EXPERIMENTALLY DETERMINED HOLDING TIMES FOR ENVIRONMENTAL SAMPLES CONTAINING LOW LEVELS OF PERCHLORATE

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ABSTRACT

As in book of abstracts:

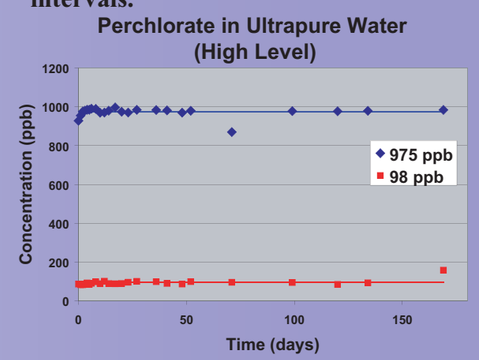
Perchlorate, a component in rocket fuels, fireworks, and some fertilizers, has been detected in many water supplies throughout the country. Because of its presence in drinking waters such as Lake Mead, the Colorado River, and various drinking water supplies, perchlorate has become a significant public health concern. It has been placed on the US EPA's Contaminant Candidate List, the list of potentially harmful drinking water contaminants for which more data are needed. Reliable, sensitive methods for detecting perchlorate in drinking water, food, and tobacco are presently under development. However, the stability of perchlorate in water samples has not been thoroughly examined, so that scientifically defensible holding times of perchlorate-bearing samples and standards are unknown. In this study the long-term stability of perchlorate standards in deionized water and in a common water matrix are determined. Sets of samples containing approximately 1000, 100, 10, and 0 µg/ml perchlorate in deionized water and also in local tap water were formulated. These samples were analyzed for perchlorate concentration against freshly prepared standards every 24 hours for the first seven days, biweekly for the next four weeks, and weekly after that for a total of 25 weeks. They were analyzed using a Dionex ion chromatograph system with AG16/AS16 columns at a flow rate of 1.25 ml/min, 35 mM KOH eluent, and 500 µl injection loop. The results for this 6-month study will be presented with a recommendation for maximum holding times of perchlorate samples after collection.

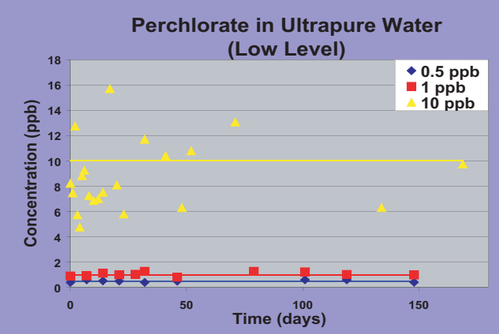
INTRODUCTION

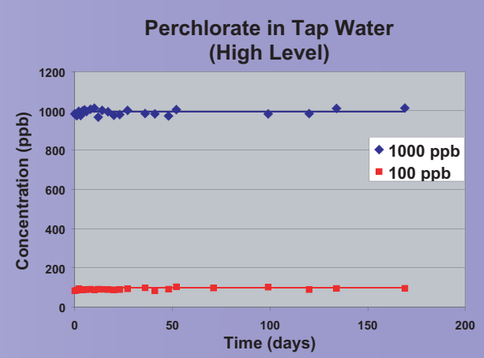
- ClO_4^- has recently been found in many water sources at high concentrations.
- Efforts are underway to determine the extent of ClO_4^- contamination nationwide.
- Until now, holding time studies have only been performed to 109 days with 50 µg/L ClO_4^- solution (Tsui et al., 1998).
- Holding time studies at low concentrations of ClO_4^- and for samples in a matrix other than ultrapure water do not exist.
- This study shows the long-term stability for 0.5 to 1000 ppb ClO_4^- samples in ultrapure and tap water.

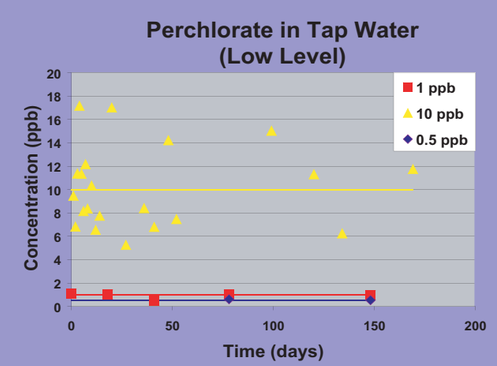
METHODS

- Samples containing ~ 1000, 100, 10, 1.0, 0.5, and 0 µg/L ClO_4^- in deionized and local tap water were formulated.
- The 1.0 and 0.5 ppb ClO_4^- tap-water samples in were pretreated with Dionex OnGuard Ba, Ag, and H cartridges to remove matrix interferences.
- These were stored at room temperature. For ~8-12 hours per day they were exposed to fluorescent lighting.
- Samples were analyzed by ion chromatography with conductivity detection. After the first few weeks of daily analyses, they were analyzed at ~2 week intervals.









CONCLUSIONS

- ClO_4^--containing waters are stable at room temperature for >169 days at high concentrations (10-1000 ppb) in both deionized and tap water matrices.
- At low concentrations (0.5-1 ppb) ClO_4^- in both ultrapure and tap water matrices are stable for >148 days.
- There is a large amount of scatter in the 10 ppb samples due to a small signal to noise ratio using a 4-mm column system. The last data point was run using a 2-mm column system which yielded a higher signal to noise ratio and therefore better sensitivity, precision, and accuracy.

RECOMMENDATIONS

- While ClO_4^- is stable for at least 169 days at room temperature, samples should be run in a timely manner.
- If it is necessary to store ClO_4^- samples for longer than the EPA recommended time, the ClO_4^- concentration will not decrease.

REFERENCES

Tsui, D.T.; Narayanan, L.; Mattie, D.; Stability and Concentration Verification of Ammonium Perchlorate Dosing Solutions; August 12, 1998; http://www.epa.gov/safewater/ccl/perchlorate/tsui3.html

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