LC Troubleshooting

Choosing the Right Instrument

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You have the money — now the problem is deciding what to buy.

One of the questions I receive most frequently requests my opinion on a particular brand or model of instrument. If you expect to find such advice here, you will be disappointed.

It is difficult to provide details about all the instrument features that are available — some will be important for your application, and some will not. Instead of recommending equipment, I've compiled a list of questions that you should address when you consider purchasing a new instrument. These questions will give you some ideas of what to look for when you read the manufacturer's literature and talk to sales representatives. Your final decision will be one of trade-offs — you won't find a single liquid chromatography (LC) system that has everything you want — like buying a house, you have to compromise to enable a timely and cost-effective decision.

Instrument selection is a timely topic for this Pittsburgh Conference issue of *LC•GC*, because many of you will be shopping for new laboratory equipment when you visit Chicago this year. Although your purchase is a weighty decision, it is hard to make a wrong decision because I don't think any bad LC systems are on the market anymore. The instrumentation companies have spent years refining their products, so for many applications the instrument from one company will be comparable to that from another supplier. First we'll look at some general system considerations and then at specific modules.

WHAT'S IT FOR?

The first thing you need to know for your decision is how the LC system will be used. If the system will be dedicated to quality assays using isocratic methods, you may find that a simple isocratic system with a fixed-wavelength UV detector will be satisfactory. If you spend most of your time developing new methods, changing from one method to another, or running gradients, a gradient system will be essential. Even if you never intend to run a gradient, a gradient system can still be an advantage because it gives you the convenience of on-line mixing and reduces waste disposal costs — mobile phases are made only when they are needed.

You should also keep your future needs in mind when you select an LC system. Do you want to use 1- or 2-mm i.d. columns? If you do, you'll need a pump that can handle low flow rates and a detector with a small cell volume. How about preparative or semipreparative separations? Do you expect to require LC–mass spectrometry (MS) detection or will UV detection do? Don't get too future oriented, however — you don't want to pay for a lot of extra features that you may never use. Expect five to seven years of service from an LC instrument. And remember, if your application focus changes drastically, often money will be available at that time for a new system.

INTEGRATED OR MIX-AND-MATCH?

In years past, it was often beneficial to buy individual components from different manufacturers and assemble a customized system that met your specific needs. Today, it is still possible to do this, but the advantages are few. There are two primary reasons for buying an integrated system. (By integrated, I mean a set of modules that are designed to work together as a system — they need not fit in one box.)

The first reason relates to the degree of system control available with today's integrated systems. Most systems from a given manufacturer can be controlled by a single data system. The convenience and cost savings of automated system operation are obvious, but you also get the bonus of system-level diagnostics and the assurance that each piece will work smoothly with the other modules.

A second reason to buy an integrated system is vendor support. You will invariably get better service and applications support when you buy an LC system from a single manufacturer. We've all heard horror stories about the service engineer from company A assuring you that the problem is due to company B's detector, yet the detector manufacturer's service person blames the autosampler. Experiences like these exist more in fantasy than in reality, but the fact remains that you generally get faster and more certain solutions to your LC system problems if all the modules are the same brand.

If you decide to purchase an integrated system — but want the flexibility of mixing-and-matching in the laboratory — be sure that the modules have a keypad for local control as well as the ability to operate as a system under data system control.

WHICH MANUFACTURER?

As I mentioned in the introduction, I don't think any bad LC systems are out on the market. So choosing a manufacturer isn't as simple as selecting a supplier that has good products. The saying "service plus support equals satisfaction" holds true. Are you going to buy a service contract? If so, you will probably be better off with a company that has a service engineer located within 50 miles of your workplace. How fast are service requests filled? A day or two of downtime can quickly cancel out the few thousand dollars you saved by buying from a company with a lower price but slow service response. Don't be afraid of asking to talk to a nearby satisfied customer. If your staff does all of your service work, service response is less important — but can your staff get parts quickly from the manufacturer?

Does the vendor offer good telephone support? Often the salesperson's responsiveness will give you an indication of how the company will meet your needs. I have seen customers switch from one company to another when their favorite salesperson took a new job — they knew that the salesperson was a reliable first line of support. If you leave messages and have to wait several days for a return phone call, what kind of response do you think you'll get when the sales representative is no longer eager to take your money?

So you're buying a second, or third, or fiftieth LC system. Do you stay with the same manufacturer or buy from someone else? Some people like to shop around and buy each system so that its features most closely match the needs of the application. Others like to stay with the same manufacturer and buy the same model of system. The more systems you have that are similar, the more leverage you have for support. You also will have lower costs for stocking replacement parts, because the same parts can be used by several different systems. I visited one facility that had 104 autosamplers of the same brand. It was tough to justify buying anything else because the manufacturer gave 1-h service response and even supplied the stock room with backup units in case of emergency. On the other hand, the users felt trapped by their inability to buy other systems whose features they thought were superior.

