Trace Level Analysis of Perchlorate and Bromate in Various Water Matrices using Suppressed Ion Chromatography

Dr. Jay Gandhi, Dr. Stuart Procter

Summary

- Perchlorate salts are used as "rocket fuel" in electroplating and other industries; yet, in 1999 a CAEN article it was scientifically proven to inhibit the human thyroid gland’s absorption of iodine — which, in turn, may cause thyroid-related diseases. Perchlorate detection/analysis in varying water matrices can be a challenge. How do we overcome the obstacles?
- Bromide is ubiquitously found in drinking water. It is introduced into source water either by contact with bromide-containing soils or seawater having a high bromide content. Bromide converts into bromate during water disinfection, for example by ozonation. Bromate can also enter drinking water when sodium hypochlorite is used as disinfectant.

Perchlorate Analysis

a) Suppressed Ion Chromatography (Perchlorate with Dual4 column)

USEPA method 314.0 enhanced:

- Anionic functionalized Monolith column specially designed for Perchlorate analysis.
- Column: Metrosep Dual4 - 100
- Column temp.: 45 °C
- Eluent: 5.5 mmol/L LiOH + 10mM 4-Cyanophenol
- Flow: 1.5 mL/min
- Loop: 1000 µL
- Detection: Sequential Suppressed Conductivity

b) Suppressed Ion Chromatography (Perchlorate with ASUPP7 column)

Confirmation column – High Resolution Anion Exchange column

- Column: Metrosep ASUPP7 - 250/4.0
- Column temp.: 45 °C
- Eluent: 10.5 mmol/L Na₂CO₃ + 25% Acetate
- Flow: 0.7 mL/min
- Loop: 1000 µL
- Detection: Sequential Suppressed Conductivity

USEPA Method 314.0 – MCT study data

- Column: Metrosep Dual 4 – 100 mm
- Element: 50 ppb Cl⁻, HCO₃⁻, SO₄²⁻
- Flow: 1.5 mL/min
- Loop: 1000 µL
- Detection: Sequential Suppressed Conductivity

Bromate Analysis

The International Agency for Research on Cancer (IARC) has classified bromate as a possible carcinogen. Based on renal cell tumors in rats, a concentration of 3 µg/L bromate corresponds to an excess lifetime cancer risk of 1·10⁻⁵. Due to the difficulty of bromide detection/analysis in varying water matrices can be a challenge. How do we overcome the obstacles?

- Bromide is ubiquitously found in drinking water. It is introduced into source water either by contact with bromide-containing soils or seawater having a high bromide content. Bromide converts into bromate during water disinfection, for example by ozonation. Bromate can also enter drinking water when sodium hypochlorite is used as disinfectant.

Conclusion:

In 2004, Metrohm developed and provided functionalized monolith column for Perchlorate analysis. Even though Perchlorate is inorganic anion, it behaves like organic molecule (hydrophobic), hence monolith column provides platform to wash off hydrophilic matrix ions without distorting peak symmetry at lowest possible concentration of Perchlorate in matrix of 3000 parts per million Total Dissolved Solids (TDS). Hence, as Matrix ions specially Chloride, Carbonate and Sulfate are increased, it becomes difficult to measure Perchlorate at trace level by Ion Chromatography and suppressed conductivity detection.

This poster presentation demonstrate trace level (1 parts per billion) Perchlorate analysis in High Ionic Matrix (3000ppm TDS) where 3000ppm TDS = 1000ppm each of Chloride, Carbonate and Sulfate ions. Sodium Salt of these ions was used to prepare standards.

Sample preparation

Typical samples may be in various phases (aqueous and organics), solid material like mud and particles. It may require centrifugation, filtration etc. before the ion analysis. Metrohm provides automated in-line sample filtration device that eliminates use of syringe filters for these type of samples.