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**OAK RIDGE
K-25 SITE**

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DEPARTMENT OF ENERGY

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**TREATABILITY STUDY FOR REMOVAL
OF LEACHABLE MERCURY IN
CRUSHED FLUORESCENT LAMPS**

February 1996

Prepared by
the Oak Ridge K-25 Site
Oak Ridge, Tennessee 37831-7133
managed by
LOCKHEED MARTIN ENERGY SYSTEMS, INC.
for the
U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-84OR21400

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**TREATABILITY STUDY FOR REMOVAL OF
LEACHABLE MERCURY IN
CRUSHED FLUORESCENT LAMPS**

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ABSTRACT

Nonserviceable fluorescent lamps removed from radiological control areas at the Oak Ridge Department of Energy facilities have been crushed and are currently managed as mixed waste (hazardous and radiologically contaminated). We present proposed treatment flowsheets and supporting treatability study data for conditioning this solid waste residue so that it can qualify for disposal in a sanitary landfill. Mercury in spent fluorescent lamps occurs primarily as condensate on high-surface-area phosphor material. It can be solubilized with excess oxidants (e.g., hypochlorite solution) and stabilized by complexation with halide ions. Soluble mercury in dechlorinated saline solution is effectively removed by cementation with zero-valent iron in the form of steel wool. In packed column dynamic flow testing, soluble mercury was reduced to mercury metal and insoluble calomel, loading >1.2 g of mercury per gram of steel wool before an appreciable breakthrough of soluble mercury in the effluent.

1. INTRODUCTION

Modern fluorescent lamps consist of a sealed glass envelope, internally coated with a phosphor material (usually 3–20- μm calcium halophosphate particles, doped with trace antimony (Sb) and/or manganese (Mn) activators), with an electrode assembly at each end (see Figure 1). The phosphor material converts ultraviolet light, produced by a low pressure mercury (Hg) arc, into visible light (Butler 1980). A typical 48-inch (122-cm) fluorescent lamp (cool white, type F40T12), weighing ~ 290 g, may contain 27–48 mg of mercury (or a total mercury (Hg) content ~ 93 –166 mg/kg, with newer lamps having less mercury) (Meyer 1992). The glass envelope represents the preponderance ($\sim 90\%$) of total lamp mass, but the high surface area phosphor component offers the greatest opportunity for condensation or sorption of mercury. According to industry sources (Foust 1994), the phosphor component, which represents less than 2% of the total lamp mass, may contain $\sim 80\%$ of the total mercury content; thus, the concentration of mercury associated with the phosphor component can be several thousand mg/kg [a typical value in used lamp phosphor powder is ~ 4700 mg Hg/kg (Truesdale et al., 1993, U.S. EPA 1994)]. Endcaps (predominantly aluminum) with leaded glass and filaments together represent the balance (about 8%) of the lamp mass.

Approximately 600 million fluorescent lamps are disposed of each year (Truesdale et al. 1993). Nonfunctional (spent) mercury-containing lamps may be considered hazardous waste under the Toxicity Characteristic Regulation issued under the Resource Conservation and Recovery Act (RCRA) provisions of the U.S. Environmental Protection Agency (EPA) [40 CFR Parts 260, 261, and 273]. If so, these lamps must be managed as hazardous waste. The EPA Toxicity Characteristic Leaching Procedure (TCLP) testing protocol is used to define the potential for select toxic constituents to leach out of the waste, simulating conditions that may exist for disposal of solid waste in an unlined sanitary landfill. Crushed fluorescent lamps evaluated by the TCLP testing protocol often fail the defining toxicity criterion for Hg (i.e., ≥ 0.2 mg/L in the leachate solution) (Meyer 1992), and may be classified as RCRA waste code D009 (characteristically hazardous due to the concentration of mercury in the leachate). To qualify for land disposal, waste failing the TCLP toxicity criterion must be conditioned to meet the Universal Treatment Standard (UTS; see 59 FR 47982, September 19, 1994); for mercury, the UTS criterion is 0.025 mg/L in the TCLP leachate.

