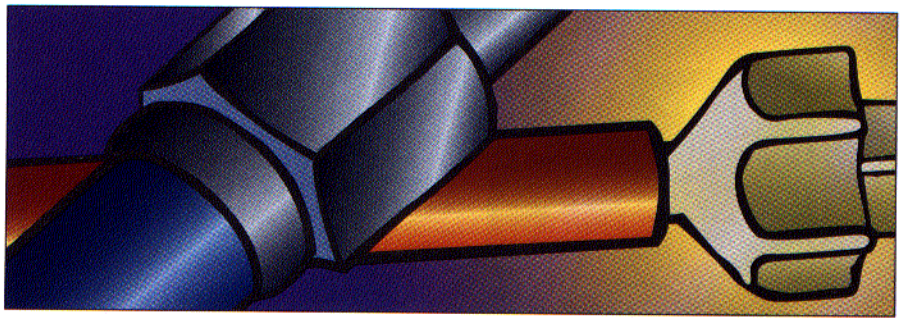
LC Troubleshooting



The System Logbook

John W. Dolan

It is never too late to start a good habit.

As I write this column it is New Year's Day, a day when many people make resolutions for the coming year. Although people traditionally make resolutions on the first of the year, it is never too late to start a good habit. On that theme, I would like to suggest starting several good laboratory habits that center around the system logbook. These practices will improve the operation and maintenance of liquid chromatography (LC) instrumentation.

However, I must offer a couple of cautions before you agree to the challenge. First, you must observe the rule of 21, which tells you that it takes 21 days to form a new habit or break an old one. This rule suggests that we need to persevere when applying a new practice in the laboratory — you may experience setbacks, but if you stick with it, the payoff will come. Second, don't try to make too many changes at once. I'm listing just a few items in this column — if you add too many more of your own, you are apt to create a monster. The magnitude of the changes you propose may be too large to apply practically. It is better to start with one or two simple changes and then add others as the first ones become a part of the routine.

A SYSTEM LOGBOOK

One important tool in an LC laboratory is the LC system logbook. If you work in a good manufacturing practice–good laboratory practice (GMP–GLP) environment, you must keep some formal records about the LC system. This logbook can fulfill that requirement.

In my laboratory, the logbook is a three-ring binder to which forms are added as needed and from which we transfer old records to the central file. We keep our logbook on top of the LC system or in a drawer directly below the system. The logbook must be readily available to LC operators at all times. Any system logbook should contain three important sections: a description of the system configuration, any maintenance records, and LC system performance records.

System configuration: You should keep a record that describes the LC system in sufficient detail that you could reconstruct the system at any future time. If you were to decide right now to walk out of the laboratory and take a month's vacation, would you be able to reassemble your LC system with the same components that are currently installed? If the answer is no, you need a better system configuration record.

We use two forms for the system configuration. One is a list of components that make up the system. The list must have enough detail to describe each component uniquely. It must list the brand, model, and serial number of each component. Alternatively, you can list components in a central inventory with assigned inventory numbers. Using inventory numbers rather than serial numbers may be more convenient for many workers.

Because the LC column is changed so often and is application specific, my laboratory does not include the column, guard column, or in-line filters in the system configuration description. Instead, the column information is listed in the daily run sheet for samples run on that system.

The second form in the system configuration is a configuration change form. This form records any changes that are made in the configuration. You should fill out a separate form for each change to the system. For example, if you replace a UV detector with a fluorescence detector, then note the date, the identity of each component, and the person making the change. This form saves time because you don't have to list all the components that remain unchanged. It also makes it simpler to see any changes made when several workers share an LC system.

When the stack of configuration change forms builds to the point that it is no longer easy to trace the current configuration, make a new list of all the components and move the old component list with all of its associated change forms to the instrument's central file folder.

These records are invaluable for LC system troubleshooting. They allow you to identify changes that were made in the system at any time. For example, if I were preparing a report at the completion of a project and noticed a step change in the data at a certain time, I could go to the system configuration records and determine if that change correlates to an LC system hardware change.

Maintenance records: A second section of the system logbook should contain records of any maintenance that was performed on the LC system. The purpose of these maintenance records is twofold. First, it is important — and in some cases a legal requirement — to keep track of all maintenance done. Second, and perhaps of more practical consequence, maintenance records help to identify failure modes and the expected life-spans of various parts. This information in turn allows users to design logical, routine preventive maintenance procedures.

In my laboratory, the maintenance record forms contain several sections on a single page for each maintenance incident. The first section is a series of check boxes that indicate the type of maintenance performed. Generally, this maintenance falls in one of three categories: repair of system failure, routine preventive maintenance, or a system configuration change. An example of a configuration change would be changing the syringe on the autosampler to enable 5-mL injections rather than the standard 1-mL maximum injection.

A second section prompts for brief description of the failure symptoms. For example, the entry might read, "Pressure pulses of ±10 bar observed for pump A. Degassing mobile phase did not help. Pump B pressure is steady." It is important to list sufficient information to describe the problem but keep the message brief so that it will be easy to review the data in the future to identify failure patterns.

