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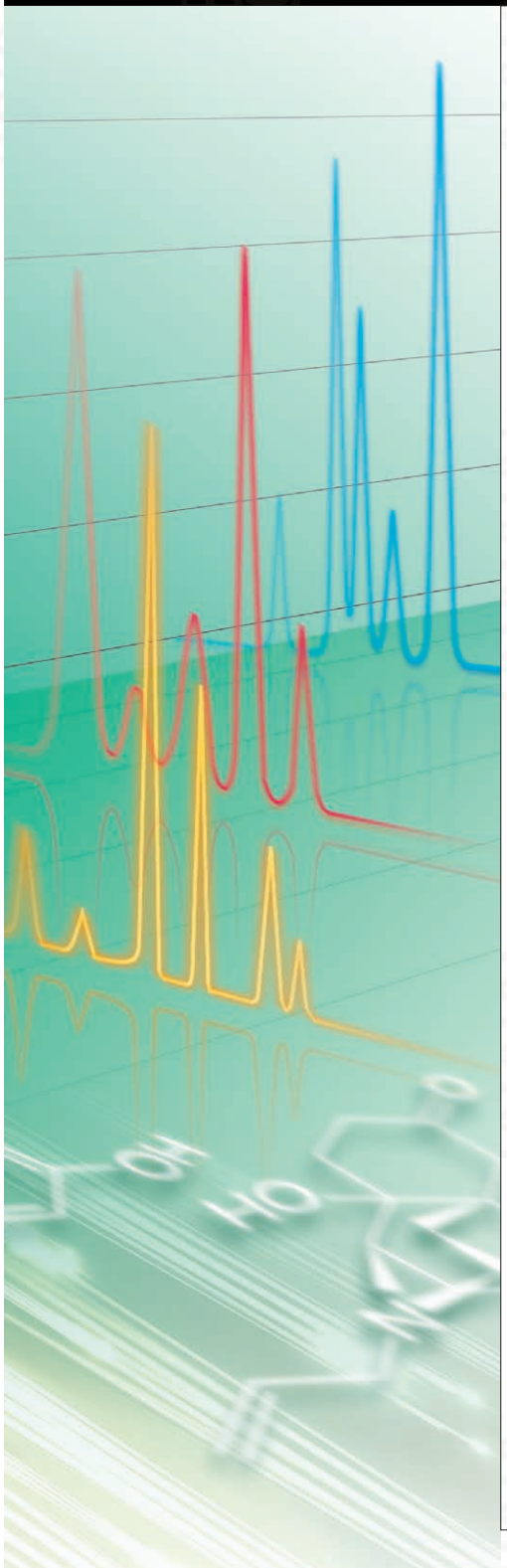
LabSolutions Batch Analysis – Bringing Greater Automation to Analysis Operations

Technical Report vol.38



1. Introduction

The first step in sample measurement is in the preparation for analysis at the analytical instrument itself. This ensures that the instrument is fully prepared to properly carry out measurement, and involves such tasks as verifying the use frequency of the instrument's consumable items, switching the flow line to the mobile phase to be used for analysis, pumping mobile phase according the conditions specified in the instrument method, and verifying baseline stability while monitoring the noise level and drift value. These measurement preparations associated with the instrument itself can be especially burdensome if analysis is to be conducted on multiple instruments.

LabSolutions is equipped with functions designed to automate analysis operations. These functions are set in a batch table and are implemented via the batch table when analysis is executed. This Technical Report describes the automation of analysis operations using the batch table functionality of LabSolutions. The practical significance of this is that the operator is now free from being stuck in front of the instrument while conducting tasks like checking the detector signal baseline stability. And, measurement of samples can now start with the assurance that the optimal instrument environment has been attained.