Approximately 80,000 fluorescent lamps are replaced each year at the U. S. Department of Energy's three sites in Oak Ridge, Tennessee, (Oak Ridge National Laboratory, the Y-12 Plant, and the K-25 Site). Recently (August 1995), the Tennessee Department of Environment and Conservation indicated that intact lamps, managed for purposes of recycling/reclamation, may be considered to be characteristic by-products, and as such are conditionally exempt from strict regulation under RCRA Subtitle C as characteristically hazardous waste. These lamps are eligible for off-site shipment for reclamation of mercury and other lamp components.

Figure 1. Schematic representation of a fluorescent lamp showing approximate lamp mass contributions for various components.

However, by K-25 Site policy (Standard Practice Procedure IAD-SPP-3008), broken or crushed lamps continue to be managed as hazardous waste, and crushed lamps that were removed from radiologically contaminated areas are managed as mixed (hazardous and radiological) waste. Included in the latter category is an inventory of ~1000 sealed 55-gallon drums of previously crushed lamps (a total of ~165,000 kg of waste) currently in permitted storage at the K-25 Site.

We describe a treatability study to identify a process for conditioning the inventory of crushed lamps to yield a solid residue that qualifies for permitted disposal at the Y-12 Plant Sanitary Landfill, while producing a minimal volume of treatment residues that must still be handled as a potential mixed waste. This would free up much-needed permitted vault storage space for other priority mixed wastes. Options for removing mercury from wastes include thermal desorption ("Best Demonstrated Available Technology" for hazardous high-level mercury wastes; 40 CFR 268.41). Some commercial lamp recycling facilities use thermal processing (roasting or retorting) to reclaim mercury; although this approach can be effective, it places a burden on the engineered control of fugitive emission and/or off-gas, and may generate substantial secondary waste. For use in on-site treatment, an alternative aqueous extraction process flowsheet is attractive, since the conditioned effluent (after mercury removal) could be discharged in a regulatory compliant manner. The Central Neutralization Facility (CNF) at the K-25 Site treats aqueous hazardous and radiological waste by iron co-precipitation for release to the surface water through a monitored and permitted National Pollutant Discharge and Elimination System.

The process flowsheet discussed in this paper (Figure 2) consists of (1) solubilization of mercury by aqueous extraction or leaching; (2) liquid/solid phase separation; (3) liquid phase preconditioning (e.g., pH adjustment, dechlorination); and (3) recovery of mercury from the spent leachant solution (by sorption or reduction, to yield a minimal volume of contaminated solid waste). The mercury-depleted leachate will be polished by treatment at the CNF prior to discharge.

Gates and co-workers (1995) investigated several combinations of chemical agents for removal of mercury from solid mixed wastes: oxidants (to convert elemental mercury to soluble mercuric ion), acid (to prevent hydrolysis, and precipitation, of Hg^{++}), and complexants (to stabilize mercuric ions in solution phase). They supplemented the glass/phosphor component of crushed fluorescent lamps with additional Hg^0 , and determined recovery in the acidic leachant solution; two reagent systems were determined to be especially effective for recovery (>98%) of dosed mercury: potassium iodide/iodine (KI/I_2) and sodium hypochlorite (NaOCl). The KI/I_2 system for mercury removal has been patented by the General Electric Corporation (Foust, 1993). The use of hypochlorite (OCl^- , the active ingredient in household "bleach") to solubilize mercury is more 'generic' technology; for example, it has been used successfully to leach mercury from sludges and residues (Rockandel, 1992; Pedroso et al., 1994).

Figure 2. Process flow sheet for solubilizing mercury from crushed fluorescent lamps with subsequent recovery of mercury by selective sorption.

