



## Perchlorate Analysis by LC/MS/MS Using the Varian 1200L Triple Quadrupole Mass Spectrometer

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### Introduction

Perchlorate is a water-soluble anion that is a strong oxidizer. It is commonly used in munitions, rocket propellants, road flares, air bags, and some fertilizers. Perchlorate is stable at room temperature, but if it finds its way into the human body, it is toxic and negatively affects thyroid function.

In 2003, it was reported that perchlorate from a plant in Las Vegas contaminated the Las Vegas wash, which subsequently led to contamination of Lake Mead and the Colorado River.<sup>1</sup> This contamination posed large environmental and health risks, as the Colorado River supplies water that is used to irrigate farmland and drinking water. The drinking water affected by this contamination spanned from Las Vegas, NV, to San Diego, CA, and into Tucson, AZ. Produce irrigated by the contaminated water source included lettuce, alfalfa (which can lead to cow's milk contamination), dates, green beans and zucchini.

The method presented herein for the analysis of perchlorate uses the Varian 1200L triple quadrupole mass spectrometer, which is a powerful instrument due to its high selectivity and sensitivity.

### Instrumentation

- Varian 1200L triple quadrupole mass spectrometer with an ESI source
- Varian ProStar™ 210 binary solvent delivery modules

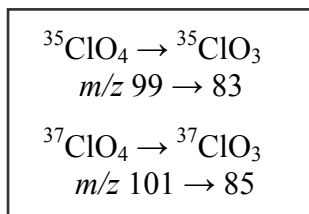


Figure 1. Monitored transitions for perchlorate analysis. Each chlorine isotope produced a unique MRM transition which may be monitored simultaneously and used for quantitation.

### Experimental

Water samples were filtered and treated to remove free chlorine and sulfate. The produce samples were homogenized, sonicated with water and then filtered. They were also treated to remove free chlorine.

### HPLC Conditions

Column: Dionex AS-16, 250 x 2.0 mm  
Mobile Phase: A: 65mM KOH  
B: Methanol  
LC Program: Isocratic Flow 300  $\mu\text{L}/\text{min}$   
60:40 A:B  
Dionex AMMS suppressor regenerant:  
72mM  $\text{H}_2\text{SO}_4$

### MS Parameters

Ionization Mode: ESI Negative

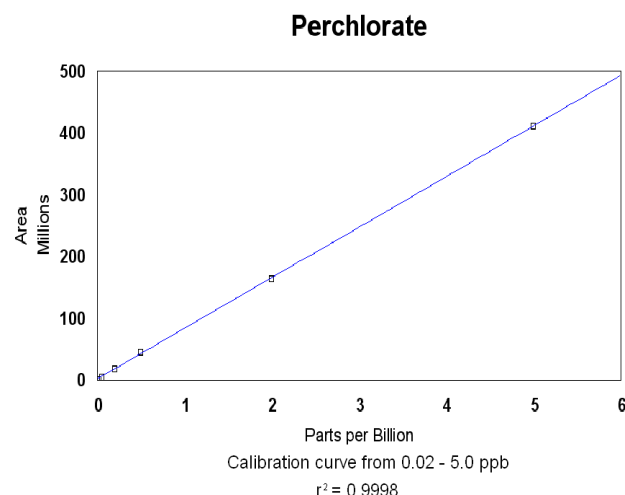


Figure 2. Calibration curve for perchlorate. The curve was found to be linear from 0.2 to 5 ppb, with  $r^2=0.9998$ .

### Results

The quantitation of perchlorate was found to be linear from 0.2 to 5 ppb with  $r^2=0.9998$  (Figure 2).

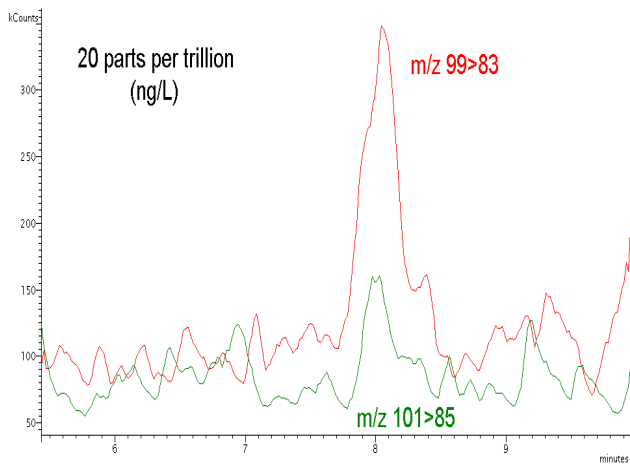


Figure 3. Chromatogram of perchlorate standard at 20 parts per trillion (ppt). The <sup>35</sup>Cl transition is shown in red (larger trace) and the <sup>37</sup>Cl transition is shown in green (smaller trace).

The detection limit for the perchlorate analysis was determined by analyzing seven replicates of a 50-ppt standard. The average concentration of the seven replicates was found to be 47.9 ppt with an RSD value of 9.3%. Based on these data, the detection limit was then calculated to be 14 ppt for the perchlorate analysis. A Multiple Reaction Monitoring (MRM) trace for perchlorate transitions at 20 ppt is shown in Figure 3.

