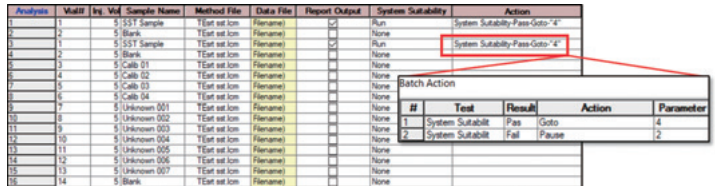

Fig. 4 System Suitability Test during batch analysis creation

5. Conclusions

- The Intelligent Start-up and FlowPilot functions can be used to automate routine procedures related to system start-up and warm-up prior to analysis.
- The user can select SST parameters for a method to fully automate the validation of a batch session, saving time and ensuring high reliability of analytical results.
- By using the shutdown function after sample analysis has been completed, a series of analysis workflows can be fully automated.

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