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IC-MS analysis of perchlorate with a new, robust, and reliable next-generation suppressor

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Introduction

Perchlorate is a highly soluble anion that can contaminate water sources, posing risks to human health by interfering with iodine uptake in the thyroid gland, which is crucial for hormone production and metabolic regulation. Accurate detection and quantitation of perchlorate in water are vital for ensuring safe drinking water and protecting public health. Ion chromatography-mass spectrometry (IC-MS) is a technique for the precise detection and quantitation of perchlorate in water samples, following the guidelines of US EPA Method 332.0.[1]

The performance and reliability of this method are significantly influenced by the suppressor used in the ion chromatography system. The newly developed Thermo Scientific™ Dionex™ Next-Generation Electrolytic Suppressor (NGES) delivers increased backpressure tolerance and features additional robustness and reliability compared to the previous Thermo Scientific™ Dionex™ Anion Dynamically Regenerated Suppressor (ADRS).[2] These improvements are particularly advantageous for IC-MS applications where stable performance under varying pressure conditions is essential.

This document demonstrates the performance of the Thermo Scientific™ Dionex™ NGES Anion Suppressor (NGES-A) for the implementation of US EPA Method 332.0, highlighting the increased robustness and resilience of the Dionex NGES suppressor compared to the previous suppressor model.

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Next-generation suppressor

The components of the Dionex NGES suppressor are shown in Figure 1. Among the notable improvements for the Dionex NGES suppressor are the reinforced membrane and optimized seal design, which provide consistent performance up to pressures of 500 psi (3.4 MPa) and offer the ease of use of electrolytic suppressors. This is a significant enhancement over the 150 psi tolerance of previous Dionex DRS suppressors. These improvements make the Dionex NGES suppressor more suitable for IC-MS analyses where excess backpressure on the suppressor can be a concern due to the addition of extra components such as transfer tubing, a diverter valve, and an ion source needed to introduce the sample to the mass spectrometer.
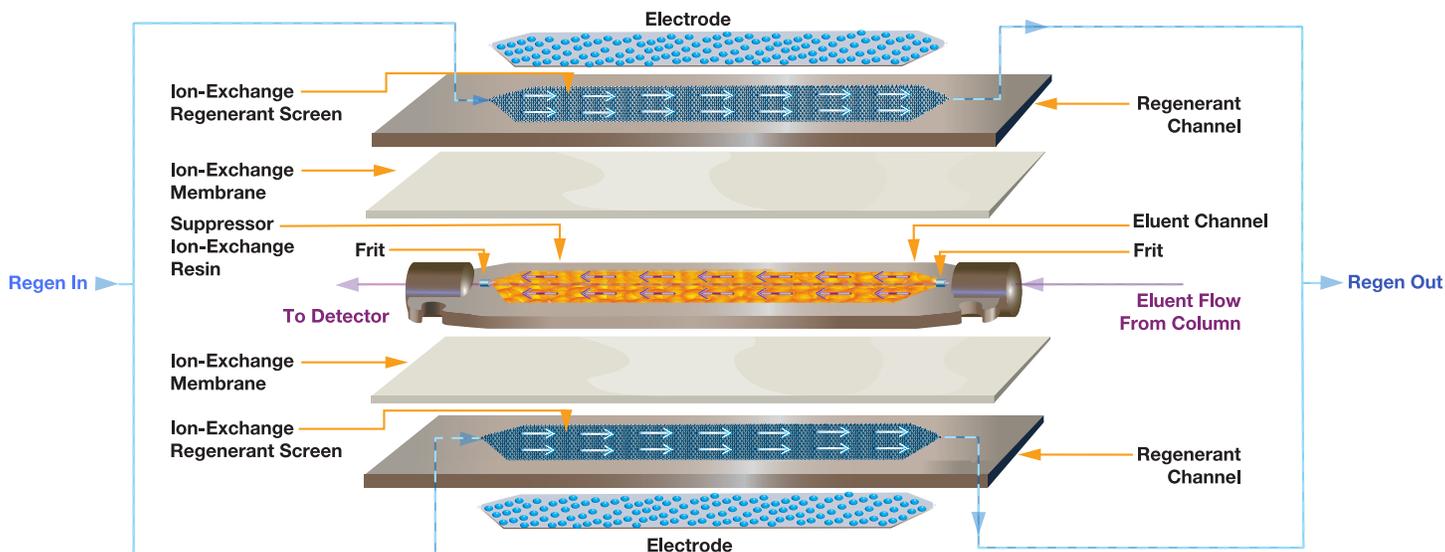

Figure 1. Schematic of Dionex NGES suppressor with reinforced membrane.