After the mercury has been solubilized, it must be removed from solution before the leachate can be released to the environment. It is desirable to recover the mercury in a concentrated form, to minimize the volume of hazardous secondary waste material for storage and/or to facilitate ultimate resource recovery. For hypochlorite leachate, preconditioning may include dechlorination (i.e., removal of excess oxidants that may interfere with sorption of mercury) and pH adjustment. Selective sorbants or reductants can be used to concentrate mercury from the dilute solution. We tested several candidate materials, including sulfur-impregnated carbons and organic ion exchange resins, but found metal wools (copper or iron) to be especially effective and economical. Iron metal (or steel) is inexpensive and environmentally friendly (nontoxic). Steel wool has been used in commercial applications to recover silver metal from spent photographic developer reagent waste; a galvanic reaction with the zero-valence iron reduces the complexed silver, which plates out in a greatly concentrated form suitable for subsequent resource recovery (Hess, 1992). In related work, we have found iron filings and steel wool to be effective reagents for removing radioactive $^{99}\text{Tc(VII)}$, and other contaminants such as Cr(VI) , etc., from aqueous solution (Bostick et al. 1995a).

2. EXPERIMENTAL

Microscopic Examination

Elemental distributions of mercury and lead in solids and sludges were determined by Energy-Dispersive X-ray Spectroscopy (EDS), using an Electroscan environmental scanning electron microscope. This instrumentation is capable of operation at relatively high pressures (5–50 torr) and at subambient temperatures. These conditions are favorable for the retention of relatively volatile mercury, which could be lost with use of conventional ambient temperature, high-vacuum instrumentation. By back-scatter imaging, heavy elements (such as mercury) give bright images, and can be readily discriminated from lighter elements (e.g., aluminum, silicon, calcium etc.) in the sample matrix.

TCLP Testing

All TCLP testing was performed by the K-25 Site Analytical Services Organization, using the approved EPA Method 1311 protocol. Sample data are archived by unique Analytical Laboratory Information System (AnaLIS) identification numbers (see Appendix A).

Quantitation of Mercury

Solutions submitted to Analytical Services were analyzed by cold vapor atomic absorption spectroscopy (EPA Method 7470). Some developmental samples were analyzed in-house using conventional atomic absorption spectroscopy. For rapid and sensitive determination of mercury sorption isotherms and packed-column breakthrough performance, we traced aqueous solutions of natural mercuric ions with ^{203}Hg , a gamma-emitting isotope with a radioactive half-life of 46.9 days. The tracer was obtained from Amersham International as a mercuric chloride solution (Product UC0751). Traced solutions were counted in a Packard Auto-gamma 500C radiation measurement system, and the results were corrected for radioactive decay.

Sorbents

The following sorbent materials were tested for their effectiveness in the removal of soluble mercury (as HgCl_2 in aqueous solution):

- * Duolite GT 73 mercury-selective cation exchange resin, from Rohm and Haas, Philadelphia Pennsylvania;

- * Nucon Nusorb Mersorb-3 and Kina-3 granular activated carbon products, from Nuclear Consulting Services, Columbus Ohio;
- * HGR sulfonated carbon from Calgon, Pittsburgh, Pennsylvania;
- * Decolorizing carbon, Norit A (alkaline), from Fisher Chemical, Fairborough, New Jersey;
- * Pulverized ASTM A518 high silicon steel, from Duriron Co., Dayton, Ohio;
- * Coarse copper wool (strand diameter $\sim 43 \mu\text{m}$), 'Elephant Brand', from International Steel Wool, Springfield, Ohio;
- * Pro's Best # 1 medium steel wool, from International Steel Wool, Springfield Ohio;
- * Medium steel wool, from Rotex Silver Recovery Co., Springfield, Ohio;
- * Coarse steel wool (strand diameter $\sim 93 \mu\text{m}$; removed from Chemical Recovery Cartridge II, catalog no. 173-4953), from Eastman Kodak, Rochester, New York.

Sorption Isotherm Testing

The techniques used are described in Bostick et al. (1995A, B). In summary, an accurately measured mass of sorbent and a measured aliquot of radiologically traced test solution are equilibrated in a glass vial for ~ 16 –24 hours on a Burrell wrist action shaker. Measured aliquots of equilibrated liquid phase, filtered through $0.2\text{-}\mu\text{m}$ pore membrane media, are counted to determine activity.

Column Breakthrough Studies

The techniques and apparatus used are described in Bostick et al. (1995B). Traced test solution was metered up through the sorbent medium packed in glass tubes with use of a calibrated peristaltic pump. Discrete effluent aliquot samples were collected directly into gamma-counting tubes at defined intervals using a programmable fraction collector. The aliquots were counted off-line, and the measured activity corrected for isotopic decay.

