SFUND RECORDS CTR
99872

Analysis of Perchlorates by LC/MS/MS

By
Paul C. Winkler, Ph.D.
Acculabs, Inc.

26 October 2001

The accurate determination of perchlorate at low ppb to sub ppb concentrations by ion chromatography is difficult due to an inherent limitation of the sensitivity of the method and also to the inherent lack of specificity of the method. The possibility of a false positive detection is very likely as a result of these limitations. In an effort to provide a better analytical method that will allow for the accurate quantitation of perchlorate, an LC/MS/MS method using electrospray ionization has been developed.

The two main advantages of the LC/MS/MS method for perchlorate analysis are that it is more sensitive than the IC method and it is far more specific than the IC method. A compound must have the correct retention time, it must form an ion at the correct parent mass as perchlorate and that ion must fragment to the correct daughter ion in order for a signal to be observed. This results in a great decrease in the probability of a false positive result and in better sensitivity due to a reduction in background signal.

The data that is presented in the perchlorate document is from an MDL study that was spiked at a concentration of 5μg/L. The calculated method detection limit from this set of data is 1.87μg/L and the Practical Quantitation Limit (PQL) was set to 4μg/L. The PQL was set to 4μg/L because that is what was required by the clients who were requesting perchlorate analysis. A review of the data indicates that the signal to noise of the MDL is least 10:1 and the actual instrument detection limit is lower than the present MDL of 1.87μg/L. Because this method is a direct aqueous injection method, the instrument detection limit may also be considered to be the method detection limit. The instrument sensitivity is capable of detecting concentration levels of perchlorate at 0.5μ g/L. A new MDL study that is spiked at a concentration of 0.5μg/L is underway and is expected to result in a PQL of approximately 1ppb.