Experimental

Equipment

The Thermo Scientific™ Dionex™ ICS-6000 HPIC System includes:

- DP dual pump module with degas option (P/N 22181-60007), or SP pump module with degas option (P/N 22181-60001) and AXP pump (P/N 060684)

- EG eluent generator module (P/N 22181-60019)

- DC detector/chromatography module (P/N 22181-60049) with CD conductivity detector (P/N 079829)

- Thermo Scientific™ Dionex™ AS-AP Autosampler (P/N 079656)

Thermo Scientific™ ISQ™ EC Single Quadrupole Mass Spectrometer (P/N ISQEC-IC)

In this experiment, the second injection valve on the DC module was used as a diverter valve. Optional AM automation manager (P/N 079833) and 6-port valve (P/N 075917) can also be used. Refer to previously published AN72587 for valve configuration.[3]

Software

Thermo Scientific™ Chromeleon™ Chromatography Data System Software 7.3.2MUe

Consumables

Thermo Scientific™ Dionex™ IonPac™ AG20 Guard Column, 2 mm (P/N 063066)

Thermo Scientific™ Dionex™ IonPac™ AS20 Analytical Column, 2 mm (P/N 063065)

Dionex NGES-A Next Generation Electrolytic Suppressor, 2 mm (P/N 060003)

Thermo Scientific™ Dionex™ EGC 500 KOH Eluent Generator Cartridge (P/N 075778)

Thermo Scientific™ Dionex™ CR-ATC Continuously Regenerated Anion Trap Column (P/N 088662)

Thermo Scientific™ Dionex™ RFIC Eluent Degasser (P/N 075522)

Reagents and standards

Deionized (DI) water, Type 1 reagent grade, 18 MΩ·cm resistivity or better

Sigma-Aldrich™ sodium perchlorate (P/N 634565) or equivalent

Thermo Scientific™ sodium perchlorate $NaCl^{18}O_4$ internal standard (P/N 062923) or equivalent

Instrument method		
System		Dionex ICS-6000, ISQ EC
Columns		Dionex IonPac AS20, Analytical, 2 x 250 mm Dionex IonPac AG20, Guard, 2 x 50 mm
Eluent source		55 mM KOH via RFIC eluent generation
Flow rate		0.3 mL/min
Column temperature		30 ºC
Injection volume		100 µL full loop
Detection	CD	Suppressed conductivity with Dionex ADRS 600 in constant current mode or Dionex NGES-A suppressor with current set at 41 mA, recycling/external water mode using the diverter valve.
	MS	ISQ EC ESI, negative mode

Ion transfer tube	200 °C	
Vaporizer	450 °C	
Aux gas	5.0 psig*	
Sheath gas	50.0 psig	
Sweep gas	0.0 psig	

Eluent flow is diverted to waste until 6 minutes.

Component	*m/z*	CID** (V)
Perchlorate 99	99	40
Perchlorate 101	101	40
Perchlorate 107	107	40
Full scan	50–300	40

Run time	14 min

*The measurement of pressure relative to atmospheric pressure.

**Collision-induced dissociation (CID).

Results

Separation

Figures 2a and 2b show chromatograms of a 500 ng/L perchlorate calibration standard with a 1.0 µg/L internal standard at m/z 99, 101, and 107. Figures 3a and 3b show chromatograms of 1.0 µg/L perchlorate in a laboratory fortified synthetic sample matrix (LFSSM) containing 1000 mg/L each of chloride, sulfate, and carbonate to illustrate the detection of perchlorate in high ionic strength matrices. In both experiments, Dionex NGES-A and Dionex ADRS 600 suppressors produced similar results.
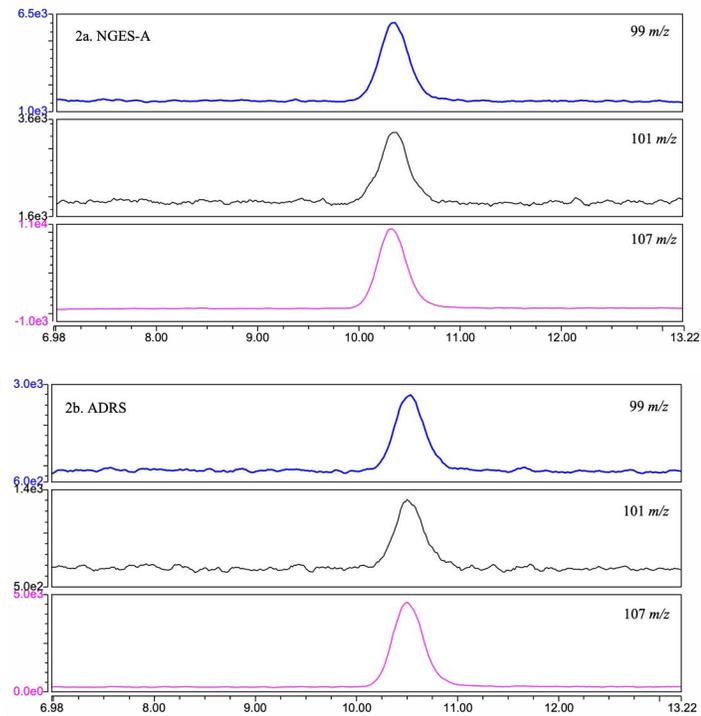