3. RESULTS AND DISCUSSION

3.1 Characterization of Waste from Storage Inventory

TCLP Testing of Crushed Fluorescent Lamps Stored at K-25 Site

Table 1 summarizes testing results for crushed lamp material sampled from inventoried waste. For these particular composite samples, the TCLP criterion for mercury (0.20 mg/L) was not exceeded; assuming a normal distribution of results for the seven sampling events, the median result for Hg in the TCLP leachate was (0.076 ± 0.05) mg/L. However, sampling and analysis of similar crushed lamps stored at the Y-12 Plant yielded an average Hg content in the TCLP extract of (0.27 ± 0.16) mg/L, with 7 of the 13 composite samples failing the toxicity criterion. Limited toxicity testing of fluorescent lamps at Oak Ridge National Laboratory, delivered to the laboratory intact before size reduction for TCLP, yielded Hg content in the TCLP extract of (0.29 ± 0.28) mg/L, with 4 of 10 lamps tested failing the toxicity criterion. Meyer (1992) has reported on the wide range of interlaboratory TCLP test results for fluorescent lamps but concludes that the testing consensus supports the handling of spent lamps as hazardous waste (see Appendix B).

Uranium Content of Crushed Fluorescent Lamps Stored at the K-25 Site

The previously described samples for TCLP testing were analyzed for total uranium (U) content (fluorimetric analysis), but no uranium was detected (<1.0 mg/g). The specific activity (SA) of uranium is a function of the isotopic distribution, which could not be determined in the low-uranium samples. Assuming natural uranium with SA 6.86×10^{-7} Ci/g, a background uranium content of $1 \mu\text{g/g}$ corresponds to an activity of ~ 0.7 pCi/g. The Waste Acceptance Criteria for the Y-12 Industrial Landfill specifies that uranium must not be present in wastes at activities >35 pCi/g (local background activity).

Total Mercury Content in a Subsample of Crushed Fluorescent Lamps Stored at the K-25 Site

A 5-gallon composite subsample was received for RCRA Treatability Study testing. The drum was cooled (to minimize possible loss of mercury by volatilization), and the contents were subdivided into a number of sealed glass containers for use in subsequent testing. Examination of identifying markings on the glass shards indicated that the material originated from lamps produced by a variety of manufacturers. To gain a figure of merit for mercury content, samples of glass envelope with attached phosphor were analyzed [by EPA Method 7471: Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)]; two subsamples gave estimates of 66 and 91 $\mu\text{g/g}$ (or an average mercury content of $\sim 80 \mu\text{g/g}$). This data appears fairly

**TABLE 1. DATA FOR TCLP EXTRACTION OF CRUSHED
FLUORESCENT LAMPS STORED AT THE K-25 SITE
(RESULTS GIVEN AS MG/L IN EXTRACT)**

AnaLIS ID ^a	Mercury ^b	Lead ^c	Arsenic ^c	Barium ^c	Cadmium ^c	Chromium ^c	Nickel ^c	Selenium ^c	Silver ^c
940608-124	0.033	1.0	<0.25	0.73	<0.015	<0.05	0.97	<0.25	<0.035
940608-123	0.11	1.4	<0.25	7.2	<0.015	0.17	14	<0.25	<0.035
940608-121	0.056	1.1	<0.25	1.1	<0.015	<0.05	1.6	<0.25	<0.035
940608-122	0.066	0.43	<0.25	0.75	<0.015	0.091	0.53	<0.25	<0.035
940608-119	0.065	<0.25	<0.25	4.2	<0.015	0.23	9.8	<0.25	<0.035
940608-120	0.031	<0.25	<0.25	1.1	<0.015	<0.05	1.3	<0.25	<0.035
910115-070	0.169	0.07	0.054	1.4	0.010	<0.01	1.3	<0.05	<0.01
Median Value	0.076 ±0.05	≤0.64	<0.25	2.4 ±2.5	<0.015	≤0.09	4.2 ±5.4	<0.25	<0.035
TCLP Criterion ^d	0.20	5.00	5.00	100	1.0	5.0	NA	1.0	5.0
UTS Criterion ^e	0.025	0.37	5.00	7.60	0.19	0.86	5.0	0.16	0.30

^aLMES Analytical Laboratory Information System Sample Identification Number (data archive).