Table 1. Quantitation results for real samples. (Water: n=4, Dates: n=3, Others: n=5)

Sample	Perchlorate Content (ppb)	%RSD
Tap Water (El Centro)	4.20	4.4
Tap Water (SFS*)	1.89	5.4
Celery	4.81	6.3
Zucchini	11.6	8.7
Green Beans	28.2	4.5
Dates	36.9	16
Date Sugar	27.9	14

\* Santa Fe Springs (SFS)

Tables 1 and 2 show the precision and accuracy of the method in various matrices. Figures 4-7 are MRM traces for perchlorate in drinking water, tap water, celery and date sugar matrices. All show excellent peak shape and precision, free of any matrix interferences.

Table 2. Percent recovery for spiked samples.

Matrix	Endogenous Perchlorate (ppb)	Amount Spiked (ppb)	Amount Recovered (ppb)	Recovery %
Tap Water (El Centro)	4.20	5.00	8.88	94
Zucchini	11.6	40.0	54.4	107
Celery	4.81	40.0	40.5	89

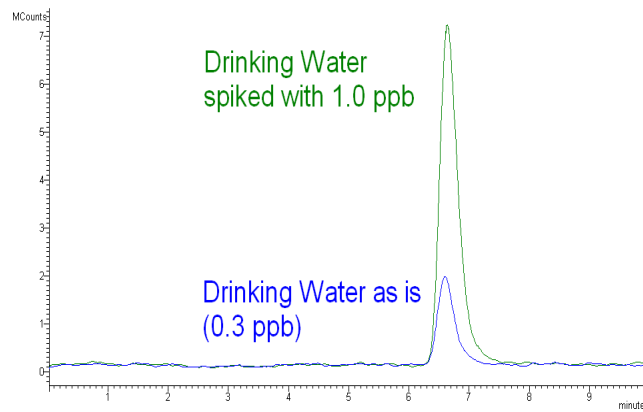


Figure 4. Drinking water spiked with 1.0 parts per billion (ppb) perchlorate (larger trace) and before perchlorate spike (smaller trace).

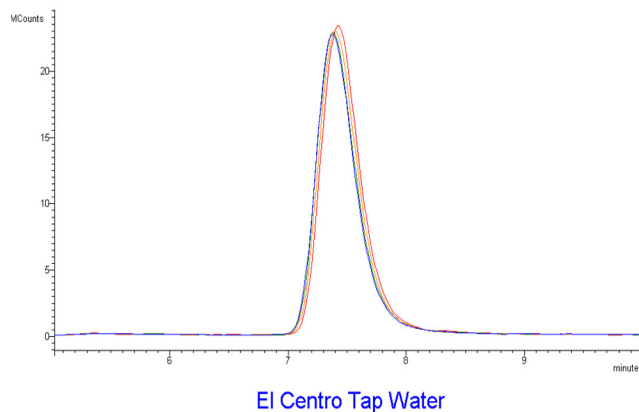


Figure 5. Chromatogram for duplicate perchlorate analyses in El Centro Tap Water (n=4). The quantitated amount was found to be 4.20 ppb.

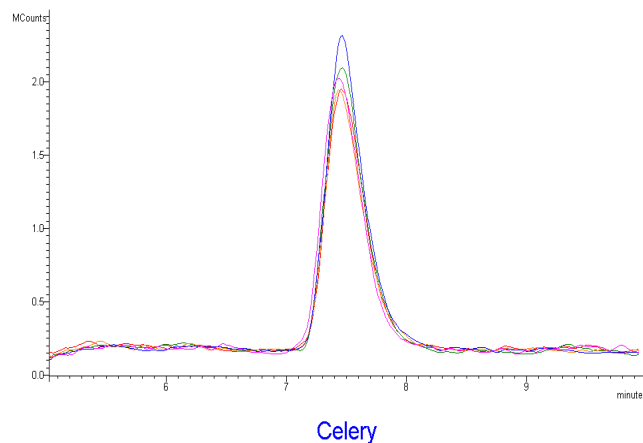


Figure 6. Perchlorate analysis in celery (n=5). The celery was found to contain 4.81 ppb perchlorate.

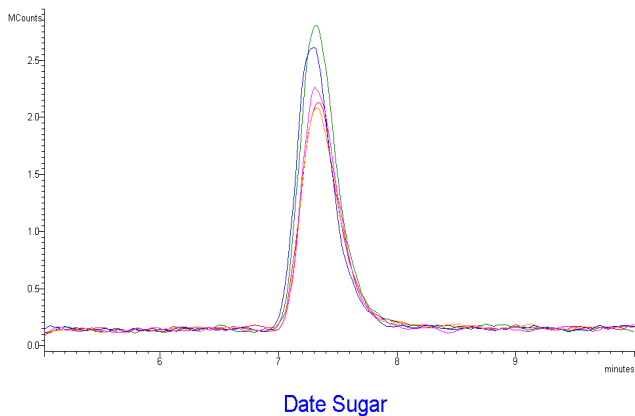


Figure 7. Perchlorate analysis in date sugar (n=5). Perchlorate content in date sugar was found to be 27.9 ppb.

### Conclusion

The Varian 1200L triple quadrupole mass spectrometer is an extremely sensitive instrument for perchlorate analysis in a variety of matrices. The method is fast, robust and sensitive. In the samples studied for this analysis, the detection limit for perchlorate was found to be 14 ppt, and the calibration curve for quantitation of perchlorate was found to be linear from 0.2 to 5 ppb.

### References

1. Article at : <http://www.ncbi.nlm.nih.gov/pubmed/10953814>

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