

Improve Cation Exchange Separations

Use Temperature as a Tool

Product Spotlight

The Thermo Scientific™ Dionex™ ICS-5000+ Reagent-Free™ HPIC™ system combined with the Thermo Scientific™ Dionex™ IonPac™ CS19-4μm Cation-Exchange column is an excellent tool for the analysis of common cations and small polar amines in a variety of complex sample matrices. The Dionex IonPac CS19-4μm column uses smaller particles to facilitate high resolution separations of cations and amines in diverse samples, however, the smaller particles also create a higher back pressure, over 3000 psi. In addition, it should be noted that lowering the temperature of the column also increases the system backpressure. Systems and columns that can operate at higher pressures provide the flexibility of using lower temperatures and/or higher flow rates, as both cause pressure increases. The Dionex ICS-5000+ HPIC system operates up to 5000 psi providing the flexibility to operate under numerous conditions. The system comprises an eluent generator, which electrolytically and reproducibly produces from deionized water the accurate concentrations of methanesulfonic acid needed for the eluent. The user needs only to provide the deionized water and the samples to the system. Both the Dionex IonPac CS19 and Dionex IonPac CS19-4μm Cation-Exchange columns were used in these examples and are made from polymeric supermacroporous particles with a weak carboxylic acid functional group.

General Effects of Temperature in Chromatography

Temperature effects in HPLC and IC are not as significant as in gas chromatography, but nevertheless it is another tool available to the user which can many times help in achieving a desired separation. The temperature effect will depend on the analyte's structure, the column chemistry and the eluent in use. In ion chromatography, a higher column temperature typically results in higher peak efficiencies and lower pressure, and it may also result in needing less (or no!) organic solvent in the eluent to elute the more hydrophobic analytes. For most compounds, higher column temperature results in shorter retention time, but this is not the rule as there are many exceptions.

There is always a maximum allowable temperature for a column, and care should be taken not to exceed this temperature. In some column chemistries, operating the column at elevated temperatures can slowly result in some column degradation or column bleeding, shortening its lifetime. Conversely, a column operated at a lower temperature could have a much longer lifetime. For molecules subject to on-column racemization or isomerization, separations are normally conducted at low column temperatures. This paper demonstrates the beneficial effects of operating a cation exchange column at a lower temperature.



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Better Resolution at Lower Temperatures?

When developing an ion chromatography method, it is the general tendency to first elevate the column temperature when the desired resolution cannot be achieved by optimizing the eluent conditions. However, in some cation-exchange applications, a lower column temperature will improve the separation. Figure 1 shows a separation at 30 °C resulting in complete co-elution of the pair dimethylamine/potassium (peaks 5 and 6) as well as partial co-elution of the pair diethylamine/morpholine (peaks 7 and 8). Though not shown here, as the column temperature is raised above 30 °C, the co-eluting pair dimethylamine/potassium can be resolved, with potassium actually eluting before dimethylamine. However, the other pair, diethylamine/morpholine will co-elute as the temperature is increased. When the temperature is lowered to 15 °C, all four analytes in question are baseline resolved from each other. At the lower temperature, potassium is retained longer in the column than dimethylamine and the elution order of this pair is reversed, with potassium eluting after dimethylamine. As demonstrated here, besides affecting resolution, temperature can be used to reverse the elution order of a pair of analytes. This can be advantageous when one of the analytes is present in much higher concentration than the other, as it is much easier to quantitate the lower concentration analyte when it elutes before the higher concentration analyte.

