ANALYSIS OF TCLP EXTRACTS BY X-RAY FLUORESCENCE

Douglas S. Kendall, Benjamin A. Burns, and Jennifer A. Suggs
National Enforcement Investigations Center, U.S. Environmental Protection Agency,
Building 25, Denver Federal Center, Denver, CO 80225

The toxicity characteristic leaching procedure (TCLP) is one test used by the U.S. Environmental Protection Agency (EPA) to determine if a waste material is hazardous; the TCLP test is widely used by the environmental testing community.

Liquid wastes are analyzed as received. Solid wastes are extracted with a dilute acetic acid solution in a 20-to-1 liquid-to-solids ratio. The liquid waste or the extract of the solid waste is then analyzed for up to 40 constituents. These constituents include a number of organic solvents and pesticides and eight elemental determinations. The elements of concern for toxicity are arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver. Regulatory limits for these elements vary. The regulatory limit is 0.2 milligrams per liter (mg/L) for mercury; 1.0 mg/L for cadmium and selenium; 100 mg/L for barium; and 5.0 mg/L for the others.

Seven elements are usually determined by inductively coupled plasma optical emission spectroscopy (ICP/OES) or ICP mass spectrometry (ICP/MS). Determination of mercury is performed using specific mercury analysis instrumentation with atomic absorption or atomic fluorescence detection. As an environmental forensic laboratory, the National Enforcement Investigations Center (NEIC) of EPA often conducts TCLP testing. It is our practice to confirm TCLP test results that may indicate a violation of a regulation. This project explored the use of X-ray fluorescence (XRF) as a confirmation technique for the determination of inorganic elements in TCLP extracts. Except for mercury, which has the lowest regulatory limit and is best determined using specialized instruments, the elements of interest in TCLP extracts can be determined by XRF.

The sample preparation approach investigated was a variation of the droplet on filter paper technique. The particular proprietary version used consisted of filter paper attached in the center of plastic film surrounded by an outer plastic support ring. Up to 50 micro liters at a time of TCLP extract could be placed on the filter paper, and then dried at 50 degrees Celsius (°C). Additional portions of sample could be placed on the filter paper, with drying in between each addition. The sample carriers were analyzed under vacuum with a conventional wavelength dispersive XRF spectrometer.

Results to be presented include single-element and multi-element calibrations, detection limit studies, linear ranges, studies of matrix effects from extraction buffers, and interference studies. The analysis of real TCLP extracts will be discussed, as well as comparisons with ICP results. The advantages and disadvantages of XRF for TCLP elemental determinations will be discussed.