QuikChem Flow Injection with Ion Chromatography Option

Flow Injection Analysis (FIA) and Ion Chromatography (IC) are complementary analytical techniques that are commonly used in the same laboratory. The QuikChem® 8500 Automated Ion Analyzer offers an Ion Chromatography Option to complement its basic FIA capability. By incorporating both technologies into a single instrument, the following benefits are achieved:

- Operation of FIA and IC simultaneously and independently on the same instrument platform
- Shared use of several peripherals including sampler, dilutor, sampling pump, electronics unit and data station
- Uniform operating protocols
- Reduced footprint
- Unified training, service and support

The QuikChem IC+ Option is available with the original system purchase, integrated at the factory, or as a simple field upgrade to an existing QuikChem 8500. With the availability of FIA and IC in a single instrument, Lachat Instruments offers the most comprehensive ion analysis solutions available today.

Frequently Asked Questions

What does IC+ mean?
Lachat's QuikChem 8500 IC+ is a natural extension of the Flow Injection Analysis (FIA) system (QuikChem 8500 FIA+). Based on the principle of "shared peripherals" the IC+ system offers simultaneous and independent operation of FIA and IC. Typically, the FIA methods are used for determinations of ammonia, TKN, cyanide, phenol, total phosphorus, alkalinity and hardness in various samples. These methods are rugged for harsh matrices and provide a throughput ranging from 60-90 samples per hour. In many instances, however, fewer samples need to be profiled for many ions such as anions, cations, and organic acids and the IC methods are more suitable for these types of analyses. The FIA and IC instruments are complementary and IC+ enables their operation on the same instrument platform. A customer could purchase both FIA and IC at the same time or they could start with either one of them and upgrade to include the other at a later time.

How does Lachat's QuikChem MicroSuppressor™ work?
In Lachat's unique approach, the suppressor is regenerated using a 10-port valve while the sample is being loaded using a 6-port valve. As the 6-port valve turns, a fully regenerated suppressor cartridge is ready to perform chemical suppression for the injected sample. The QuikChem MicroSuppressor is mounted between ports 4 and 7. The 10-port valve performs the following two functions:

• Performing chemical suppression and filling the loop with regenerant solution during the Inject state.
• DI water pushing the regenerant solution through the suppressor followed by wash out during the Load state.

Where does the water dip in the anions method come from?
A water dip that is often present in a chromatogram for anions results from the ionic strength of the injected sample being smaller than that of the eluent. The anions in the sample displace the anions from the eluent (carbonate and bicarbonate) that are adsorbed onto the column packing material. These displaced carbonate and bicarbonate anions when passed through the suppressor column appear as a negative peak. If the ionic strength of the sample is the same as that of the eluent, there will be no water dip. If the ionic strength of the sample is greater than that of the eluent, there will a positive water dip.

Are the water-dip and fluoride peak separated?
Because of the unique column technology and suppression device with minimal dead volume, the water-dip and fluoride peak are fully resolved.

Can you preconcentrate samples to achieve lower detection limits?
We use a large sample loop on a small ID column and the ions get preconcentrated at the head of the column as the sample is injected onto the column.

Does Lachat have methods for drinking water Disinfection By-Products (DBPs)?
Yes, our QuikChem IC method 10-540-00-1-A has method detection limits of 1.6 µg/L for chlorite, bromate, and chlorate and 1.7 µg/L for bromide. These methods meet the requirements as outlined in Information Collection Rule Stage I. Chlorate and chlorite may be present in drinking waters that are disinfected by a chlorination process, whereas bromate may be present in drinking waters that are disinfected by an ozonation process.

Does Lachat have methods for organic acids?
Yes. Our IC QuikChem method 21-550-00-1-A separates oxalic, maleic, malic, citric, tartaric, succinic, formic, acetic, fumaric, and adipic acids in about 18 minutes. The method is based on ion-exclusion separation of these acids followed by suppressed conductivity detection.
Methods 23-550-00-1-A and 23-550-00-1-B are suitable for on-line monitoring of fermentation processes. The analytes determined are: 2-ketoglutarate, acetate, butyrate, formate, fumarate, malate, malonate, propionate, succinate as well as phosphate.

