

Determination of Trace Level Perchlorate in Different Vegetables Extracts by Ion Chromatography–Mass Spectrometry

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Perchlorate salts are being used as rocket propellants in fireworks and in the electroplating industry. Currently, there is a major concern for bio-accumulation of perchlorate in major vegetable and fruit crops. The farming industry is monitoring perchlorate in crops of lettuce, spinach, green onions, lemon (citrus fruits), alfalfa and strawberries among others. It is evident that any vegetable which has high water content will accumulate more perchlorate. There are several challenges for the analysis by ion chromatography and conductivity detection for these types of sample matrices. However, this problem is solved by a Metrohm ion chromatograph hyphenated with an Agilent (Wilmington, Delaware) mass spectrometer.

Experimental Conditions

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

Results and Discussion

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

Figure 1 is a demonstration of extracts of various vegetables such as extracts of lettuce, green onions, spinach, lemon, broccoli, and alfalfa. Also, the same extracts were fortified with 2–5 ppb of perchlorate. Spike recoveries were around 90–95% for each sample matrix. A green onion sample matrix can be especially challenging as it contains high quantities of mercaptans. Mercaptans show similar response to that of perchlorate on anion exchange columns.

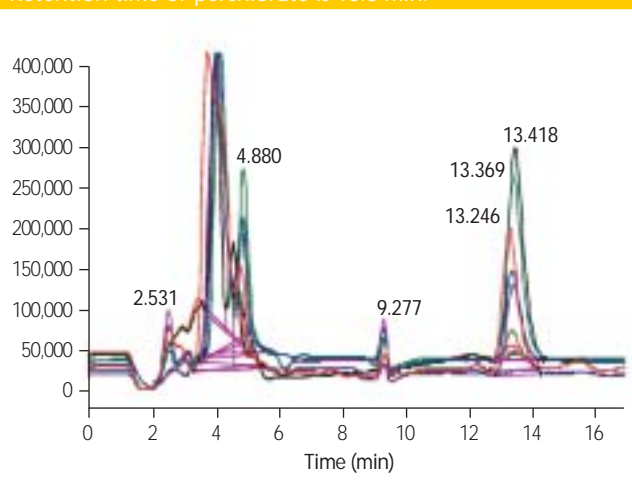
Conclusion

Metrohm's superior suppressor technology and industry standard Agilent Mass Spectrometer enabled flawless detection of Perchlorate in various vegetable extracts. There was virtually no ionic suppression observed at the MSD interface. This analysis can be complimented by using ultra filtration technique on the auto sampler to minimize usage of sample filter cartridges.

References

1. Metrohm Application Works AW-US6-0066-122003.
2. LCGC 22(2).

Figure 1: TIC of sample and sample spikes. Samples = extracts of lettuce, green onions, spinach, lemon, broccoli and alfalfa. Spike = extracts fortified with 2 to 5 ppb perchlorate. Retention time of perchlorate is 13.8 min.



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