2. What Is "Automation of Analysis Operations?"

The typical analysis flow is shown in Fig. 1. The steps from switching on the power to the instrument and PC to the start of sample analysis are: (1) Switch the flow line to the mobile phase to be used for analysis. (2) Start oven temperature control according the analytical conditions, and start solvent delivery. (3) Check the state of field equilibration by monitoring the detector signal on the software baseline monitoring window until baseline stability is attained. (4) And finally, measure the control samples and or standard samples to conduct system suitability test (SST) and/or generate calibration curves. In addition, while conducting the above tasks, the use frequency of consumable items is checked, measurement samples are set in the autosampler rack, and a batch schedule is created. If the instrument is not working correctly, or if a calibration curve is not properly generated, the software-driven batch analysis sequence is stopped, and SST and/or measurement of the standard samples must be repeated. Thus, the operator must perform the above tasks to verify the readiness of the instrument for analysis. LabSolutions fully automates these tasks with its automated analysis operation functions, relieving the operator of such burdensome tasks.

Switch on power to the instrument and PC

↓ Verify operational state of analytical instrument (system check function)

↓ Mobile phase, system flow line solvent replacement (auto-purge function)

↓ Column equilibration (baseline check function)

↓ Measurement of standard sample and control sample (system suitability test, SST), calibration curve generation)

↓ Redirection of batch analysis according to SST results and calibration curve linearity judgment (sample re-injection, analysis halt, etc.)

↓ Measurement of unknown samples

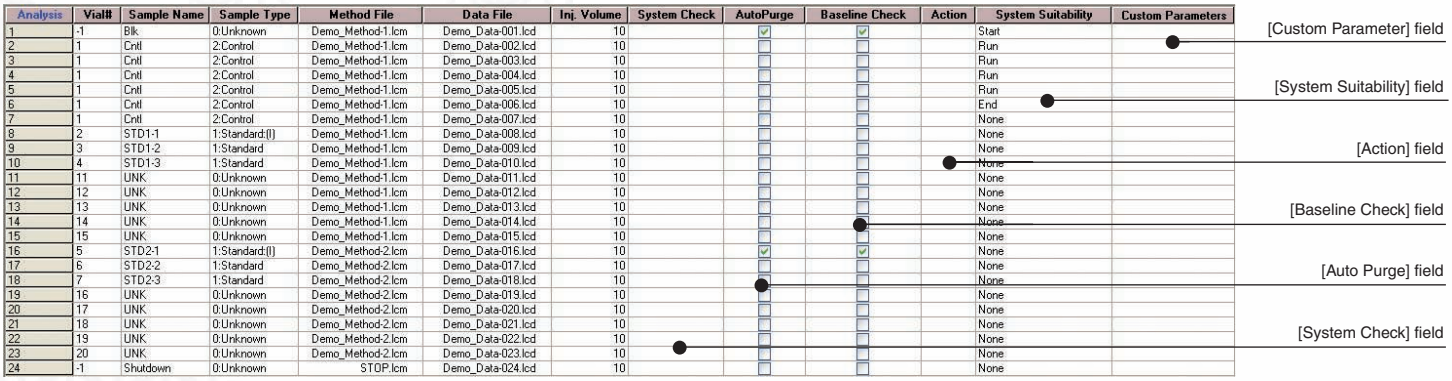
Instrument shutdown operations

Fig. 1 Analysis Operation Flow

3. Setting Up a Batch Table

Settings for automatic analysis are made using the batch table. Clicking the [Realtime Batch] icon on the [Main] assistant bar displays the [Batch Table] window (see Fig. 2). The batch table is constructed by setting the vial number and sample name for each sample, specifying whether the sample type is a standard or an unknown, the method file name, data file name and injection volume, etc. to be used for the analysis. Batch table items also relevant to analysis automation include the system check, auto-purge, baseline check, actions, system suitability and custom parameters.

In addition to the above, the batch table contains a variety of other functions, including output settings for individual reports and summary reports, background compensation settings used for blank data, etc. Customization of the batch table display itself is possible by selecting [Table Style] from the right-click menu which is accessed by right-clicking on the batch table. This allows displaying or hiding these additional functions in the batch table, as well as changing their position sequence in the table. Since a batch table is saved with its modified item content display and position sequence, the batch table display item settings can be customized according to the analytical objective.



