

Determination of Trace Level Perchlorate by Ion Chromatography–Mass Spectrometry

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Perchlorate salts are being used as rocket propellants in fireworks, and in electroplating industry. Currently, perchlorate is being monitored under USEPA Un Regulated Contaminant Rule (UCMR-1) at 4.0 parts per billion (USEPA method 314.0). Recently, it is believed by the scientific community that perchlorate hinders the iodine absorption ability of the thyroid gland, posing higher health risk for the public. In June 2004, the National Academy of Science (NAS) is to release a report on the accurate health risk of perchlorate. As per EPA method 314.0, it is easy to monitor perchlorate at the 4.0 ppb level. As common anions such as carbonate, chloride and sulphate increase in ground water, it is more difficult to consistently monitor low-level perchlorate. To overcome this, a mass spectrometer was combined with IC, which gives better sensitivity. USEPA Region-VI, Metrohm-Peak and Agilent Technologies performed a collaborative study for trace-level perchlorate in high ionic waters.

Experimental Conditions

The Metrohm Advanced Modular ion chromatography system with Agilent 1100 series MSD is used for the analysis. The Model 819 conductivity detector, the 818 dual piston IC pump, the 788 autosampler, the 820 IC separation centre, the 833 MSM module, and the IC-Net 2.3 are also used. A MetroSep ASUPP5 (4 × 100 mm) column is employed for this analysis. Eluent = 30 mM NaOH + 30% methanol, with flow-rate of 0.7 mL/min.

Results and Discussion

Perchlorate was monitored for $m/z = 99$ and $m/z = 101$ at a ratio of 75:25 percent (natural abundance of chlorine isotope 35/37).

Figure 1 is a demonstration of a high ionic matrix containing 1000 ppm each of chloride, carbonate and sulphate fortified with 1 ppb perchlorate ion. Signal-to-noise ratio at 1 ppb indicates a calculated detection limit around 60 parts per trillion. A series of experiments demonstrated zero carryover, and no matrix effect at MSD interface. There is a choice of column geometry that can be used. 4 mm columns permit the use of different loop sizes. For this experiment 50 μL , 100 μL and 250 μL loops were tested with the 100 μL loop giving the desired results. A different matrix concentration fortified with 1 ppb perchlorate resulted in recoveries of 88–110%. Another interesting fact is that there was very minimal bisulphate background (m/z 5 99) observed.

Conclusion

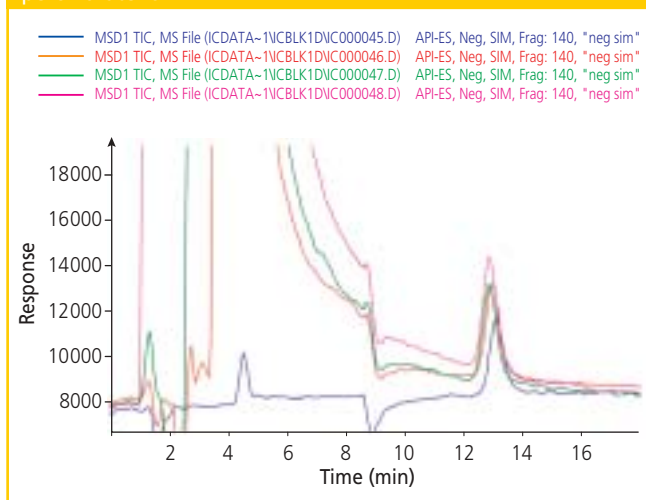
Using 4 mm ID column and no flow splitting trace level detection of perchlorate by IC–MS was achieved. Virtually no ionic suppression was observed at the MSD interface. Sequential blank after 500 ppb perchlorate standard demonstrated zero carryover. Affordable, simple, rugged single quad IC–MS systems are available for the analysis of perchlorate and other analytical needs of the high throughput laboratories.

References

Metrohm Application Works AW-US6-0066-122003 US EPA method 314.0.

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Figure 1: TIC of high ionic matrix containing 1000 ppm of chloride, carbonate and sulphate fortified with 1 ppb of perchlorate ion.



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