Application Note
#AA3007

BUCK Scientific

# Determination of the Environmental TCLP Metals in Waste-Waters, Solid Wastes, and Soils by Flame Atomic Absorption Spectrophotometry

**Problem**

Hazardous wastes can originate from many sources—industrial processes, product formulations, synthetic operations, virtually any manufacturing business. The nature and composition of the "wastes" can vary so much that one industry's discharge can serve as another's raw materials. It is the tremendous transfer of these materials—their storage, transportation, spillage and leakage into the environment—that has caused great concern. Regulation of these discharged solid and liquid materials is a crucial part of the U.S. EPA and the State-level Departments of Environmental Protection programs for defining the "degree of hazard" of a particular material. The basic methods describe procedures to "leach" any soluble heavy metals from samples using acid(s) (to simulate acid rain, for example). While there is a lot of printed material available from these Government agencies, the basic protocols for the collection and preparation of this type of sample is referenced in the Standard Methods of SW-846 as part of the Resource Conservation and Recovery Act (RCRA).

**Principle**

The Toxic Characteristic Leach Procedure (TCLP) is a specific protocol to extract and decompose any complex or bound metals in a waste-water, solid waste or soil sample. This makes a solution suitable for atomic analysis by Flame Atomic Absorption Spectroscopy (FAAS) or Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). Flame AAS is an inexpensive, efficient method that has been time tested for many decades. ICP-OES is a newer technique that is faster, but significantly more expensive than FAAS. The sample preparations range from boiling water samples with acid, to shaking a solid sample in an acid slurry for 18 hours, to digesting at controlled temperatures in a fluoro-polymer microwave oven pressure vessel. The protocols for microwave oven assisted leachings are defined in the U.S. EPA Methods 3015 (liquids) and 3051 (solids).

**Practice**

An assortment of samples is prepared according to the appropriate methodology, and the resulting solutions are ready for instrumental analysis. The Buck Model-210VGP Flame AA system with the Nitrous Oxide assembly and the Model-420 Hydride unit are set up for running the metals listed in the TCLP program. Accuracy is ensured and detection limits are maximized by using the unique "in-line" Deuterium Background Corrector and the wavelength-independent Variable Giant Pulse Corrector (standard parts of the 210VGP) to eliminate any interferences in the sample measurements.

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SIC: 071, 8734, 951

# Determination of the Environmental TCLP Metals

**Samples**: A composite wastewater sample, a 30' deep drilling core soil sample, and a settling pond sludge sample were analyzed for 12 metals.

**Preparation**: (modified from the EPA guideline methods for TCLP & RCRA)
[1] 100ml. wastewater, boiled for 5 minutes with 5ml of nitric acid.
[2] 50 grams soil, leached with 100ml of 10% nitric acid / 5% acetic acid.
[3] 10 grams sludge, digested with 25ml of 25% nitric acid / 10% acetic acid.

**Calibration**: Certified EPA-type standards, containing the 12 analytes at levels from 0.1 to 5 ppm (mg/L), were used for a linear calibration curve.

**Instrument**: Buck 210VGP Atomic Absorption Spectrophotometer, $D_2$ Corrector, Nitrous Oxide burner, Hydride generator with Argon/Hydrogen.

**Conditions**: Air/Acetylene flame (Ag, Cd, Pb, Ni, Cr)
$N_2O$/Acetylene flame (Al, Ba, Be)
Hydride Generator (As, Se, Sn)
Cold Vapor Generator, Quartz tube (Hg)

* Note - Values are mg/L (ppm) in the original sample // D.L. = 3-sigma detectability

| Element | Wave-Length (nm) | Waste-D.L. | Water | Soil | Sludge |
|---|---|---|---|---|---|
| Ag | 328 | 0.04 | 0.09 | 0.36 | 6.97 |
| Cd | 228 | 0.02 | 0.23 | 0.69 | 1.21 |
| Pb | 283 | 0.15 | 0.72 | 1.82 | 8.03 |
| Ni | 232 | 0.10 | 2.48 | 2.17 | 5.85 |
| Cr | 357 | 0.08 | 1.34 | 0.61 | 3.53 |
| Al | 394 | 0.65 | 5.63 | (100) | (100) |
| Ba | 553 | 0.35 | 4.52 | 2.43 | 0.54 |
| Be | 234 | 0.02 | 0.03 | <0.02 | 0.39 |
| As | 193 | 0.001 | 0.28 | 0.13 | 0.74 |
| Se | 196 | 0.002 | 0.16 | 0.05 | 0.83 |
| Sn | 286 | 0.005 | 0.08 | 0.17 | 1.49 |
| Hg | 253 | 0.00001 | 0.0008 | 0.0012 | 0.0185 |

The above data shows the powerful flexibility and stability of the Buck 210VGP systems for the wide-ranging requirements of the Environmental industry. The overall high sensitivity of the various Trace metals supports the interference-free quality of the data. The combination of components provides for an unmatched system in economy and performance.

**Basic System: $12,950.00**

**Turnkey System: $20,718.00** Includes: All recommended lamps, standards, and accessories for normal operation.


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