^bAtomic Absorption Spectroscopy (EPA Method 7470).

^cInductively Coupled Plasma Spectroscopy (EPA Method 6010).

^dToxicity Characteristic Leaching Procedure (40 CFR 261.24).

^eUniversal Treatment Standard for hazardous waste: TCLP extract (nonwastewater) (60 CFR 264).

consistent with industry reports. Data presented by Meyer (1992) indicates that the total mercury content in the glass/phosphor component of typical 4-foot long $\times$ 1.5-inch diameter cool-white fluorescent lamps (F40T12) may be in the range of $\sim 100\text{--}180\text{ }\mu\text{g/g}$, with the higher value being associated with older lamps. It is probable that some mercury is lost from the crushed lamp inventory by volatilization or particulate entrainment during the crushing operation (which is conducted at reduced pressure, with off-gas processing through an activated carbon canister) (EPA 1994).

3.2 Investigations in Support of the Proposed Treatment Flowsheet

Microscopic Examination of Glass/Phosphor

A used (nonwaste) F40T12 lamp was broken to evaluate the association between mercury and phosphor on the glass envelope. In agreement with manufacturers' data, the preponderance of phosphor particles ranged from approximately $2\text{--}20\text{ }\mu\text{m}$ in greatest dimensions. Mercury-rich phases were identified in association with the phosphor particulate phase; in general, these mercury-rich phases appear as small spherical particles, possibly as liquid mercury droplets. A band of dark discoloration was noted on the glass near the electrode filament ends; this band was notably enriched in small lead-containing particles and was likely the result of volatilization from the leaded glass components during service.

Solubilization of Mercury

Gates and co-workers (1995) demonstrated that a 6% solution of NaOCl, adjusted to pH ~ 2 , was highly effective in solubilizing elemental mercury. Hypochlorous acid (HOCl) ($\text{pK}_a = 7.48$) is a strong oxidant and is the predominant hypochlorite species in acidic solution. We tested the effect of three variables (solution pH, NaCl concentration, and hypochlorite concentration) on the solubilization of liquid mercury. Of these, the hypochlorite concentration was the most significant factor (more mercury was solubilized with higher concentration of hypochlorite). At a given level of hypochlorite, somewhat greater quantities of mercury were solubilized at lower pH values (i.e., 4 versus 7) and at moderate concentrations of NaCl (to form the stable complex HgCl_4^{2-} ion; Pedroso et al., 1994). At pH values much less than 4, the formation of chlorine gas is favored, which may be lost due to its volatility (Pedroso et al. 1994), and at pH values much greater than 7, the hydrolysis of mercury can occur with loss due to precipitation. The mercury leachant composition selected for further testing contained ~ 0.5 weight percent (wt %) NaOCl and 75 g/L NaCl, adjusted to an initial pH value of 4.

Hypochlorous acid should also be effective in solubilizing certain radiological contaminants, if they are actually present in the waste stream. Gates et al. (1995A) show that neither the hypochlorite/chloride nor iodine/iodide extraction systems solubilize appreciable ceric oxide (CeO_2) (a nonradiological surrogate for insoluble uranium oxide; see also Stockdale et al., 1994); however, these systems should readily solubilize uranium contamination if it is present in the form of uranyl compounds. The other radionuclide of interest that could be present in waste at the K-25 Site is ^{99}Tc —a weak beta emitter—that would be readily solubilized as the pertechnetate ion in oxic media (Pourbaix, 1974). Technetium-99 in packaged debris waste cannot be easily detected by nondestructive assay (NDA)—unlike uranium and its decay daughters, which have penetrating characteristic gamma emission. Therefore, extraction of ^{99}Tc will aid in the certification of solid debris waste as “no radioactivity added” by NDA survey.

