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TECHNICAL NOTE 179

Effect of Temperature on IC Signal Noise

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Keywords
Conductivity detection, electrolytic suppression, Integrion, signal-to-noise, thermostatted compartment

Introduction

Noise is an important parameter of any analytical instrument and is defined as an unwanted modification of the signal from known or unknown sources.

Ultimately the sensitivity of any chromatography detector is defined by its signal-to-noise ratio. The aim of this sensitivity optimization experiment is to increase signal, decrease noise, or some combination of the two.

Equipment

The results shown in this technical note were gathered on the equipment listed below. However, the principles of electrolytic suppression and conductivity detection are conserved no matter which IC system is used, so results should be similar across a wide range of IC instruments , including the Thermo Scientific™ Dionex™ ICS-5000+ system and Thermo Scientific™ Dionex™ Integrion™ HPIC™ system.

- Dionex Integrion HPIC system with the following features:
- Analytical pump
- Eluent degas
- Eluent generation (EG)
- 6-port injection valve
- Column oven
- Conductivity detector
- Thermostatted compartment
- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System (CDS) software, version 7.2 SR4

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Reagents and Standards
18 MΩ-cm deionized (DI) water
Fisher Scientific reagents, ACS Grade

Standard Preparation
A 10 g/L sulfate stock solution was prepared by dissolving 14.79 g of sodium sulfate in 1 L DI water. A 10 mg/L sulfate standard solution was prepared by diluting the stock solution. Dilutions were made with DI water without any pretreatment prior to injection.

System Configuration
The Dionex Integrion HPIC System with reagent-free ion chromatography (RFIC™) capabilities was configured according to standard RFIC system operating conditions as outlined in the Dionex Integrion HPIC system Operator's Manual. The suppressor was mounted in the thermostatted compartment; thus the temperature of the suppressor could be controlled by setting the thermostatted compartment temperature. An extended length of 0.007" i.d. tubing (25 cm) was used to connect the outlet of the column to the inlet of suppressor; this ensured thorough stabilization of the eluent temperature to the temperature in the thermostatted compartment before entering the suppressor. To ensure changes in detector compartment directly affect suppressor temperature, an extended length of 0.020" i.d. tubing (50 cm) was used to connect the outlet of the conductivity detector (CD) cell and the inlet of the suppressor regen. These extra lengths of tubing were used to isolate the impact of temperature on the various components in the system. Under normal operating conditions the tubing lengths should be minimized to optimize dead volume.

The CD cell is installed inside the thermostatted compartment and has only heating ability. For stable operation, it must be kept at a temperature at least 7 °C higher than the thermostatted compartment.

Experimental Design
In each experiment, data was collected over a 1 h period at each temperature after a 3 h equilibration period. Each data point represents an average of 60 one-minute noise readings. Error bars represent the standard deviation of these noise values.

Conditions

Columns:	Thermo Scientific™ Dionex™ IonPac™ AG11 (4 x 50 mm) Thermo Scientific Dionex IonPac AS11 (4 x 250 mm)
Eluent Source:	Thermo Scientific Dionex EGC 500 KOH Eluent Generator Cartridge Thermo Scientific Dionex CR-ATC™ 600 Continuously Regenerated Anion Trap Column
Eluent:	38 mM KOH, unless otherwise stated
Flow Rate:	1.2 mL/min
Column Temp:	33 °C
Inj. Volume:	15 µL
Detection:	Suppressed Conductivity, Thermo Scientific™ Dionex™ ERS™ 500 (4 mm) Electrolytically Regenerated Suppressor, recycled eluent mode 113 mA, unless otherwise stated
System Backpressure:	2600–2800 psi

Standards of 10 mg/L sulfate solution were infused into the injection loop from a 1 L polypropylene container. The standard was delivered via a gravity siphon feed.

Results and Discussion
It is important to isolate the effect of temperature on the conductivity cell from the effect of temperature on the suppressor. Thus, the Dionex Integrion HPIC system, with thermostatted compartment, is an ideal platform for studying these effects as the temperature of the suppressor and the cell can be controlled independently with the ability to cool in the thermostatted compartment.

The first two experiments were designed to isolate the effect of temperature on the CD cell and suppressor respectively. The later experiments were designed to test the system under conditions where the CD cell and thermostatted compartment are maintained at a constant 7 °C difference, but are varied together.

Effect of Variation of CD Cell Temperature

Figure 1 shows the effect of varying the CD cell temperature on total conductivity signal noise while holding the suppressor temperature constant at 20 °C. The results show that increasing the temperature of the CD cell results in a decrease in recorded noise on the signal.

