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Ion Chromatography Troubleshooting Guide

Resolving IC analysis issues

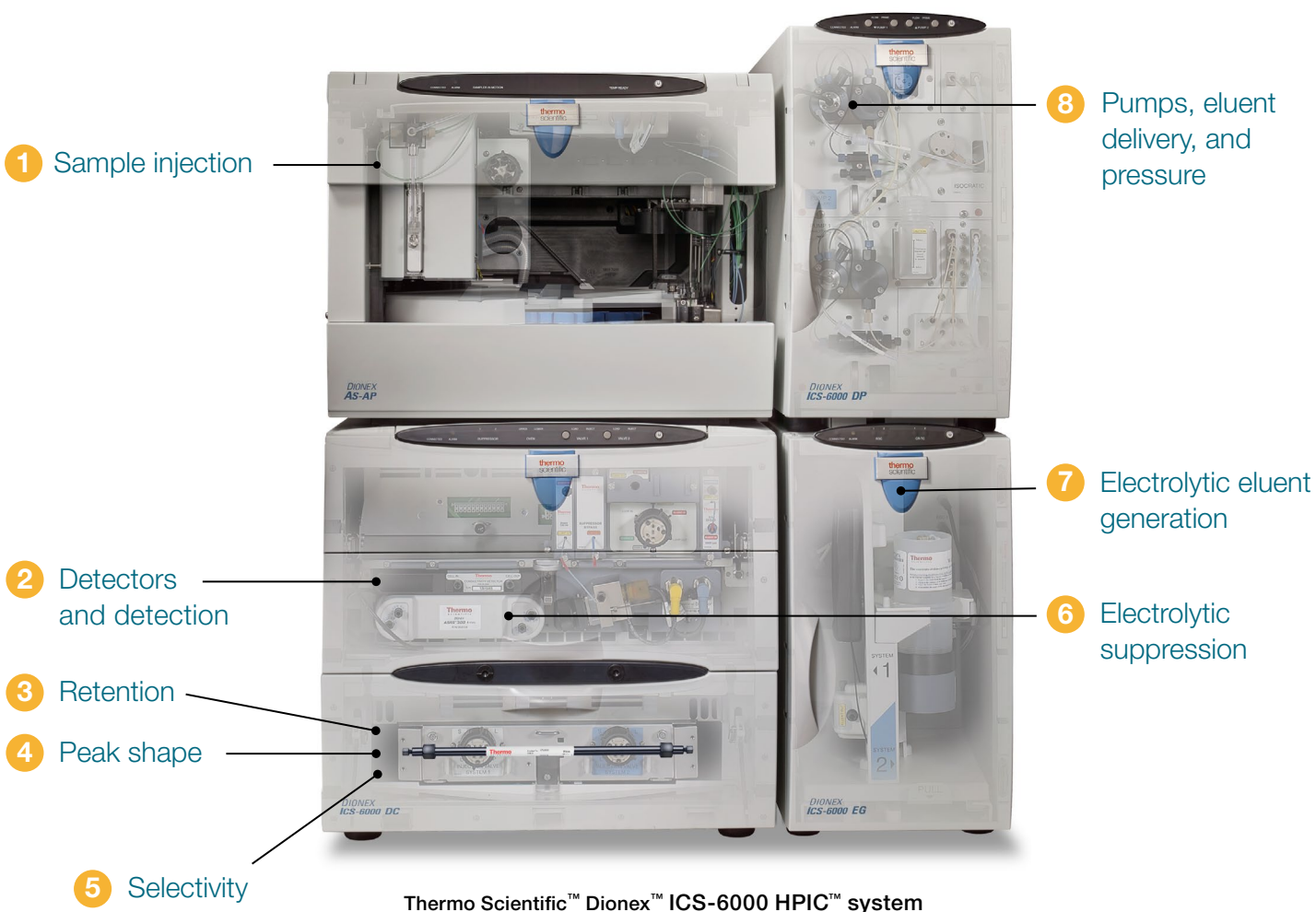
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Resolving IC analysis issues

Ion Chromatography (IC) is the premier technique for determining ionic compounds in solution. The basic components consist of an eluent source, pump, sample injector, separating column, suppressor, and detector. As with any lab instrumentation, your IC system should be serviced regularly to ensure peak performance. If problems arise that are not addressed by routine maintenance, this troubleshooting guide provides you with suggested solutions so you can quickly resume generating the accurate, reliable data that Thermo Scientific™ Dionex™ IC systems are known for.

