

## PERCHLORATE TESTING GUIDANCE

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Perchlorate testing has been generally carried out by using ion chromatography (IC). For water, the most commonly used method is EPA 314.0, which was promulgated for water testing. A similar method is EPA 9058 which was written for surface water, mixed domestic water, and industrial wastewaters. The EPA 9058 quality control requirements appear to be not as stringent as EPA 314.0. The two methods for perchlorate analysis use a retention time window for identification of perchlorate.

Since the identification of perchlorate relies on the retention time of a sample peak, a false positive result may occur. This happens when a substance has a retention time close to or identical to the retention time of perchlorate and falls within the retention time window established for the identification of perchlorate. EPA 314.0 recommends a retention time window size of +/- three times the standard deviation of the average retention time, but should not extend beyond +/- 5% of the average retention time for perchlorate. Similarly, EPA 9058 states that +/- three times the standard deviation of the average retention time can be used to calculate a suggested window size.

To improve the reliability of a positive perchlorate detection in an environmental sample, the following guidance is provided:

1. For the analysis of water samples, EPA 314.0 can be used. For the analysis of surface water, mixed domestic water, and industrial wastewaters, EPA 314.0 or EPA 9058 (EPA 9058 November 2000 version is available from <http://www.epa.gov/SW-846/pdfs/9058.pdf>; and EPA 9058 January 2004 version is available upon request) can be used.
2. For the analysis of soil samples, an extraction step is necessary prior to testing with EPA 314.0 or EPA 9058.
3. At a minimum, the following quality assurance/quality control samples should be included:
  - a. Method Blank
  - b. Laboratory Control Sample
  - c. Duplicate Sample (or Spiked Duplicate Sample as appropriate)
  - d. Spiked Sample
  - e. Field Blank
  - f. Field Duplicate

The above mentioned quality control samples should be analyzed at a frequency of a minimum of 5% of the samples being analyzed, per batch of samples, or per sample matrix, whichever is more frequent. Some or all of the quality control samples may be omitted if the analytical method being used already covered such quality control samples analyses with equivalent or more stringent requirements. A differently named quality control sample may be equivalent to one of the quality control samples discussed above provided the differently named quality sample serves the same purposes. For example, a method standard sample (spiked blank) may be equivalent to a laboratory control sample under some circumstances.

4. All the quality assurance/quality control protocols and other requirements specified in a method being used for perchlorate analysis should be followed. If a specified protocol is not followed, the modification should be agreed to by the client, and a justification for the deviation should be included in the report.
5. For any sample positive with perchlorate, the presence of perchlorate in the positive sample should be confirmed. As a preliminary and inexpensive measure, a positive perchlorate detection can be tested with a separate sample portion spiked with a known concentration of perchlorate near the suspected sample perchlorate concentration. The appearance of two peaks indicates that perchlorate is not detected while a resulting simple peak supports the presence of perchlorate. This preliminary measure may be useful to demonstrate the absence of perchlorate when the spiked sample yields two or split peaks. It may not be sufficient to confirm the presence of perchlorate when the spiked sample yields a simple peak because the interfering substance may have an identical retention time as the perchlorate.

Other useful confirmation techniques, as recommended in EPA 9058, include further analysis using a second column with dissimilar ion chromatographic conditions, ion chromatography/mass spectrometry (IC/MS), liquid chromatography/mass spectrometry (LC/MS), liquid chromatography/mass spectrometry/mass spectrometry (LC/MS/MS), or ion selective electrode testing. One potential interfering substance for IC testing is p-chlorobenzene sulfonic acid (p-CBSA) and its salts; p-CBSA is a by-product of DDT manufacture, and may be present near landfills which received wastewaters from DDT manufacturers. If p-CBSA is suspected in the area of sampling, a modified IC method, using a modified mobile phase and longer elution time, or another confirmatory technique, should be used to distinguish perchlorate from p-CBSA. For brackish water samples, potential interference may require sample dilutions before IC testing. If so, the reporting limit for perchlorate will be raised.

A confirmation by a technique other than MS may be more economical but a confirmation by an MS technique yields more reliable data. Confirmation with an MS technique should be considered for a new finding of perchlorate in an area.

The need for the continuing confirmation testing for perchlorate positive samples from the same sampling location should be determined by the project requirements.

**NOTE :** This Guidance is subject to change because of the emerging advances in technology. For its current status, please contact:

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