Figure 2. 500 ng/L perchlorate calibration standard with 1.0 µg/L internal standard using Dionex NGES-A suppressor (a) and ADRS 600 suppressor (b).
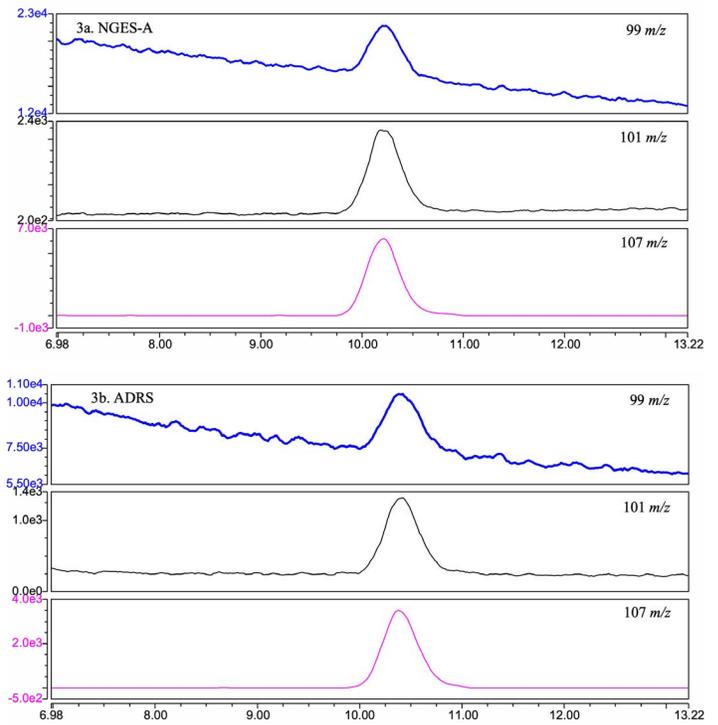

Figure 3. 1.0 µg/L perchlorate LFSSM with 1.0 µg/L internal standard containing high ionic strength matrix using Dionex NGES-A suppressor (a) and ADRS 600 suppressor (b). The downward slope for 99 m/z traces is caused by excess sulfate that elutes before perchlorate. Approximately 4% of sulfur isotopes are ^{34}S, which gives sulfate an m/z value of 99, the same mass we are looking for in perchlorate.

Calibration

Relative standard error (RSE) was used as an evaluation method of calibration curves instead of traditional relative standard deviation (RSD). This is because RSE standardizes precision assessment across various scales and enhances calibration quality.[4,5] The area ratio of m/z 99/107 and m/z 101/107 were plotted to create a method calibration curve and their results for both the Dionex NGES-A suppressor and the Dionex ADRS 600 suppressor are shown in Table 1. In both experiments, Dionex NGES-A and Dionex ADRS 600 suppressors produced similar results with excellent fit, indicated by low RSE value.

	NGES-A		ADRS 600	
	RSE (%)	R^2	RSE (%)	R^2
Perchlorate (*m/z* 99)	1.22	0.998	6.57	0.999
Perchlorate (*m/z* 101)	2.74	0.999	3.22	0.991

Table 1. Calibration information for perchlorate using Dionex NGES-A and ADRS suppressors. Number of calibration levels = 6 (NGES-A), 7 (ADRS 600). Calibration standard range = 0.1–10 µg/L. Calibration method = internal standard (107 *m/z*). Calibration type = quad, with offset, 1/A^2.

Conclusion

The new Dionex NGES suppressor's design enhancements ensure reliable analysis of perchlorate in water samples, thereby promoting safer drinking water and protecting public health. With its improved robustness and reduced leakage, the Dionex NGES suppressor meets the growing demands of the IC market, especially for IC hyphenated solutions like IC-MS, making it an ideal choice for high-efficiency separations.

References

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