



IC APPLICATION NOTE

Perchlorate Detection by IC Using Hydroxide as the Eluent

1 part per billion MRL for perchlorate as per US EPA 314.0

Perchlorate is a chemical compound that's been drawing significant attention in recent years. Commonly used in products like rocket fuel, fireworks, and explosives, perchlorate can also enter the environment through industrial discharge and natural processes. Its presence in drinking water has become a public health concern due to its potential to disrupt thyroid function.

This method utilizes a Supp 21-150/4.0 column in combination with the 948 Continuous IC for the analysis of perchlorate. The high capacity of the A

Supp 21 column helps preserve the perchlorate peak shape even in the presence of 3000 ppm equivalent of TDS. The use of 948 Continuous IC ensures continuous, automated preparation of hydroxide eluent. This method doesn't use any organic solvent in the eluent.

SAMPLE AND STANDARD PREPARATION

1000 ppm stock standard solution of perchlorate was prepared by dissolving 0.141 g of sodium perchlorate monohydrate in 100 mL of UPW. The

water samples were introduced into the IC without undergoing any sample preparation step.

EXPERIMENTAL

This new and optimized method to execute EPA method 314 was carried out using an A Supp 21-150/4.0 mm column, maintained at a constant temperature of 55 °C. Perchlorate was measured via conductivity detection in sequential suppression mode with an eluent flow rate of 1.0 mL/min and an injection volume of 1000 µL. Potassium hydroxide at a concentration of 60 mmol/L generated continuously using 948 CEP was used as the eluent.

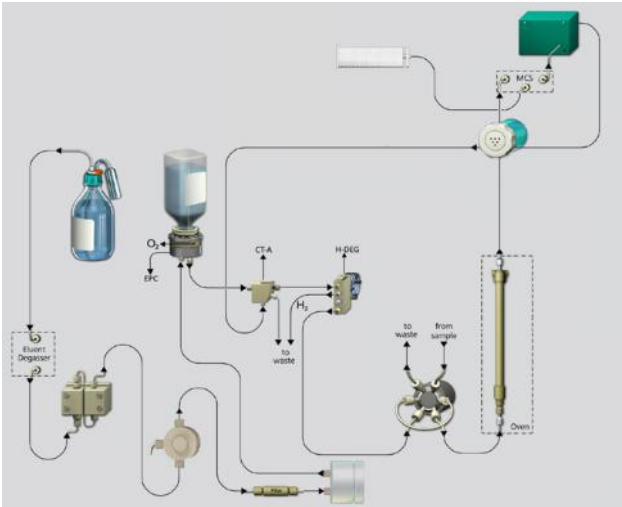


Figure 1. Flow path for the determination of perchlorate by suppressed ion chromatography.

RESULTS

A linear calibration curve was generated for perchlorate within the range of 0.5 ppb to 25 ppb. Tap water sample was analyzed for perchlorate, and it was also subjected to spiking with a known concentration of perchlorate. Perchlorate wasn't detected in tap water sample; the 2-ppb perchlorate spike was found at 2.02 ppb with a recovery of 101 %. To study the extent of perchlorate peak distortion in the presence of very high concentrations of anions, a Maximum Conductivity Threshold (MCT) study as per USEPA Method 314.0 was conducted. Mixtures of anions (chloride, carbonate, and sulfate) were prepared at different levels, from 200 ppm each to 1000 ppm each, and spiked with 5 ppb of perchlorate.

Table 1. Perchlorate concentrations in tap water samples (triplicate runs per sample)

Sample	Perchlorate conc., ppb	% RSD
Tap water	Non-Detect	-

Table 2. Spike Recovery Results (%)

Sample	Pre-Spike, ppb	Spike, ppb	Post-Spike, ppb	Recovery, %
Tap water	0.00	2.00	2.02	101

Table 3. Minimum reporting limit (MRL)

Inj. #	Actual Conc., ppb	Measured Conc., ppb	% Accuracy
1	1.00	1.07	107
2	1.00	1.20	120
3	1.00	1.05	105
4	1.00	1.01	101
5	1.00	1.20	120
6	1.00	1.06	106
7	1.00	1.07	107

Table 4. MCT study data (as per EPA 314.0, section 9)

Chloride ppm	Carbonate ppm	Sulfate ppm	Total TDS	PGF
200	200	200	600	0.96
400	400	400	1200	0.93
600	600	600	1800	0.97
1000	1000	1000	3000	0.98