Analysis	Vial#	Sample Name	Sample Type	Method File	Data File	Inj. Volume	System Check	AutoPurge	Baseline Check	Action	System Suitability	Custom Parameters
1	-1	Blk	0:Unknown	Demo_Method-1.lcm	Demo_Data-001.lcd	10		☑	☑		Start	
2	1	Cntl	2:Control	Demo_Method-1.lcm	Demo_Data-002.lcd	10		☐	☐		Run	
3	1	Cntl	2:Control	Demo_Method-1.lcm	Demo_Data-003.lcd	10		☐	☐		Run	
4	1	Cntl	2:Control	Demo_Method-1.lcm	Demo_Data-004.lcd	10		☐	☐		Run	
5	1	Cntl	2:Control	Demo_Method-1.lcm	Demo_Data-005.lcd	10		☐	☐		Run	
6	1	Cntl	2:Control	Demo_Method-1.lcm	Demo_Data-006.lcd	10		☐	☐		End	
7	1	Cntl	2:Control	Demo_Method-1.lcm	Demo_Data-007.lcd	10		☐	☐		None	
8	2	STD1-1	1:Standard:(I)	Demo_Method-1.lcm	Demo_Data-008.lcd	10		☐	☐		None	
9	3	STD1-2	1:Standard	Demo_Method-1.lcm	Demo_Data-009.lcd	10		☐	☐		None	
10	4	STD1-3	1:Standard	Demo_Method-1.lcm	Demo_Data-010.lcd	10		☐	☐		None	
11	11	UNK	0:Unknown	Demo_Method-1.lcm	Demo_Data-011.lcd	10		☐	☐		None	
12	12	UNK	0:Unknown	Demo_Method-1.lcm	Demo_Data-012.lcd	10		☐	☐		None	
13	13	UNK	0:Unknown	Demo_Method-1.lcm	Demo_Data-013.lcd	10		☐	☐		None	
14	14	UNK	0:Unknown	Demo_Method-1.lcm	Demo_Data-014.lcd	10		☐	☐		None	
15	15	UNK	0:Unknown	Demo_Method-1.lcm	Demo_Data-015.lcd	10		☐	☐		None	
16	5	STD2-1	1:Standard:(I)	Demo_Method-2.lcm	Demo_Data-016.lcd	10		☑	☑		None	
17	6	STD2-2	1:Standard	Demo_Method-2.lcm	Demo_Data-017.lcd	10		☐	☐		None	
18	7	STD2-3	1:Standard	Demo_Method-2.lcm	Demo_Data-018.lcd	10		☐	☐		None	
19	16	UNK	0:Unknown	Demo_Method-2.lcm	Demo_Data-019.lcd	10		☐	☐		None	
20	17	UNK	0:Unknown	Demo_Method-2.lcm	Demo_Data-020.lcd	10		☐	☐		None	
21	18	UNK	0:Unknown	Demo_Method-2.lcm	Demo_Data-021.lcd	10		☐	☐		None	
22	19	UNK	0:Unknown	Demo_Method-2.lcm	Demo_Data-022.lcd	10		☐	☐		None	
23	20	UNK	0:Unknown	Demo_Method-2.lcm	Demo_Data-023.lcd	10		☐	☐		None	
24	-1	Shutdown	0:Unknown	STOP.lcm	Demo_Data-024.lcd	10		☐	☐		None	

Fig. 2 Batch Table Editing Window

3-1. Auto-Purge Function

The auto-purge function refers to the automatic replacement of mobile phase in the flow line from the solvent bottle to the solvent delivery pump all the way to the sample injection valve of the autosampler. This auto-purge function can be set at any desired row in the LabSolutions batch table. In whatever row the checkbox is selected in the [Auto Purge] field, auto-purge is performed for the operation specified for that row. This functionality can offer greater efficiency, especially with a method development system designed for investigating the differences in compound elution positions and separation patterns. For example, by enabling auto-purge and baseline noise/drift assessment (baseline check function, see 3-2) in the batch row associated with the sample measured with specific analytical conditions (method file), batch analysis can be conducted without wasting time for setting an extra row in the batch table.

In addition, not only are there auto-purge-related settings for selecting the mobile phase replacement line and for the purge execution time, the purge can also be conducted using the initial mobile phase composition specified in the method file (analytical conditions, see Fig. 3).