Common IC Issues

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1 Sample injection

Ensure injection-valve materials are compatible with eluent conditions before use. If possible, filter all standards and samples before injection, and ensure that the sample syringe is free of bubbles before sample loading. If possible, use suction to draw sample into loop to avoid contamination or carryover from syringe.

Symptom	Solution
No peaks	Check that the injector valve is plumbed correctly and is switching correctly. If using an autosampler, check for trapped air bubbles, check correct location of samples and wash solutions, and check that sampling needle is reaching the sample. Check needle tip for damage and ensure needle is aligned properly with the injection port and is not leaking. For dual systems sharing an autosampler, check the injection valve configuration and ensure the correct valve is switching.
Poor injection precision	Ensure use of factory recommended autosampler settings and that the injection valve waste line is routed as recommended in the manual. Check sample transfer line calibration. Check syringe operation and replace, if necessary. As above, check the autosampler operation, and check for air bubbles. Check and wash the rotor and seals, replacing if necessary. For manual injection, leave the needle in the needle port while switching the valve to inject. Check the stability of the sample and standards.
Sample carryover	Check the sample and standard compatibility with eluent and for signs of sample precipitation. Flush the injection loop with up to 10 volumes of sample before injection. For problematic (viscous) samples, dilute further if possible.
Reduced or reducing peak heights	Check for adsorption, precipitation, or evaporation of sample or sample components within sample vial or connecting tubing. Carry out the above maintenance procedures to ensure correct valve operation.
Excessive pressure and pressure spikes during injection	Confirm that the valve is switching quickly and smoothly. Ensure that the valve and loop are free from salt deposits and signs of precipitation. Wash (sonicate) or replace rotor, seals, and loop as necessary.
Leaking injector	May be caused by a worn or scratched injector valve rotor or a damaged injector rotor and needle seals. Remove each, wash, check, and replace if necessary.

2 Detection and detectors

Suppressed conductivity detection is by far the most common detection mode for modern IC. Other commonly used detectors include UV absorbance and electrochemical. For all detector types, eluent purity and correct preparation are key to successful IC separations.

Symptom	Solution
Excessive noise	Check all predetection modules (including suppressor) for sources of noise and eliminate. Check for trapped air bubbles in detection cell. Check detection cell for any visible contamination and clean if necessary. Check lamp usage time (UV absorbance detection), replace if necessary.
Baseline drift	Check for changes or drift in flow rate. Check for fluctuations in room temperature. Use column and detector cell temperature control (conductivity detection). Check for column bleed and contamination (remove column and remeasure baseline drift). For absorbance detection, ensure sufficient warm-up and check lamp usage time.
Periodic baseline fluctuation	If fluctuations appear to be synchronous with pump cycle, purge the pump and check for general functionality. To eliminate the potential for air in the fluidics, degas eluents or source water (for eluent generation) and purge pump.
No peaks	Check the injector operation. Check standard integrity. As above, ensure that all predetector modules are operating correctly. Check the baseline noise. To verify detector operation, remove the column and inject standard to observe response. For UV absorbance detection, ensure correct wavelength setting.
Lack of analyte sensitivity	Carry out a signal-to-noise analysis and compare to previous data. Verify the eluent conditions and suppressor function (conductivity detection). Check the eluent composition and background conductivity or absorbance (high background signal can obscure analyte peaks.) Check for proper injector functioning.
Nonlinearity	Check the system precision, both with and without column. Check the baseline and standard stability over time. Ensure that the signal-to-noise ratio is constant. Work above 10:1 signal-to-noise ratio for quantitation. Make sure you are not overloading the column. Establish and work within linear range. Note: For weak acid and weak base species, conductivity is fundamentally nonlinear.

3 Retention

In IC, analyte retention is dependent upon eluent strength and, for weak anion and cation exchangers, pH. Variation in retention is generally the result of eluent preparation errors or pump issues.