ALL IN A BOX?

LC systems come in two varieties: individual components that can be configured by the user for a particular application and systems that come preassembled in a single cabinet. Choosing between the types is largely a matter of personal taste. Space considerations are important. One-box systems often are more compact, requiring less precious bench space. However, some modular systems are stackable and take even less space than the preassembled systems. Consider where you will place the system and how much space is available.

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RESERVOIRS AND DEGASSING

Now let's look briefly at the various system modules. You can use nearly any clean, inert container for a mobile-phase reservoir, ranging from a solvent jug to laboratory glassware to a commercial reservoir. One advantage of commercial reservoirs is that they tend to be more compatible with automatic degassing systems. I am a strong proponent of helium sparging, so I favor systems that allow you to degas the solvent and keep a slight head pressure on the reservoir. Membrane degassers and vacuum degassing are also popular. Bubbles are one of the most common LC problems, and degassing is a sure way to minimize bubble problems. I have yet to see an LC system that doesn't work better with degassed mobile phase.

PUMPS AND MIXING

An ongoing debate compares the relative merits of low-pressure mixing with high-pressure mixing. In a nutshell, low-pressure mixing is usually less expensive because it requires only one pump, but it is more susceptible to bubbles because the solvents are mixed at atmospheric pressure. High-pressure mixing has the advantage of generally lower dwell volumes, so the isocratic lag at the beginning of gradient methods is small. Many systems using high-pressure mixing operate reliably with little or no mobile-phase degassing because the solvents are mixed under sufficient pressure to keep any excess gas in solution (watch out for bubbles in the detector, though). In our laboratory, we have LC systems that perform both types of mixing — each system has its own idiosyncrasies. The choice is largely one of personal preference.

Pumps are equipped with one, two, or three pistons. Historically, more pistons meant more pulse-free delivery, but with microprocessor control of the delivery cycle and the addition of pulse dampers these distinctions have faded. Once again, each manufacturer promotes a particular design, but if one design were clearly

superior I suspect that all manufacturers would use a very similar design.

If you routinely run mobile phases that contain high concentrations of salts (>100 mM) or are corrosive, you may want to consider special pump features. Many pumps incorporate a feature that allows you to flush behind the pump seal to remove salt deposits that can damage the piston. Some methods may require corrosion-resistant or biocompatible pump parts.

AUTOSAMPLERS

You need to consider your typical workload when looking at autosamplers. Be sure that the tray capacity is sufficient for the maximum number of samples you are likely to run at once. Do you require special vials to accommodate very small or very large sample volumes? If you have thermally unstable samples, you may need an autosampler with temperature control. Are you willing to intersperse standards with the samples on the tray or do you want a sampler that will return to the same vial each time a standard is required? Will all injections be the same volume for a set of runs, or do you require individual vial programming? Some of the more sophisticated autosamplers will perform chemical derivatizations and extractions — the extra cost of such a unit can be offset quickly by the reduction in sample preparation costs. Be sure to consider the cost of sample vials and caps — these can exceed the cost of the autosampler over the unit's lifetime. Universal sample vials purchased from third-party suppliers can significantly reduce your expenses.

DETECTORS

Selectivity and sensitivity probably are the two most important specifications of a detector. UV detectors are the most popular detectors used in LC today. Fixed-wavelength UV detectors are the least expensive and most sensitive units for sample compounds that can be detected at one of the limited wavelength choices. Variable-wavelength UV detectors are much more flexible and considerably more expensive. Diode-array UV detectors are very popular because they can gather spectral information and provide valuable information about peak purity. Diode-array detectors are much more expensive than fixed- or variable-wavelength detectors, and the sensitivity suffers in many detectors.

Although UV detectors can handle the majority of LC methods, several other detectors are used routinely for many applications. Ion chromatography uses conductivity detectors. The universal detection capability of refractive index detectors makes them useful for samples that lack chromophores, including many polymers. Mass spectrometry is widely used as an LC detector if analysts need structural information, such as unknown identification and forensic work. Light-scattering detectors can be useful for determining sample molecular weights, and these detectors' prices are a fraction of the cost of a mass spectrometric detector.

DATA SYSTEMS

Selecting the proper data system to collect your sample data and control the LC system can be a more important decision than selecting any of the other LC system components. This month's "Data File" column (see page 106) includes guidelines for choosing the best data system for your use.

SO, WHAT'S YOUR CHOICE?

Buying an LC instrument is not an easy task. Your decision will affect your work as well as that of your co-workers for several years. Gather all the information you can find. Read the manufacturer's literature — after you read about pumps from several suppliers, you'll begin to sort out which specifications are important and which are not. Talk to other users to see what they like and don't like about a given system. I don't think you'll find a perfect system — each has its own good and bad points. Similarly, you are not likely to find a lemon — with today's product offerings, most systems are adequate for routine applications.

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