However, in situations where the inlet tubing from the solvent bottle to the solvent delivery pump is filled with mobile phase, or the inlet tubing contains many bubbles, it may not be possible to adequately aspirate the replacement mobile phase or to remove the bubbles. In such a case, it is recommended to prevent these types of problems by opening the purge valve at the pump and conduct a manual purge prior to analysis.

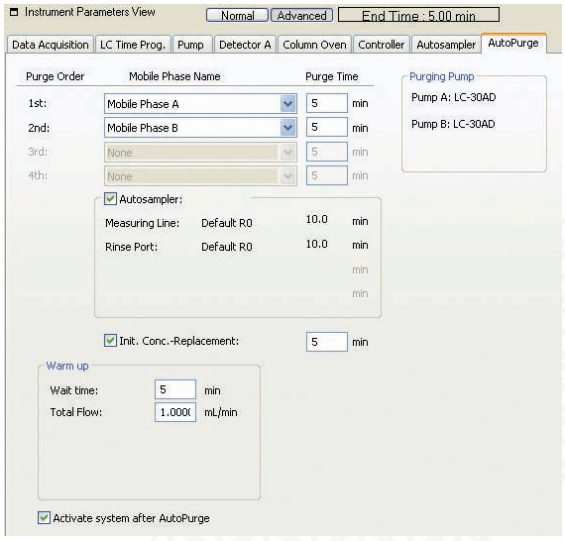


Fig. 3 Auto-Purge Setting Window (Instrument Method Setting Window)

3-2. Baseline Check Function

The baseline check function is used to verify the stability of the baseline. This involves setting a time range and threshold in the [Baseline Check] window in the method file for assessing the stability of the noise and drift values (see Fig. 4). The baseline check is executed based on these parameters by selecting the checkbox in the [Baseline Check] field of the desired row in the LabSolutionsbatch table. Typically, this baseline check is set together with auto-replacement of the mobile phase (auto-purge function, see 3-1), and it is effective when using the auto-start analysis function (analysis starts automatically when the baseline noise and drift values reach the threshold specified in the method) in measurement using a refractive index detector (RI detector), which typically takes a while to achieve baseline stability.

Selection of the calculation method for assessing the noise value, and setting of the detector channel(s) are also conducted in the [Baseline Check] window. Other settings include setting the extended period of time to continue analysis when the current baseline exceeds the threshold, and the action to be taken if a failing assessment is generated (interrupt measurement, skip that measurement row and continue to analysis of the next row, continue analysis of that measurement row, etc.).

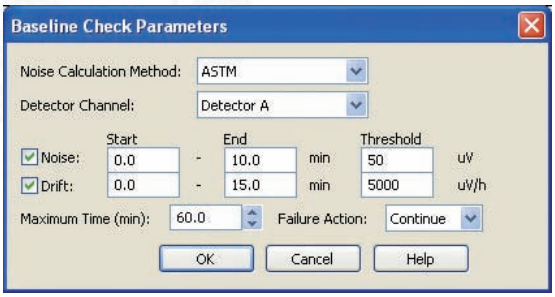


Fig. 4 Baseline Check Function (Parameter Setting Window)

3-3. System Check Function

The system check function refers to such pre-analysis verification operations as determining the cumulative delivery volume of pumps, total number of autosampler injections and total detector lamp illumination time, etc. at the current time. Enabling this function provides a guideline for when to replace such consumable items as the pump plunger seal, autosampler injection valve and the detector lamp. Clicking the [System Check] field in the LabSolutions batch table opens the system check settings window (see Fig. 5). This is where settings are made for implementing output of various system-related checks (consumable items check, instrument self-diagnostics, detector wavelength calibration, and the output of the various logs). To print out the result, just enable the associated printout checkbox. In addition, the results of the various system checks are stored with the chromatogram and quantitative results information, along with each data file. Therefore, if an obviously abnormal value is noticed in the chromatogram, this system check result can be used as one means of determining whether the problem might be attributed to the instrument itself.

When these analysis automation functions including the system check, auto-purge (see 3-1) and baseline check (see 3-2) are all enabled in a single row of the batch table, the order of execution is the system check first, then the auto-purge, and finally the baseline check. Then, depending on the assessment results of the baseline check, the measurement for that row begins, or the batch analysis skips that row and is redirected to the next operation.