What are the matrices you can support using Lachat methods?
The existing methods can support various matrices such as drinking waters, surface waters, ground waters, waste waters, certain soil extracts including sulfate in phosphate extracts of soils, anions and cations in soil-water extracts, anions in plant samples extracted with 5 mM HCl, high purity waters, cations in food stuff and antifreeze and Bayer liquors.

What detectors are supported?
We currently support conductivity and UV-VIS (post-column reaction).

Does Lachat have ion-exclusion columns?
The organic acids method is based on ion-exclusion column. The QuikChem methods for the organic acids are 21-550-00-1-A, 23-550-00-1-A, and 23-550-00-1-B.

Is it possible to clean an IC column?
Filtering of eluent and samples will increase the column life. Despite all of the care taken, the columns do get contaminated. Typical symptoms include increasing back pressure, peaks eluting too fast, split peaks, and diminished detector response for one or many peaks. The guard column should be cleaned first because it is smaller in size and hence clean up is accomplished much faster than cleaning the relatively longer

analytical column. The sample is then injected again. If the above mentioned symptoms disappear, it is not necessary to clean the analytical column. As part of preventive maintenance, the guard column should be replaced periodically.
The anion columns, guard and analytical, could get contaminated by metals e.g., iron, copper, and lead, surfactants and other organics and particulate. Pumping sodium EDTA through the columns can eliminate the metal contamination. Particulates get trapped in the frit at the column head and will cause higher back pressure than normal and/or split peaks. The anion analytical column has a unique guard disc that can be replaced easily. Anions could contaminate the cation columns in addition to the contaminants listed above. For cation columns, higher strength eluent is used, e.g., 50 mM methane sulfonic acid to remove these contaminants.

What is a guard disc?
The novel Guard-Disc® column protection system consists of an adsorptive disc positioned directly inside the anions column head. The disc prevents any particulate material from entering and disrupting the high efficiency column bed. The adsorptive material is encapsulated in a solid polymer matrix bed. The disk and the adsorptive material can be easily examined to see if particulate or colored material is being retained.

What is a capacity factor? Can OmnionIC software calculate it?
Capacity factor, denoted by k, is a measure of retention time of a peak relative to that of unretained peak. In the case of IC for anions, the unretained peak is the water dip. The capacity factor is calculated as follows:

k = ((retention time of a peak)/(ret. time of unretained peak, i.e. water dip)) - 1.

OmnionIC software can indeed calculate various performance measures including the k-factor (i.e. the capacity factor). Simply click on Method, Performance Options, and fill out the column length, unretained peak time (1.7 min for water dip in the case of IC for anions), and check the box that states calculate the performance options. You could annotate the chromatogram with performance measures.
The user can also export the k-factor for every run. An ASCII file will be created for the method that was run. For example when EPA300A.met is run and the data exported, an ASCII file created for ESTD concentration will be EPA300A.AES and the file for k-factor will be EPA300A.ACF. When using IC for anions, the retention times getting smaller is usually equivalent to k-factor getting smaller because the water-dip will not shift drastically.

How often should the k-factor be calculated and checked?
The user can check the k-factor for the first sample and the last sample for a day. Along with the capacity factor, the user should note the peak shapes, resolution between adjacent peaks, especially chloride and nitrite, and the system backpressure. We state that the resolution between chloride and nitrite at a concentration ratio of 10:1 should be 1.5. Loss in column performance can be seen by several symptoms such as peak tailing (because of a column void), poor resolution between adjacent peaks, e.g. it will be 1.1 between chloride and nitrite at a concentration ratio of 10:1, and split peaks (because of a bad frit).

What k value means that the column is fine, or needs cleaning or cannot provide any useful data?
Example:
• Sulfate retention time of a new column = 9.5 minutes, retention time of water-dip = 1.7 minutes, k-factor = 4.59. In
other words, k-factor of 4.59 for sulfate is for a good column.
• Sulfate retention time of a column after 200 injections of real world samples = 8 minutes, retention time of water-dip
= 1.65 minutes, k-factor = 3.85.

