APPLICATION PROOF NOTE

No. 203

Determination of Perchlorate by U.S. EPA Method 332.0 Using a Compact Ion Chromatography System Coupled with Mass Spectrometry (IC-MS)

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Introduction

Perchlorate has been used as an oxidizer in rockets, munitions, and fireworks since the 1950s. It has been found to cause thyroid dysfunction, and has been linked to tumors in humans. Perchlorate is regulated under the Safe Drinking Water Act (2011). Additionally, numerous U.S. states have promulgated enforceable standards for the levels of perchlorate in drinking water. Massachusetts and California have established standards for drinking water of 2 µg/L and 6 µg/L respectively. More than 10 other states have also set advisory levels or health-based goals for perchlorate in drinking water ranging from 1 to 18 µg/L.

U.S. EPA Method 332.0 — Ion Chromatography with Suppressed Conductivity and Electrospray Ionization/Mass Spectrometry is one of the most sensitive and robust characterization methods available for perchlorate. Mass spectrometry (MS) provides lower detection limits in high-ionic-strength matrices than conductivity detection alone. The selectivity of the mass spectrometer allows the quantification of perchlorate in high-ionic strength matrices at well below currently enforced action levels.

This application proof note demonstrates the determination of perchlorate in environmental waters using the method published in Thermo Scientific Application Note 151 and U.S. EPA Method 332.0. Here, the method is performed using a Thermo Scientific™ Dionex™ Integrion™ HPIC system, a Thermo Scientific™ Dionex™ IonPac™ AS20 column set, and Thermo Scientific™ MSQ Plus™ Single Quadrupole Mass Spectrometer. Figure 1 shows the determination of 1.00 µg/L of perchlorate in a Laboratory Synthetic Sample Matrix (LSSM), which includes 250 mg/L each of chloride, bicarbonate, and sulfate.

Method

Conditions	
IC System:	Dionex Integrion HPIC system
MS Detector:	MSQ Plus Mass Spectrometer
Columns:	Dionex IonPac AG20 (2 x 50 mm) Dionex IonPac AS20 (2 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:	55 mM KOH
Flow Rate:	0.3 mL/min
Column Temp:	35 °C
Inj. Volume:	200 µL via Thermo Scientific™ Dionex™ AS-AP AutoSampler
System Backpressure:	~2000 psi
Detection:	Suppressed Conductivity, Thermo Scientific™ Dionex™ AERS™ 500e (2 mm) External Water Mode via Thermo Scientific™ AXP™ Pump, 41 mA
Mass Spectrometric Detection, details below	
Divert Valve Switch Time:	6.0–14.0 min (0.0–6.0 min to waste, 6.0–14.0 min to MSQ Plus MS)
Desolvation Pump Flow Rate:	0.3 mL/min., 50:50 water:methanol via Thermo Scientific AXP Pump
Ionization:	ESI Negative Mode
Needle Voltage:	-3 kV
Cone Voltage:	-70 V
Probe Temperature:	500 °C
Scan:	SIM mode; 99, 101, 107, 191 m/z
Dwell Time:	0.3 s

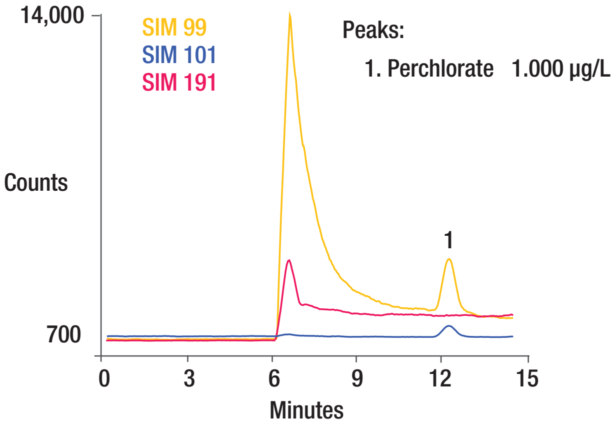

Figure 1. 1.00 µg/L Perchlorate in Laboratory Synthetic Sample Matrix (250 mg/L Chloride, 250 mg/L Bicarbonate, and 250 mg/L Sulfate).

References

1. Thermo Scientific Application Note 151: Determination of Perchlorate in Environmental Waters by Ion Chromatography Coupled with Electrospray Mass Spectrometry (IC-MS). [Online] https://appslab.thermofisher.com/App/1949/an151-determination-perchlorate-environmental-waters-by-ion-chromatography-coupled-with-electrospray-mass-spectrometry-icms (accessed Aug. 1, 2016).
2. U.S. Environmental Protection Agency, Method 332, Determination of Perchlorate in Drinking Water by Ion Chromatography with Suppressed Conductivity and Electrospray Ionization Mass Spectrometry, Rev. 1.0, 2005. [Online]

Find out more at **www.thermofisher.com/ICMS**

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