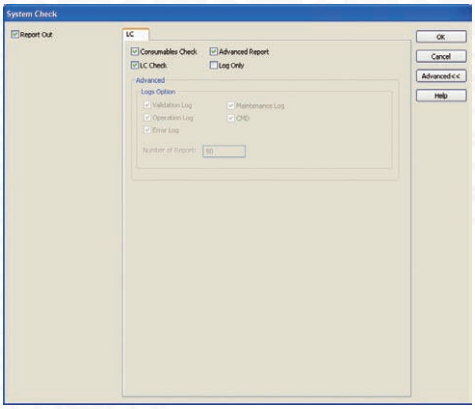


Fig. 5 System Check Function (Parameter Editing Window)

3-4. Batch Action Function

A batch action refers to an "action" operation which causes a redirecting of the batch analysis triggered by a failing result with respect to a specified test item. The action might require any of various measures, for example, interruption of the batch analysis or redirecting the measurement back to the first row or to the last row for measurement of that row. Using this function in system suitability test (see 3-5), for example, if peak retention time repeatability or peak area repeatability does not satisfy the specified criterion value, or if the contribution rate of the generated calibration curve does not satisfy the specified criterion value, the batch analysis run can be set to move automatically to the last row, where shutdown of the instrument could be executed without continuing analysis of the standard and unknown samples. Fig. 6 shows the setting for redirecting the batch analysis operation to the 15th row (goto command) in the event of a system suitability test failure, at which point system shutdown is executed.

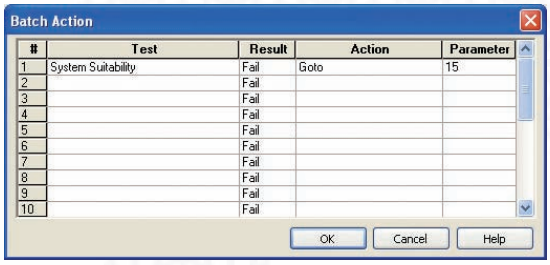


Fig. 6 Batch Action Function (Parameter Setting Window)

3-5. System Suitability Function

The system suitability function is used to check the suitability of the system to conduct analyses by analyzing control or standard samples prior to unknown samples. System suitability test (SST) is normally conducted using repeated analysis, and the retention time repeatability and peak area/height (area ratio/height ratio) repeatability of each constituent of the control sample is judged based on whether it is within the set criterion limit. Parameters that can be used for evaluation in SST include, in addition to those mentioned above, field performance verification parameters such as theoretical plate number, symmetry coefficient and resolution, as well as the relative retention time, S/N (signal-to-noise) ratio, quantitation limit and detection limit parameters.

To perform SST, the system suitability test must be enabled in the method file. In the LabSolutions analysis window, select [System Suitability Settings] on the [Method] menu. For each of the relevant compounds listed in the displayed settings window (see Fig. 7), set the parameters to be used for assessment, the upper and lower limit values, and the %RSD. In addition, select whether the results are to be output in text format or CSV format, and which statistical calculation results are to be output. At the same time, the output format for each parameter can be set. After completing the settings, save the settings to the method file, and set the data to be used for statistical calculations in the batch table. Fig. 2 shows an example of the system suitability settings in the batch table, in which the start is indicated by [Start] in the [System Suitability] field, and the end is indicated by [End], and between these rows [Analysis] is set to indicate data acquisition for the system suitability test.