In other words, a drop of k-factor from 4.59 to 3.85, or about 20%, suggests that the column needs cleaning or it needs to be replaced. When this happens, the user will also see that the resolution between chloride and nitrite is getting worse, it may have dropped from 1.5 to 1.1.

How are columns kept in storage?
At the end of the day's work, pump deionized (DI) water through the columns. The anion columns should be

stored in DI water if not used for more than a day. The cation columns should be stored in eluent if not used for more than 3 days. To store them, simply disconnect the tubing and fitting on the columns and connect the end plugs on the columns. Columns should not be stored without the end plugs connected because the columns will dry out.

How long do the columns last? How much do the columns and other consumable cost?
Column life is affected by several factors such as, filtering the eluent through 0.2 µm filter, filtering the samples through 0.45 µm filter, routine maintenance, and the frequency with which the columns get used. Routine maintenance is clearly described in the manuals and is also emphasized during the training. Like HPLC columns, IC columns last longer if they are used more frequently. A good guard column, and a good guard disk in the analytical column, also extends the life of the analytical column.
With proper care, the analytical columns and suppressor cartridge should last for at least one year. A typical symptom of a column getting old is that the peaks start eluting faster than under normal conditions. When this symptom is noticed, the columns can still be used by lowering the eluent strength, or by reducing the flow rate. The guard columns should last for about 6 months. In the USA, a typical laboratory can allocate about $1500 per year for consumables (eluent, DI water, regenerant, other chemicals, tubing, and fittings). Internationally, the yearly estimate for the cost of consumables can vary from country to country. Please check with your distributor.

When determining whether or not the available IC methods could be used for analyzing samples of interest, why it is important to know not only ions as analytes, but also other ions that could be present in the sample?
An example of such a scenario is determining nitrite at 0.05 mg N/L in samples that have chloride at a concentration of 500 mg Cl-/L. Let us assume that the customer is interested only in nitrite and not chloride. In our QuikChem method 10-510-00-1-A, the nitrite peak elutes adjacent to chloride. Although the customer is not interested in chloride, the peak will show up when the sample is injected. At a concentration of 500 mg/L, this peak will completely mask the nitrite peak, and essentially one will not be able to see the nitrite peak. When deciding whether our methods can be used to meet the customer requirements it is therefore important for us to know concentrations of ions of interest as well for ions that could be present in the samples but are not of interest for analysis.

Can Lachat do manual sample injection? If so, how?
Yes, we can. However, we do not need a syringe to do a manual injection. The sample is introduced to the valve using the peristaltic pump. The valve toggles to load and inject using an electronic actuator. Such an ion chromatograph can be upgraded to either a sequential sampler or an XYZ sampler.

Can you do in-line filtering?
Lachat currently does not support in-line filters for the IC+ System.

What sample preparation is essential before injecting a sample into an IC? The samples must be filtered through a 0.45 µm nylon filter. This is essential to prevent clogging of ports on the injection valve and the connecting tubing. There are many commercially available filters that adapt to a syringe.
The sample pH should be in the range of 2-12. Samples exceeding this pH range must be adjusted. Again, there are many commercially available cartridges that adapt to a syringe and, by simply pushing a sample through an appropriate cartridge, the pH can be adjusted to 2-12.
Oily and greasy samples need to be passed through a C18-type cartridge that will selectively remove oil and grease.
If the sample contains too high a concentration of an ion that will mask the adjacent peak(s), that ion must be removed. A typical example is 2000 ppm of chloride in a sample that has to be analyzed for nitrite. In this case, a silver-saturated cartridge is very useful. This cartridge will selectively remove chloride, bromide, and iodide from the sample.
Dilution is another useful technique for diminishing the masking effect due to adjacent peaks. This can be performed either manually or pre-programmed through the software if using Lachat's XYZ sampler/Auto-Dilutor option.