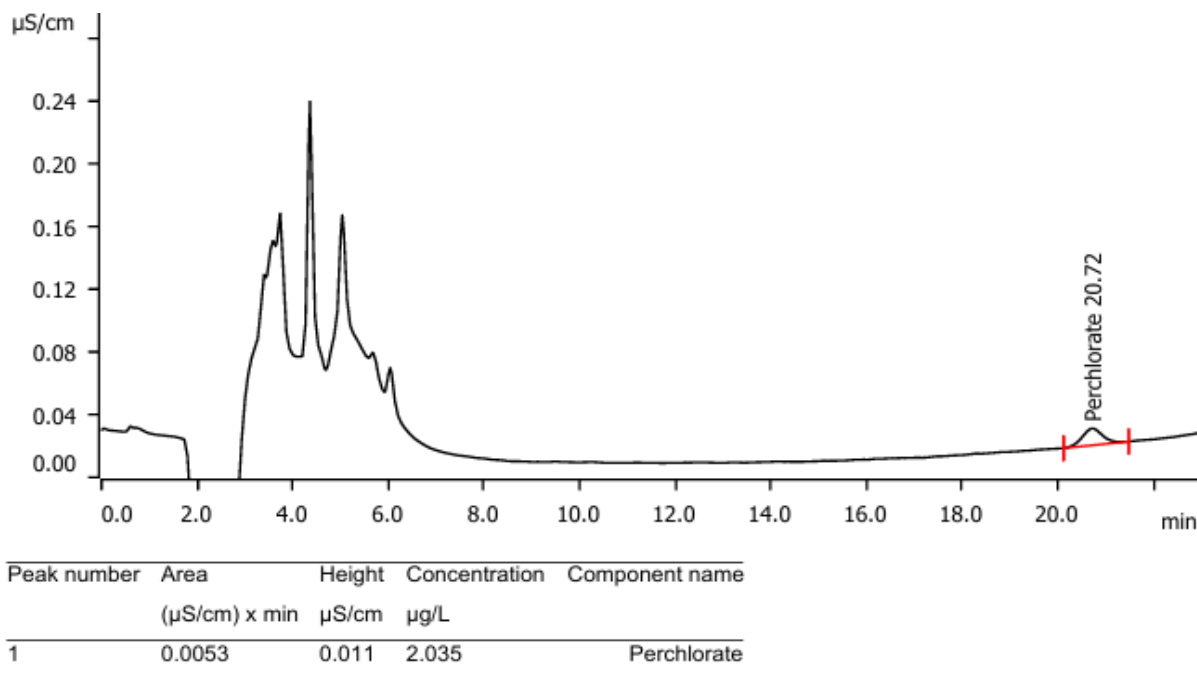


Figure 2. Standard 2 (2 ppb perchlorate)

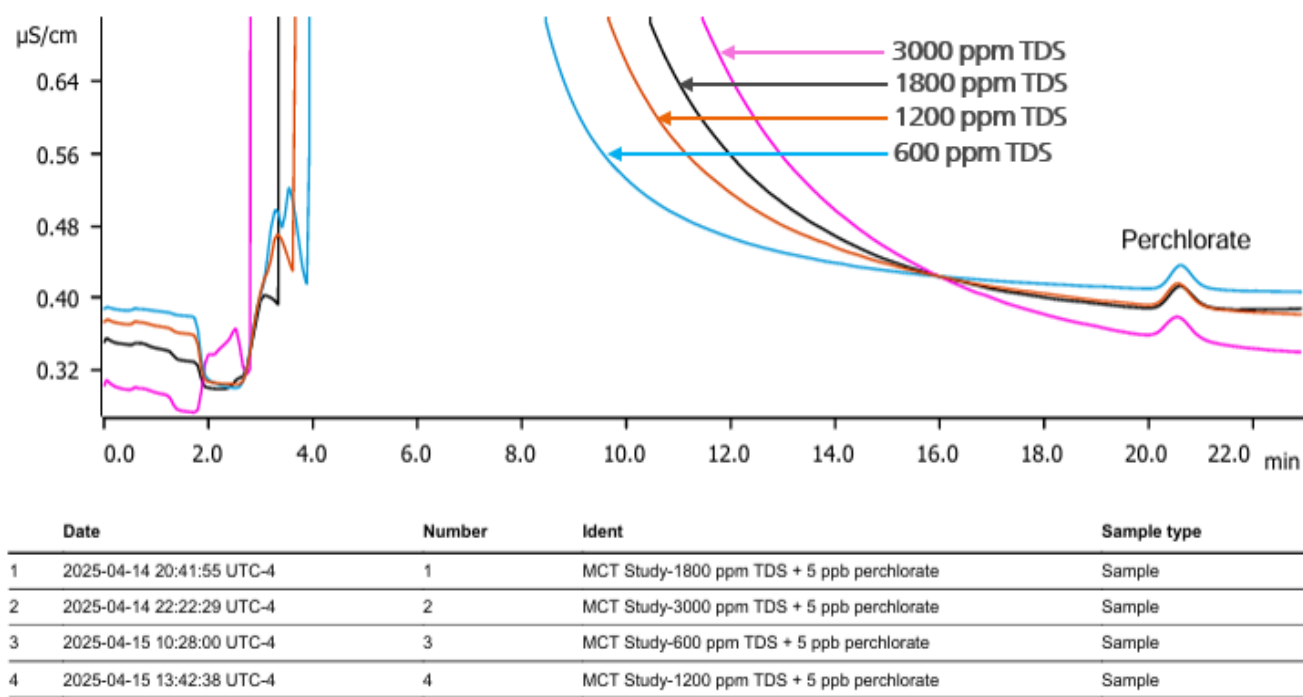


Figure 3. MCT Study

CONCLUSION

A new method using hydroxide as the eluent was developed for the determination of perchlorate in water. This method does not require any organic solvents in the eluents and is free from interferences from high concentrations of chloride, carbonate, and sulfate.

Analyte:	Perchlorate
Matrix:	Water
Method:	Ion chromatography
Industry:	Water
Standards:	USEPA 314.0