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Session III - Leaching Science

Overview of Current Approaches - Sara Harwell

Sara Hartwell, Science Applications International Corporation, provided an overview of the leaching tests currently in use. A copy of Ms. Hartwell's presentation materials is available through the following link: hartwell1.pdf. She noted that batteries of leaching tests have been proposed or promulgated not only by the US, but by France, the Netherlands, Germany, Sweden, Denmark, the European Union and Canada. Both the government and private sector have developed and incorporated leaching methods into their programs. These methods go way beyond the single-batch testing protocol of the TCLP, and address additional waste management scenarios, leaching properties and waste types. Tests have been developed to account for variability in the ratio of leaching fluid to solid waste, chemical composition of the leaching fluid, testing of monolithic and granular wastes, as well as stabilized and solidified wastes, and to address radioactive wastes. Within the US, standardized leaching protocols have been published by the American Society for Testing and Materials, the International Atomic Energy Agency, the US Army, ANSI/ANS, and the ISO.

Leaching test methods can be divided into 2 general categories: static extraction tests and dynamic extraction tests. In static extraction protocols, leaching takes place with a single, specified volume of leaching fluid. In dynamic extraction protocols, the leaching fluid is renewed throughout the test. In static extraction protocols, a specific amount of leaching fluid is placed in contact with a specific amount of waste for a specified length of time, with no replacement of, or addition to, the leaching fluid. The leachate from the test is removed at specified times, usually the end of the test, for analysis. Static extraction protocols assume that a steady state condition is achieved by the end of the test. Types of static extraction tests include: agitated and non-agitated extraction tests, sequential chemical extraction tests, and concentration buildup tests.

In agitated extraction tests, the waste and leachant are co-mingled and agitated, so the test reaches steady-state conditions as quickly as possible. Test protocols frequently incorporate particle size reduction steps to increase the amount of surface area available for contact, thereby reducing the amount of time required to reach a steady state condition. Agitated extraction tests measure the chemical properties of a waste/leachant system rather than the physical, rate-limiting mechanisms. Because of this, this type of test may over-estimate the short-term release of constituents. Examples of agitated extraction tests include the TCLP, EP Tox, California's WET, and the SPLP.

In non-agitated extraction tests, the waste and leachant are co-mingled and <u>not</u> agitated. Non-agitated extraction tests measure the physical, rate-limiting mechanisms, rather than the chemical properties of a waste/leachant system. This type of test is based on the assumption that the physical integrity of the waste matrix affects how much material can be leached out of the matrix. Non-agitated extraction tests take longer to reach steady state conditions than the agitated tests. Example of non-agitated extraction tests include the Strategic Leach test and High Temperature Static Leach test, both from Canada.

A sequential chemical extraction test is a series of agitated extraction tests, using increasingly aggressive leaching fluids. There are two ways to set up a sequential extraction test: you can set up multiple tests, each one with a different leaching fluid - this assumes that each leaching fluid will extract what the less aggressive fluid extracted, plus some additional contaminant or quantity. Alternatively, the same aliquot of waste can be subjected to each successive leachant - this version assumes that each successive leachant will extract something that the previous leachant could not . In either case, the hypothesis is that increasing chemical aggressiveness will result in increasing amounts of extracted contaminants. An example of a sequential chemical extraction test is ASTM's Sequential Batch Extraction of Wastes with Acidic Extraction Fluid. Other sequential extraction tests are being developed.

Protocols for concentration build-up tests call for aliquots of the waste to be contacted repeatedly with the same leachate, at a very low liquid to solid ratio. This type of test models a volume of fluid flowing through a body of waste, with the concentration of extracted contaminants building up in the leachate as it progress through the system. These tests may be a good simulation of the pore-water composition of a waste. An example of a concentration build-up test is Wisconsin's Standard Leach Test, Procedure C.

In dynamic extraction protocols, the leaching fluid is renewed, either continuously or intermittently, to drive the leaching process. Because the physical integrity of the waste is usually maintained during the test, and the information is generated as a function of time, dynamic extraction tests provide information about the kinetics of contaminant mobilization. In general, dynamic extraction tests can be categorized as: serial batch tests, flow-around tests, flow-through tests, and Soxhlet tests.

In a serial batch test, a portion of a crushed, granular sample is mixed with leachant and agitated for a specified time period. At the end of the time period, the leachate is separated, fresh leachant added, and the process repeated until the desired number of leaching periods has been completed. The concentrations of contaminants measured in the serial leachates can provide kinetic information about contaminant dissolution. Examples include the Multiple Extraction Procedure (SW-846 Method 1320); the Availability Test and Serial Batch Test from the Netherlands, and the US Army's Graded Serial Batch test.

Flow-around tests use either monolithic samples, or samples that are somehow contained. The sample is placed in a test vessel, with space around the sample, and leachant is added so that it flows around the sample. The leachant may be renewed continuously, in which case it is sampled periodically, or it may be replaced intermittently. In either case, the liquid to solid ratio is expressed as the ratio of volume of leachant to surface area of sample. Examples of flow-around tests include the ISO Leach Test 6961, ANSI 16-1, the Monolithic Diffusion test from the Netherlands, and ASTM's Static Leaching of Monolithic Waste Forms for disposal of

Radioactive Wastes.

Flow-through tests differ from flow-around tests in that the leachant flows through the sample rather than around it. This means that the sample must be porous, rather than monolithic. Beyond that, the tests are conducted very similarly. Flow-through tests are usually constructed as columns or lysimeters, and pose particular experimental challenges such as channeling, flow variations caused by the hydraulic conductivity of the waste, clogging of the system by fine particulates, and biological growth in the system. Notwithstanding, flow-through tests can be set up to mimic site-specific conditions. Examples include Canada's Waste Interface Leach test, NVN 7344 Column Test, and ASTM's Column Test.

A Soxhlet extractor can be used to leach wastes by continuously contacting the waste with leaching fluid. In this test, the leachant is continuously renewed with "recycled" leachant from which previously extracted contaminants have been removed. This test is designed to maximize the amount of extracted contaminants, as quickly as possible. An advantage of the Soxhlet test is that it concentrates contaminants in the leachate, which may help overcome analytical detection issues, however, it is limited to relatively low boiling solvents as leachants, and it is not suitable for use when the contaminants of interest are volatile at or below the boiling point of the leachant. Canada's Soxhlet Test (MCC-5s) is a good example of this type of test.

She closed her presentation noting that each of these wide variety of tests has advantages and disadvantages, and each one is more relevant to some waste disposal scenarios than other.