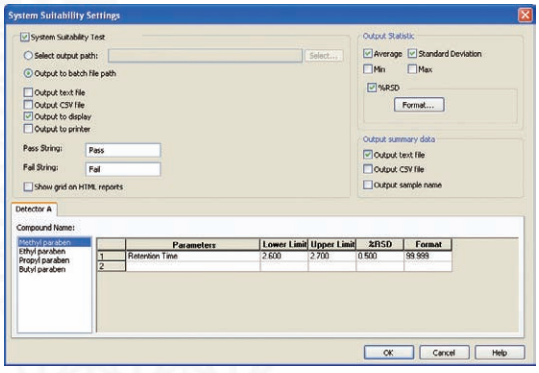


Fig. 7 System Suitability Test (Parameters Editing Window)

3-6. Startup / Shutdown Function

In addition to the functions described above, LabSolutions includes automation of the startup and shutdown operations. The startup function (see Fig. 8) refers to the operation in which a specific time is set for when batch analysis will automatically start. For example, if baseline stabilization will take a significant amount of time, sufficient time can be allocated in the startup time prior to starting the batch analysis. It can also be used to conduct solvent delivery at a low flow rate before the measurement temperature is attained, and then start measurement based on the specified startup method file after a fixed period of time has elapsed. In either case, the batch analysis start time and method file selection settings are made by right-clicking on the batch editing window, selecting the [Settings] menu, and then the [Startup] tab page in the displayed window. The shutdown function (see Fig. 8) refers to the operation in which the state of the instrument following completion of the batch analysis is established using various settings. Similarly as with the startup function described above, the settings in the [Shutdown] tab page are used to set the flow rate and oven temperature, switch OFF the lamp, etc. via the specified shutdown method file, as well as the cool-down period prior to executing the shutdown method.

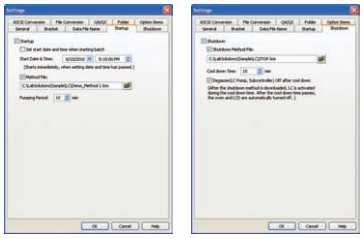


Fig. 8 Startup / Shutdown Functions (Batch Setting Window)

3-7. Custom Parameters Function

The LabSolutions batch table features a custom parameters function which allows the quantitation results to be calculated by applying formulas, containing user-defined factors, to peak area values, height values, concentration values and retention times of identified peaks. For example, useful functions include the calculation of elution quantity in elution tests for pharmaceutical products, etc., and for calculating the ratio of the main peak to peaks of similar substances in the measurement data. The custom parameters function can be set in each row of the batch table, and is applied only to the data in that row. Click the [Custom Parameter] field of the relevant row to display the parameter editing window (see Fig. 9). Enter the title and formula to be displayed in the compound table (quantitation results table) in the LabSolutions window and in the output report. Up to 5 formulas can be set simultaneously in the custom parameters editing window, so a calculation using peaks in the same data, or a calculation using peaks existing in different data of the batch table can be executed at the same time, and both results will be output. Moreover, when creating formulas using multiple factor values, those factor values can be defined using up to 3 constants, A, B and C. By entering factor values in the constant fields, the factor values can be easily changed and checked, even for multiple formulas.

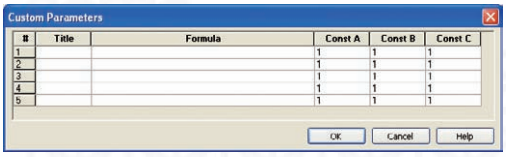


Fig. 9 Custom Parameters Function (Parameter Editing Window)

3-8. Batch Table Creation Support Functions

LabSolutions provides many support functions for table creation, not to mention a table creation Wizard. Table functions are available for simple setting of vial numbers, method files, injection volume and other basic parameters, in addition to table entry support functions such as numerical entry "auto increment" and "set by copying", as well as auto incrementing of data file names, and "undo" and "redo" of entry information. Furthermore, the created batch table can be saved as a batch template. Thus, in daily analysis work, the same work flow (for example, executing an SST and generating calibration curves) can be accomplished by batch analysis, and for repeat analysis of samples, a template can be utilized to eliminate the need to create new batch tables, allowing analysis to be started easily.

4. Conclusion

Using the great variety of batch table automation functions in LabSolutions as introduced above, the complete series of analysis operations from analysis start to field equilibration, system stability check, measurement execution to instrument shutdown can all be automated, allowing operations to keep pace with the lab work flow.
In advancing efficiency improvements in lab analysis operations, automation of analysis and ease of data analysis are important factors in selection of "easy-to-use" software. LabSolutions, which powerfully satisfies this requirement, greatly contributes to work improvements for everybody in the lab.

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