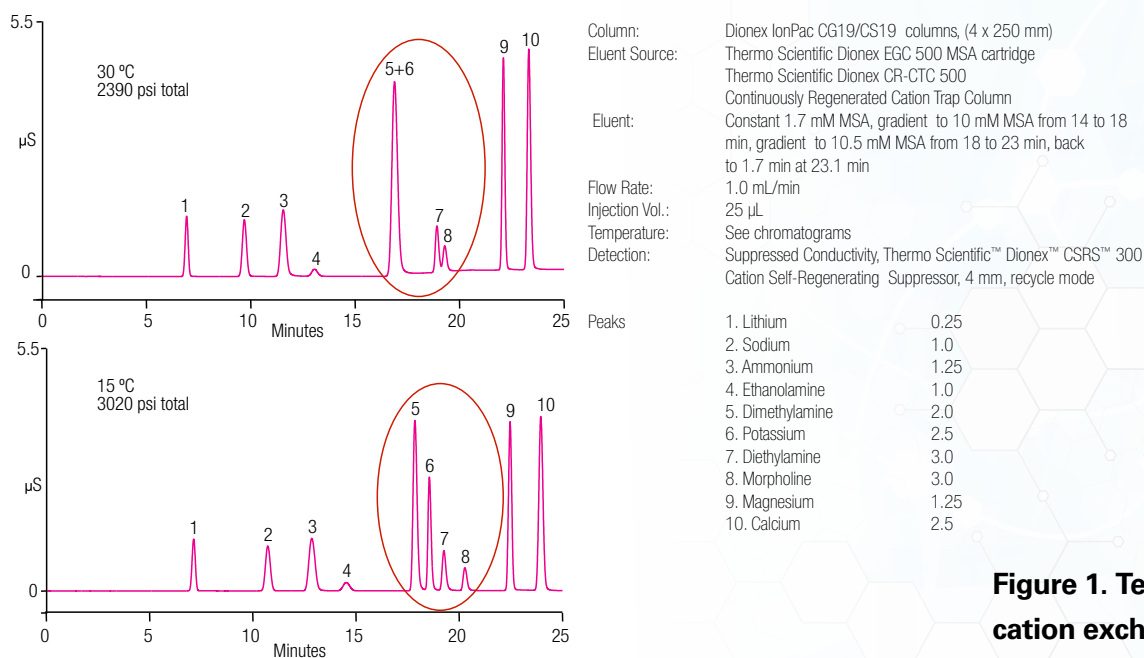


Figure 1. Temperature effect on a cation exchange separation.

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Faster Analysis at Lower Temperatures?

The combination of a lower temperature with a gradient eluent can significantly decrease the analysis time while improving the separation. The separation of the common six cations and ethylamines in Figure 2A took about 40 minutes when an isocratic eluent was used with the column at 30 °C. In the process of optimizing the separation and minimizing the analysis time, a gradient eluent was used in combination with a reduced column temperature of 15 °C resulting in a 25 minute run time shown in Figure 2B. Furthermore, as the column temperature was lowered, an impurity (peak #7) co-eluting with diethylamine (peak #6) was resolved from it. Manipulation of the column temperature enabled this improved resolution, illustrating how column temperature optimization can aid the user. Combining both optimized column temperature with gradient elution is a powerful tool to improve cation-exchange separations.

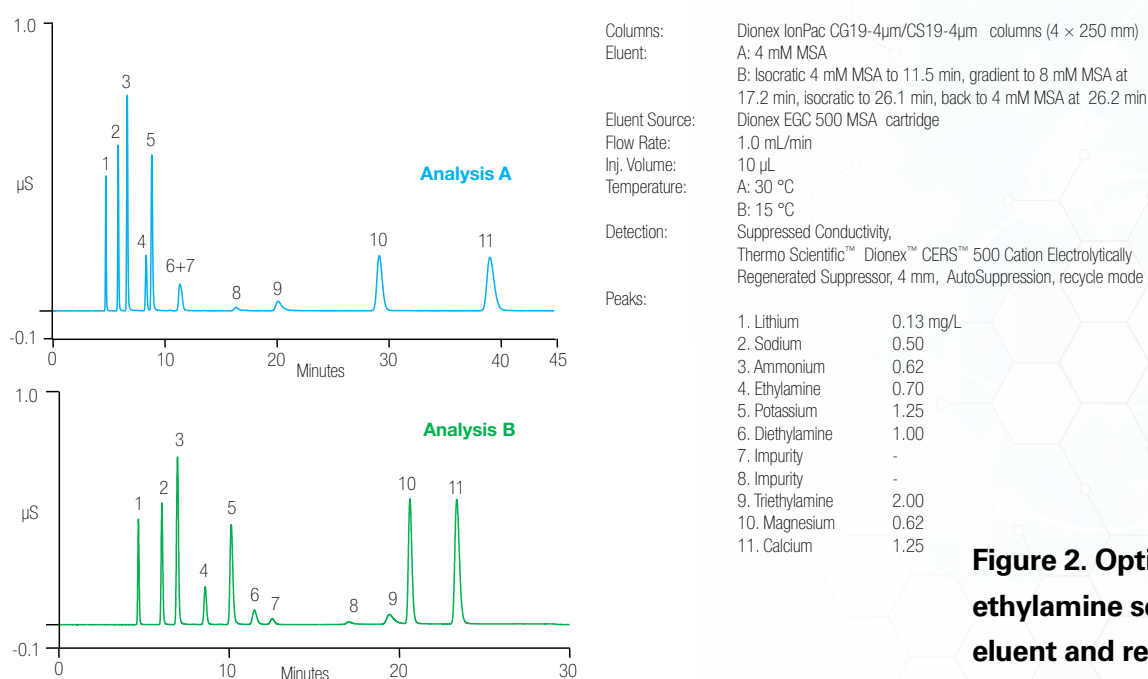


Figure 2. Optimization of a cation and ethylamine separation using a gradient eluent and reduced column temperature.

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