Effect of Variation of Suppressor Temperature

Table 1 and Figure 2 show the effect of varying the suppressor temperature on total conductivity signal noise while holding the conductivity cell constant at 47 °C. This temperature was chosen to ensure the CD cell remained at least 7 °C above the highest detector compartment temperature in the data set.

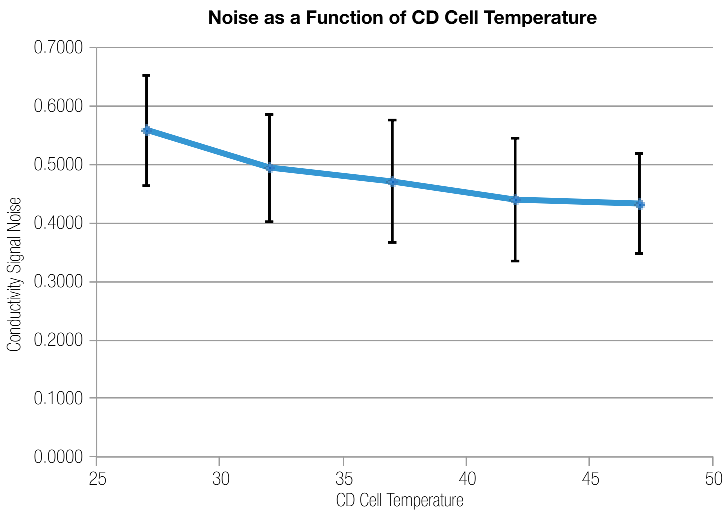

Figure 1. Suppressor temperature was kept constant at 20 °C. Error bars show a single standard deviation of recorded noise values over a period of one hour.

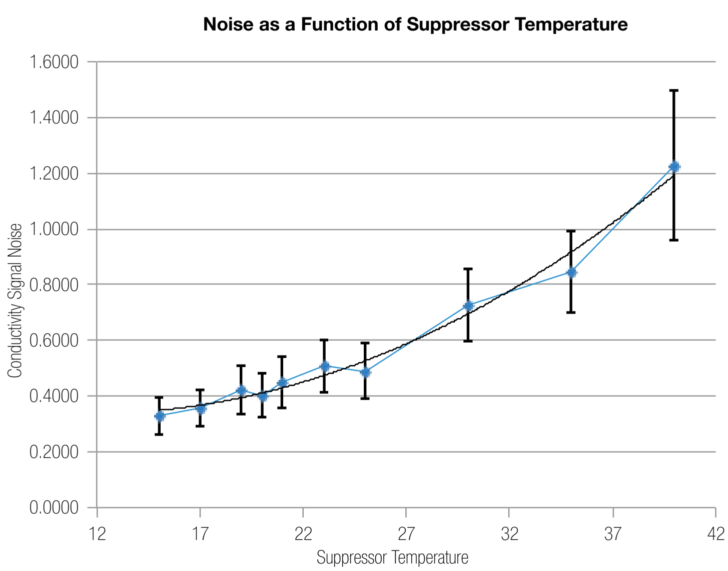

Figure 2. CD cell temperature was kept constant at 47 °C. Error bars show a single standard deviation of recorded noise values over a period of one hour.

The results show that increasing suppressor temperature results in an increase in recorded noise on the signal.

Table 1 reveals that raising the suppressor temperature in the 15–25 °C range has a moderate impact on noise, effectively adding 49% more noise to the signal. Raising the temperature above 25 °C has an even greater effect on noise, increasing the noise nearly four fold at 40 °C.

Supp. Temp (°C)	Signal Noise (µS)	% Increase
15	0.328	
20	0.402	23%
25	0.490	49%
30	0.726	121%
35	0.847	158%
40	1.228	275%

Table 1. Conductivity signal noise as a function of suppressor temperature. CD cell temperature was kept constant at 47 °C. % Increase values are based on the 15 °C baseline.

Effect of Variation of CD Cell and Suppressor Temperature

Table 2 and Figure 3 show the effect on total conductivity signal noise of varying both the CD cell and suppressor temperature simultaneously. The CD cell was kept a constant +7 °C warmer than the suppressor during the course of this experiment. As expected, changes in the suppressor temperature overwhelmed the positive impact of varying the CD cell temperature, thus an increase in suppressor and CD cell temperature led to an increase in recorded noise on the signal.

The data in Table 2 reveal that raising the suppressor temperature in the 15–25 °C range has a modest impact on noise, effectively adding 39% more noise to the signal; less than the impact of raising the temperature on the suppressor alone. However, raising the temperature above 25 °C has a deleterious effect on noise, effectively adding 204% at 35 °C and 393% at 40 °C.