Symptom	Solution
Decreasing retention times	Column usage can affect capacity. Reproduce the production test chromatogram using conditions noted on the Quality Assurance Report that was shipped with the column to assess column performance. If capacity has been lost, attempt column regeneration per the manufacturer's recommendations or replace the column. Check the eluent concentration. Irreversible adsorption of highly charged species from samples will reduce available capacity. Check the sample for potential sources of column fouling. Use IC sample preparation to minimize matrix effects and prevent column fouling.
Increasing retention times	Verify that the actual eluent concentration and flow rate are what have been specified in the method. For organic ions, check secondary hydrophobic interactions (switch to more hydrophilic ion exchange column if available).
Variable retention times	Check the eluent concentration and pump stability. Ensure that there is sufficient equilibration time between runs. Check injection volumes and concentrations of sample and standard; if the column is being overloaded, try a higher capacity column, or inject a smaller volume. To eliminate fluctuations due to changes in room temperature, place column in thermostatted oven.

4 Peak shape

Poor peak shapes are generally a sign of secondary (non-ion exchange) interactions with components within the system, stationary phase, suppressor membranes, or adsorbed contaminants (such as metal ions). For increased IC column lifetime, always prepare eluents from deionized water (resistance over 18 MΩ -cm) and highest purity (manufacturer-recommended) reagents. Always use a guard column.

Symptom	Solution
Tailing peaks	Reproduce the production test chromatogram using conditions noted on the Quality Assurance Report that was shipped with the column to assess column performance. Check for improper tubing connections (use of Dionex IC PEEK Viper fittings is recommended). Check if tailing occurs with all peaks or only with certain analyte classes or individual ions (this will help identify if secondary interactions are causing tailing). For unwanted hydrophobic interactions, use a more hydrophilic column or use an organic solvent additive to eluent (check complete system compatibility). If metal contamination is suspected use the manufacturer's recommended column reconditioning procedures.
Fronting peaks	Check the column capacity and avoid sample overloading. Check the eluent concentration setting for eluent generation cartridge in Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software, or manual eluent preparation calculations and pH. Check for possible column voiding or packing irregularities and replace the column, if necessary.
Split peaks	These are most often caused by column overloading. See above advice for fronting peaks. This may be a sign of column void, in which case the column requires replacing.
Ghost peaks	Check the system for contamination. Sources can include deionized water system tubing, manually prepared eluent reagents, and autosampler wash water. Ensure complete elution of strongly retained ions before the next sample injection. System peaks are often seen when peaks appear because of eluent ion retention.

5 Selectivity

Selectivity refers to the relative order in which retained analytes elute from a column. Considerable variety exists in IC stationary phase chemistry, including a range of strong, weak, and mixed-mode ion exchangers based upon nonporous, porous, and agglomerated resins (and monoliths), together with a large range of silica-based phases. In IC, eluent pH can play a significant role in selectivity for weakly acidic and basic ions, and affect retention and selectivity on weak ion exchangers. Use the **Virtual Column online tool** available on AppsLab to find the best Thermo Scientific™ Dionex™ IC column for your analytes of interest.

Symptom	Solution
Reduced resolution	Check eluent concentration and pH (particularly important when using weak ion exchangers and nonsuppressed detection modes). Verify proper pump function. Check the column temperature stability and use a thermostatted column oven, if available. Reproduce the production test chromatogram using conditions noted on the Quality Assurance Report that was shipped with the column to assess column performance. Check for sample matrix effects.
Increased resolution	As above. Increased retention of solitary or specific ions indicates system contamination. Apply regeneration or washing procedures and repeat.
Changes in column-to-column selectivity	Check to see if the difference is due to lot-to-lot variation by comparing the column performance certificates (Quality Assurance Report, QAR). Check the column usage history. Apply regeneration or washing procedures.

6 Electrolytic suppression

Always ensure that the manufacturer's guidelines for correct hydration, startup, operation, shutdown, and storage procedures are followed precisely to avoid irreversible damage. If error messages are observed, consult the operation manual before attempting a solution. Always check solvent compatibility of electrolytic modules before using organic solvents within IC eluents.

