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Novel Function of Auto-diagnostics and Auto-recovery for Detecting Air Bubble Trapped in LC Solvent Delivery Pump

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1. Introduction

In order to improve productivity in modern analysis laboratories, it is essential to reduce analysis time and maximize throughput through regular maintenance. One issue to be resolved is the interruption of analysis due to unforeseeable problems. An example of this is air bubbles in the flow line, which can cause shifts in retention times, pulsating baselines, and unexpected changes in peak shapes. In this poster presentation, we introduce the effectiveness of auto-diagnostics and auto-recovery functions in detecting and resolving this problem automatically. In addition, we also describe the algorithm for air bubbles detection. These functions minimize system downtime due to air bubbles and contribute to the optimization of laboratory productivity.

ANALYTICAL INTELLIGENCE

2. Bubble Formation in Flow Lines

The amount of gas that a liquid can absorb depends on several factors, such as the pressure and temperature gradients, and the nature and type of the liquid and gas (see reference). Gas bubbles are produced in a liquid when the amount of dissolved gas in a solution exceeds the saturated solubility (supersaturation). Usually, the bubbles are removed through the degassing unit. However, in rare cases, they can appear in the flow line of an HPLC / UHPLC and reach the pump. These bubbles can cause shifts in retention times, pulsating baselines, unexpected changes in peak areas, and irregular peak shapes. This can dramatically affect the analytical results due to inaccuracies, poor precision, or inability to distinguish between trace amounts of analytes and the baseline. It also prevents the identification of analytes that are close to their detection limits.

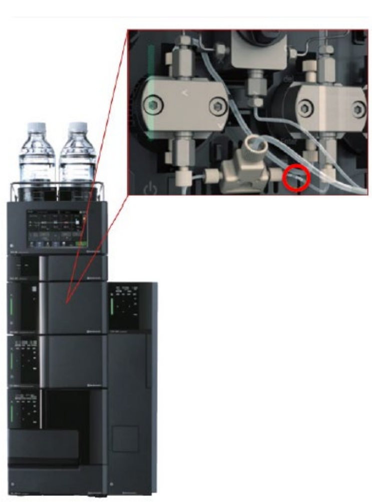

Fig.1. Diagram of the Nexera™ solvent delivery unit flow lines

3. Auto-diagnostics and Auto-recovery

3-1. Overview

Air bubbles can appear in HPLC/UHPLC systems when air has not been removed from the mobile phase, when room temperature varies dramatically or surfactants are added to the mobile phase. When air bubbles are encountered, they require the presence of an operator to be dealt with. The operator will usually remove bubbles by stopping the analysis in progress and purging the flow lines. When the instrument is running unattended (e.g. at night), undetected air bubbles within flowlines can affect a large number of analysis samples, resulting in data loss and time-consuming re-runs.
Auto-diagnostics and auto-recovery functions prevent data loss and waste of samples by automatically detecting abnormal pressure variations triggered by air bubbles within the system and performing corrective actions such as flow line purging until the system regains normal operational status (Fig. 2).

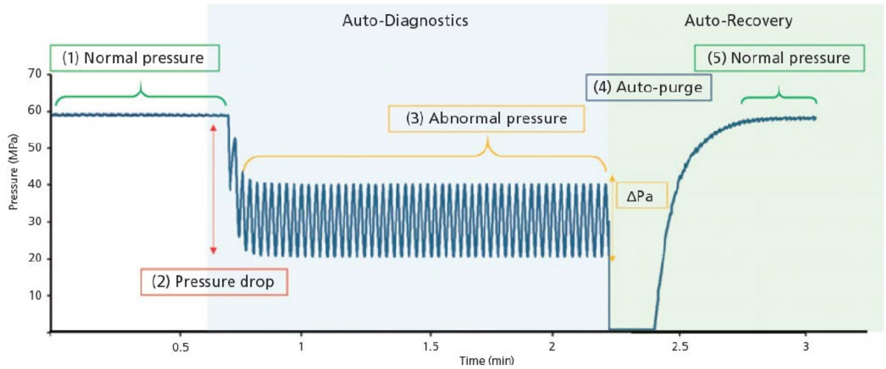

Fig. 2 Pressure changes during auto-diagnostics and auto-recovery
The time spent in each phase can vary depending on analytical conditions and user-defined settings.

3-2. Sequence of Auto-diagnostics and Auto-recovery

Auto-diagnostic and auto-recovery functions are based on a specific algorithm providing the following capabilities. When air bubbles appear in the system, the pressure will drop (Fig. 2, stage 2), and this abnormal pressure will continue (Fig. 2, stage 3). If the new pressure variability ΔPa is abnormal compared to the reference value, the auto-recovery function will be triggered.
In this case, all the subsequent analyses are temporarily suspended. An auto-purge is performed in order to remove any air bubbles from the flow lines (Fig.2, stage 4) and a column rinse is performed. After the auto-recovery process, the pressure profile is checked and compared to the reference values. If pressure variability is normal, the system will return automatically to analysis mode and resume all analyses in the queue.
After auto-recovery, the user can choose to start the interrupted analysis again or to skip this and start from the next line of the batch.