The third section holds descriptions of what was done to correct the problem. Whenever I replace a part, I list the part number for any replaced parts. This list helps clarify problems created if the exact replacement part was not used. Some parts look similar or are available with a similar description but have different characteristics. For example, some manufacturers make pump seals out of different materials for different applications. Third-party vendors also sell replacement pump seals for most common pumps. If you observe premature pump seal failure, it may be useful to review the previous pump seal change to see if the correct part was installed and who supplied it. This information would be unavailable if the entry simply read, "changed pump seals." But if the part number also were included in the entry, tracing the seal source would be simple.

Finally, the maintenance record should include a notation of the date and time the LC system was taken out of service and when it was placed back in service. The name of the person performing the maintenance also should be listed. In some cases, a supervisor's signature may be required before the system can be placed back in service. Each logbook in my laboratory contains a bright red Out of Service tag that is placed prominently on the LC system when it is taken out of service. This tag serves as a visual reminder to others that the system is nonfunctional. After the problem is corrected, the tag should be placed back in the pocket at the front of the notebook.

The real power of the maintenance records is the help they provide in determining failure modes and designing preventive maintenance plans for each instrument. For example, my laboratory uses cartridge-type check valves from a manufacturer that uses the same check valve cartridge for inlet and outlet check valves. Only the holder differs. This design is convenient, because we no longer need to distinguish between inlet and outlet check valves in our supply cabinet; therefore, the laboratory can reduce its inventory, and the cartridge configuration costs less than the system manufacturer's unitized check valves. Are the cartridges as reliable as the unitized check valves? Well, the cartridges seem to work fine on a short-term basis, but it will take a review of maintenance records after several months to determine if check-valve problems are no more frequent with the cartridges than we saw historically with the conventional check valves.

System check: The final section of a system logbook should contain records of the LC system's performance. As described in a previous "LC Troubleshooting" column (1), my laboratory has a standard operating procedure that calls for a performance check of each LC system once each calendar quarter or when major maintenance is performed. This system check includes several items listed in Table I and discussed in reference 1.

TABLE I: LC System Checks

Gradient step test
Gradient linearity
System dwell volume
Autosampler reproducibility
Flow rate accuracy
Pressure decay

The gradient step test checks proportioning accuracy. This test comprises a series of 10% mobile phase changes using an acetone–water mobile phase, which enables users to check the proportioning at any mobile-phase composition. The gradient linearity check is simply a linear gradient from solvent A to solvent B using water as solvent A and water–acetone as solvent B. Because of its critical nature when using retention modeling software, I determine the dwell volume of each system. The

TABLE II: Instrument Use Log Headings

Date	Operator Initials	Comments (Including Solvents Used, Type of Samples, Unusual Conditions, and Noted Errors or Failures)	Maintenance Record Completed?

autosampler reproducibility test assures us that the autosampler is performing properly under the demanding conditions of a small-volume injection as well as in the 50-µL range. I check the flow rate for each pump in the system.

Another useful test I added since the earlier description of our quarterly test is the pressure decay test described in last month's "LC Troubleshooting" column (2). This test involves blocking the pump outlet and then turning the pump on, allowing the pressure to rise to 3000–4000 psi, and turning the pump off. If the check valves and pump seals are working properly, the pressure should drop less than 15% in 10 min.

INSTRUMENT-USE LOG

The sections of the system logbook I described above can help you track system configuration, maintenance, and performance. In my laboratory, operators move from one system to another depending on their current needs rather than assigning one or more LC systems to individuals. This policy means that when an application requires an electrochemical detector, an operator would move to a system that is configured for that use rather than swapping detectors on the system that was used for the previous application. Moving from one system to another can make it difficult to track some of the more subtle problems, so the laboratory has instituted an instrument-use log.

The instrument-use log is a form that is slipped in the clear plastic pocket on the front of the system logbook. Table II shows an example of this form's headings. At a minimum, operators list the date and project for which they used the instrument in the comments section. They make notes whenever they use a maintenance record or make a system change. For example, over a two-month period, one system was used by five operators for eight projects. During that time, the operators encountered pump problems twice. A quick glance at this form allowed them to determine the most common system problems and to see if they correlated to a given project or operator. For example, if pump problems correlated to one project, perhaps the mobile-phase formulation needed to be examined, whereas if the problems correlated to an individual operator, some additional training could help correct the problem.

CONCLUSIONS

The key to any record-keeping system is to make it simple and easy to use. Each laboratory will want to customize the record-keeping process to meet specific needs. This customization may mean creating paper forms such as the ones in my laboratory or a computerized database for other users. Whatever form the system logbook takes, it provides two valuable functions. First, it provides proof of the system configuration and maintenance for regulatory purposes. Second, the logbook allows retrospective evaluation of LC system performance. This information can influence decisions about preventive maintenance procedures, operator training, and system purchase choices.

REFERENCES

(1) T. Culley and J.W. Dolan, *LC•GC* **13**(6), 456–458 (1995).

(2) J.W. Dolan, *LC•GC* **15**(2), 110–113 (1997).

"LC Troubleshooting" editor John W. Dolan is president of LC Resources Inc. of Walnut Creek, California, and a member of the Editorial Advisory Board of LC•GC. *Direct correspondence about this column to "LC Troubleshooting,"* LC•GC, *859 Willamette Street, Eugene, OR 97401, e-mail John.Dolan@LCResources.com.*