Symptom	Solution
Leaking suppressor module	Check that connectors and tubing are correctly fitted. With suppressor module off-line, check back pressure of downstream modules and eliminate any excess pressure sources (see suppressor manual for detailed instructions). Check inlet and outlet flow for both regenerant and eluent solutions independently to detect leaks across suppressor membranes. If breakthrough is detected, replace suppressor.
Increasing background and baseline noise	Check and reduce excess back pressure (ensure that the conductivity cell is operating at recommended back pressure). Check eluent and regenerant solution purity and flow. If noise continues, carry out suppressor cleaning and regeneration procedures per the manufacturer's recommendations.
Baseline spiking	Ensure that the correct suppressor settings are being used. Ensure the purity and gas-free condition of all solutions. Remove traces of contamination with cleaning procedures and rehydrate the module.
Poor linearity and deteriorating peak shapes	For anion-exchange IC, poor peak shapes can result from metal ion contamination of the membrane. Regeneration or cleanup of the membranes using the manufacturer's recommended procedures should be attempted.

7 Electrolytic eluent generation

Always ensure that the manufacturer's guidelines for correct startup, operation, shutdown, and storage procedures are followed precisely to avoid irreversible damage. If error messages are observed, consult the operation manual before attempting a solution. Always check solvent compatibility of electrolytic modules before using organic solvents within IC eluents.

Symptom	Solution
Leaking eluent generator cartridge	Check and tighten the fittings. Check for tubing blockages and high system back pressure. If the cartridge itself leaks, replace cartridge.
No power	Check the power supply, connections, and contacts.
Low system pressure	Check cartridge and EG degasser for leaks and if present, replace. Ensure high-pressure pump is primed.
No peaks	Confirm that the correct concentration has been set in the instrument method. Ensure that the eluent concentrate cartridge has not been exhausted.
High eluent background	Check the water supply purity. Ensure that the continuously regenerated trap column is replaced each time the eluent cartridge is replaced or after one year of use. Verify proper suppressor operation.

8 Pumps, eluent delivery, and pressure

Typically, in ion chromatography, inert (PEEK) dual piston pumps are used. Stainless steel pumps designed for use with HPLC, should be avoided due to incompatibility with IC eluents. Check all deionized water and eluent reservoirs and connecting tubing for signs of contamination and particulates. Only use the highest quality sources for preparation of eluents and filter them before use.

Symptom	Solution
Fluctuating and pulsing flow	Purge the pumps with degassed eluent. Persistent bubbles in the pump heads usually can be removed using degassed methanol or propanol. If pulsing persists, remove the check valves and wash (sonicate in water-methanol). If the problem persists, consider replacing the seals and cleaning the pump heads. Ensure that sufficient system back pressure is provided.
Fluctuating pressure reading	As above, carry out purging, cleaning, and maintenance procedures.
Air bubbles	Filter using a 0.45 µm inert hydrophilic filter (avoid cellulose) and degas all eluents thoroughly, using sonication under vacuum. Purge the pumps each time fresh eluent or water is prepared.
Leaking pump heads	Check the seals and replace, if necessary. Check instrument manual for eluent compatibility with pump seals.
Salt deposits	Wash with water to remove (check piston for scratches and replace, if scratched). Check the piston heads for leaks. Replace the seals if necessary.
High back pressure	Ensure pump flow rate is not set too high. Check for excess backpressure tubing installed between the pump and injection valve, and remove or replace, as needed. Ensure the IC system can support the typical operating pressure of the installed column as specified in product manuals. Check the guard column for blocked frits and replace, if necessary. Check each module and waste line for blockages and ensure that fittings are not overtightened. Ensure tubing of the correct internal diameter is used throughout the IC system and that there are no excessive bends.



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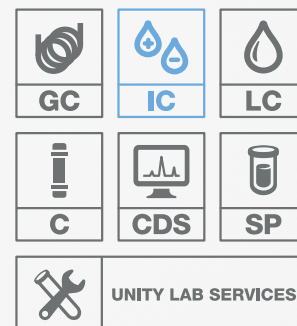
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