4. Bubble Detection Algorithm

4-1. Mechanism of solvent delivery and pressure drop by air bubble

Our solvent delivery pump adopts a cam driven and parallel dual plunger system. It delivers solvent by operating the left and right plungers alternately (Fig. 3). When air bubbles are formed and reach one side of the pump head, as compared with the normal case, the pressure drops at the timing of the discharge operation of the air trapped side. Thus, we can detect air bubbles by comparing the difference in the amount of periodical pressure change between operation cycles of the two plungers (Fig. 4).

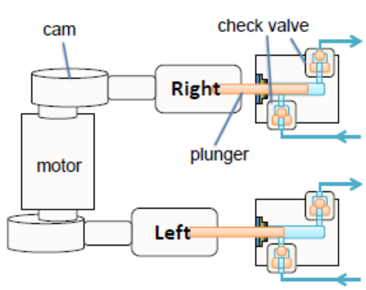

Fig. 3 Overview of solvent delivery mechanism

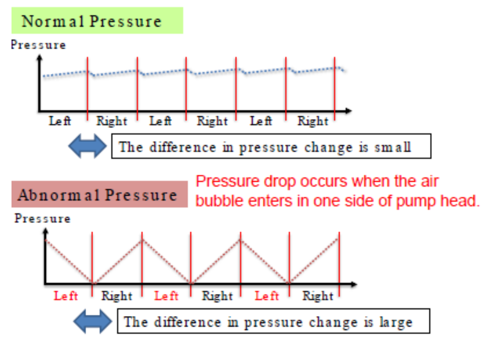

Fig. 4 Overview of air bubble detection

Eq. 1 shows the formula for detecting the air bubble. L1, L2, R1, and R2 represent the pressure values when the left plunger starts and finishes discharging, and when the right plunger starts and finishes discharging, respectively. The threshold value ΔPTh is determined based on experimental data.

$$\Delta P_{Th} \leq \frac{|(L_2 - L_1) - (R_2 - R_1)|}{2} \qquad \text{Eq. (1)}$$

4. Bubble Detection Algorithm

4-2. Distinguishing normal pressure change from the pressure drop caused by an air bubble

Fig. 5 shows the actual experimental data. It shows a case where air bubbles reach the left flow line. The blue line represents L1, the blue dotted line represents L2, the red line represents R1, and the red dotted line represents R2. It can be seen that the pressure decreases at the timing of the discharge operation on the left side, and recovers at the timing of the discharge operation on the right side.
In addition, there are various cases where the pressure changes during the analysis. For example, pressure change occurs at the moment of injecting sample or during gradient analysis, and these must be regarded as a normal pressure change. In order to specifically detect the pressure changes by air bubbles, we check not only the threshold value of Eq. 1 but also the direction of the pressure change synchronized with the two plungers movement. By combining these concepts, we realized a stable air bubble detection.

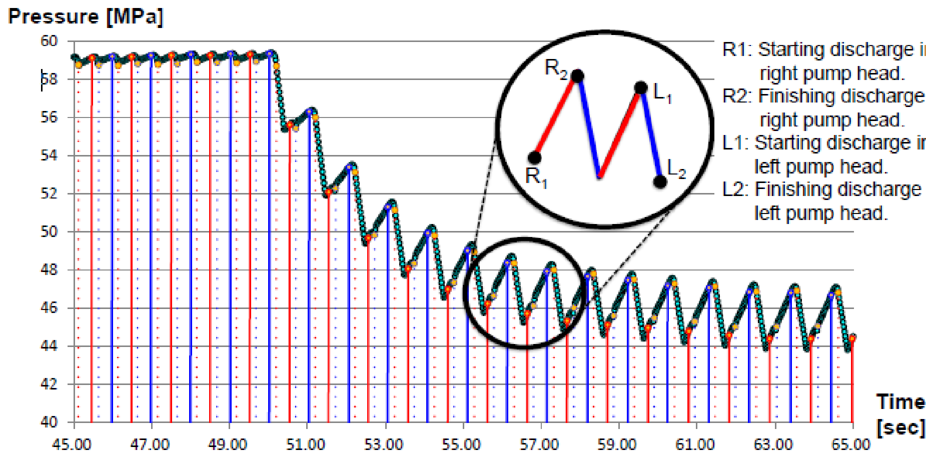

Fig. 5 Pressure transition when air bubbles are trapped in left pump head
(Flow rate : 1 mL/min, Pressure : 60 Mpa, Solvent : Water, Amount of air bubble : 5 μL)

5. Conclusions

- Auto-diagnostics works for automatic detection of air bubbles that appeared in the flow line, and the auto-recovery function allows the system to return to a normal condition.
- Auto-diagnostics function realized stable air bubble detection by using an algorithm based on the solvent delivery method.
- These functions are fully automatic and do not require any human intervention, resulting in increased overall analytical efficiency.

References

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