

"Sample Holding Times and Validity of Analytical Results"
(Key Words: Hazardous waste definition; sample; testing;
toxicity characteristic)

QUESTION: When characterizing waste as hazardous or nonhazardous under RCRA Subtitle C, Section 262.11 provides that a generator has the option of either applying knowledge of the hazardous characteristics of the waste or testing the waste. If the generator chooses to test a waste to determine if it exhibits the toxicity characteristic under 40 CFR Section 261.24, the Toxicity Characteristic Leaching Procedure - Method 1311 (TCLP) must be used to generate a waste extract. This extract is then analyzed for the 39 constituents listed in Table 1 of Section 261.24. To ensure that accurate results are obtained, there are specific quality control measures for the extraction and analysis procedures, including limits on the amount of time samples can be held during testing. When these sample holding times are exceeded, can the results of constituent analysis on a TCLP extract still be used to determine if a waste exhibits the toxicity characteristic?

ANSWER: When sample holding times are exceeded, TCLP analytical results will be considered the minimum amount that could leach from the waste; an identical sample analyzed within the prescribed holding times might yield higher concentrations of toxicity characteristic constituents (Part 261, Appendix II, Section 8.4). EPA's manual Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW-846, Second Edition) defines sample holding time as the storage time allowed between field collection of a sample and completion of analysis in a laboratory. Specific maximum holding times are set for quality assurance and quality control purposes. Certain constituents in waste, such as volatile organic compounds, can degrade or volatilize over time. When constituents in a sample are lost through these natural chemical processes, analytical measurements become inaccurate. To limit sample degradation, maximum holding times are developed based on the propensity of the waste constituents to degrade or volatilize. General guidelines for sample holding times are presented in Volume 1, Chapters 2, 3, and 4 of SW-846, and additional method-specific requirements may appear in the individual test methods themselves. Outlined in section 8.4 of Method 1311 (Part 261, Appendix II), maximum TCLP sample holding times range from a total of 28 days for volatiles

to 360 days for metals. These overall time frames are further broken down, specifying the length of time allowed for each step in the analytical process. For example, volatile samples are allowed 14 days for leachate extraction and another 14 days for constituent analysis, while the breakdown for semivolatiles allows 14 days for leachate extraction, 7 days for extract preparation, and 40 days for constituent analysis. When sample holding times are exceeded, measurements may be inaccurate and the TCLP analysis may be invalid or inconclusive.

When analysis is conducted after a sample holding time is exceeded, however, the results may still have limited applicability. Because some constituents are lost through volatilization or degradation while awaiting testing, constituent concentrations in expired samples will be lower than if the sample were fresh. If a sample exceeds a recommended holding time and analysis demonstrates that concentrations are above the regulatory threshold for one or more constituents, then these concentrations can be treated as minimum values and the waste is hazardous for the toxicity characteristic. No further testing is required. If, on the other hand, a sample exceeds a recommended holding time and analysis demonstrates that concentrations are below the regulatory threshold for one or more constituents, further testing may be necessary to demonstrate that the waste is nonhazardous. If the generator chooses to conduct further testing, additional samples would be required to ensure accurate measurement of constituents and to provide a definitive waste determination under RCRA Subtitle C. (June 1993 Monthly Hotline Report)