Supp. Temp (°C)	Signal Noise (µS)	% Increase
15	0.363	
20	0.427	18%
25	0.504	39%
30	0.615	70%
35	1.102	204%
40	1.789	393%

Table 2. Conductivity signal noise as a function of suppressor temperature. CD cell temperature was varied with a constant +7 °C difference to the suppressor temperature. % Increase values are based on the 15 °C baseline.

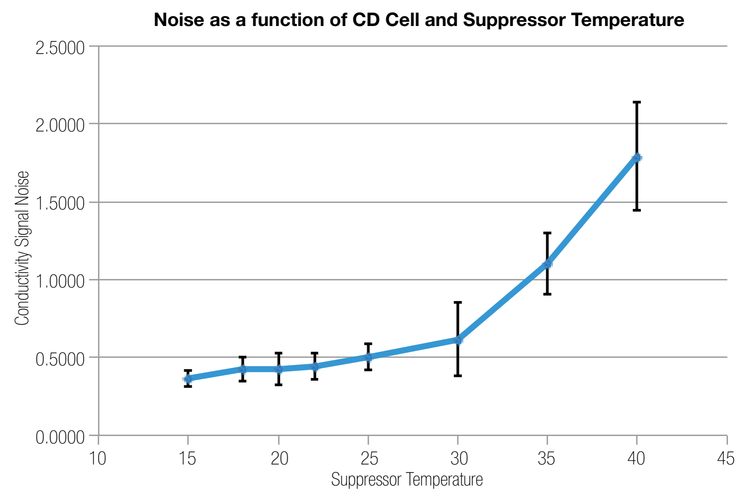

Figure 3. CD cell temperature was varied with a constant +7 °C difference to the suppressor temperature. Error bars show a single standard deviation of recorded noise values over a period of one hour.

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Table 3 and Figure 4 show the effect of varying both the CD cell and suppressor temperature simultaneously on peak height and calculated signal to noise ratio. At each temperature, 15 µL injections of 10 ppm sulfate were made and the height of the peak was taken as the signal strength. As the temperature was lowered on the detector compartment and the detector cell, the peak height (signal) simultaneously increased while the noise on the signal decreased. Thus the overall signal-to-noise ratio is improved by a factor of 4.5 over the temperature range of 40 °C to 20 °C.

Conclusion

This work conclusively demonstrates the benefits of active cooling of an electrolytic suppressor. Minimizing the operating temperature of the suppressor maximizes the sensitivity of the system while simultaneously reducing the variability in noise, leading to a more stable response. Ideally, temperatures of 15 °C should be employed for maximum performance. Thus, an actively cooled, thermostatted compartment is critical for maximizing signal sensitivity.

Supp. Temp (°C)	Peak Height	S/N
20	2.9900	6997
22	2.9867	6710
25	2.9333	5821
30	2.8567	4643
35	2.8533	2590
40	2.8100	1571

Table 3. Sulfate standard peak height and calculated signal-to-noise ratio as a function of suppressor temperature. CD cell temperature was varied with a constant +7 °C difference to the suppressor temperature. To determine peak height, a 15 µL injection of 10 ppm sulfate standard was injected.

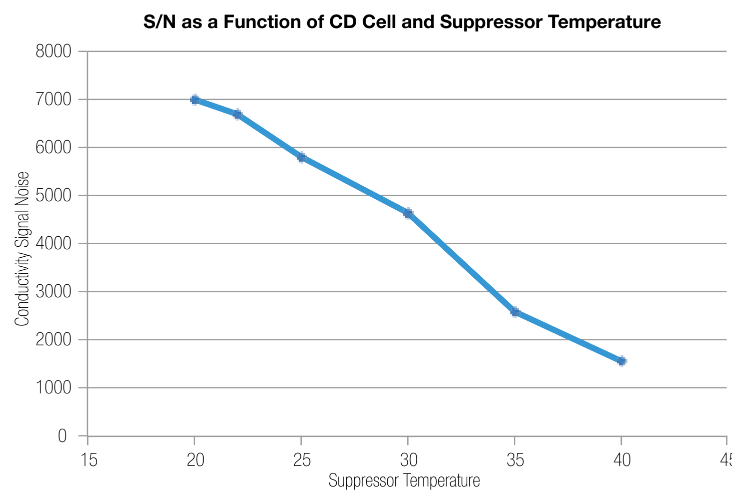

Figure 4. CD cell temperature was varied with a constant +7 °C difference to the suppressor temperature. To determine signal strength, a 15 µL injection of 10 ppm sulfate standard was injected.

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