Mercury Recovery from Hypochlorite/Chloride Leachate: Batch Equilibrium Sorption Studies

Table 2 (from Cooney et al., 1992) summarizes typical results attainable for treatment of mercury salts in plating wastewaters. In initial screening tests we examined the utility of several sorbent media for removal of soluble mercuric ions from aqueous solution (mercuric chloride in distilled water). In this solution traditional mercury sorption media, such as sulfur-impregnated granular activated carbon (Mersorb) and ion-exchange resin (Duolite) (cf. Table 2) were less effective than metal wool (copper and steel); see Figure 3. In Figure 4, we compare efficiencies for trace soluble mercury, in both de-ionized water (cf. Figure 3) and in 7.5 wt % salt solution, using two of the more effective media (steel wool and Duolite resin). In this testing, both media appeared comparably effective for removal of mercury in salt solution; however, the steel wool reagent was selected for use in dynamic flow testing because it is less expensive.

Copper and steel wools and steel wool with a thin layer of galvanically-deposited copper (see Equation 2) were comparable in their effectiveness (Figure 5) with each material removing ~3.5–4 g of Hg per g of metal at equilibrium from concentrated solution (cf, molecular weight ratio, $\text{Hg}/\text{Fe} = 3.6$). (Copper flashed on steel wool may decrease the loss of steel substrate due to parasitic corrosion). The electrochemical displacement and precipitation of a metal from solution by a metal higher in the electromotive series (see Table 3) is referred to as “cementation” and iron metal is most often used as the sacrificial anode (Cooney et al., 1992). Zero-valent iron can also spontaneously reduce mercurous and mercuric ions to elemental mercury (Equations 3 and 4).

Figure 3. Removal of soluble mercury (as HgCl_2 in distilled water) with use of various media.

Equilibrium Concentration vs. Dosage for Sorption of Hg ($C_0 = 10$ ppm)
in DI Water (cf. 7.5 wt% NaCl soln)

Fig. 4. Comparison of Rotex steel wool and Duolite GT-73 resin for the removal of trace soluble mercury ($C_0 = 10$ mg/L) from de-ionized water and from 7.5 wt % sodium chloride solution. (Sorbent dosage reported as dry-weight equivalent.)

Figure 5. Comparison of metal wool products for removal of HgCl_2 in distilled water at equilibrium.

**TABLE 2. TREATMENT TECHNOLOGY EFFECTIVENESS FOR MERCURY
IN PLATING WASTEWATER**
(From Cooney et al., 1992)

Technology	Lower Limit of Treatment Capability (mg/L)
Sulfide Precipitation	10-20
Ion Exchange	1-5
Alum Coprecipitation	1-10
Iron Coprecipitation	0.5-5
Activated Carbon Adsorption	
High Initial Mercury	20
Moderate Initial Mercury	2
Low Initial Mercury	0.25

**TABLE 3. SELECTED STANDARD ELECTRODE POTENTIALS
FOR ACIDIC AQUEOUS SOLUTIONS AT 25°C***

Couple	Electrode Reaction	E ⁰ , V
Al ³⁺ /Al	Al ³⁺ + 3 e ⁻ = Al	-1.662
Zn ²⁺ /Zn	Zn ²⁺ + 2 e ⁻ = Zn	-0.762
Fe ²⁺ /Fe	Fe ²⁺ + 2 e ⁻ = Fe	-0.447
Pb ²⁺ /Pb	Pb ²⁺ + 2 e ⁻ = Pb	-0.126
Cu ²⁺ /Cu	Cu ²⁺ + 2 e ⁻ = Cu	+0.342
Hg ₂ ²⁺ /Hg	Hg ₂ ²⁺ + 2 e ⁻ = 2Hg	+0.797
Ag ⁺ /Ag	Ag ⁺ + e ⁻ = Ag	+0.800
Hg ²⁺ /Hg	Hg ²⁺ + 2 e ⁻ = Hg	+0.851

*Compiled from *CRC Handbook of Chemistry and Physics*, 68th Ed.

(Standard cell potentials are computed from the tabulated data presented in Table 3. By sign convention, a positive cell potential represents a thermodynamically favored reaction).

Rockandel (1992) reports that inexpensive ground steel scrap is an effective cementation medium for mercury recovery and that the optimum cementation pH is in the range of 2–4. This cementation reaction produces relatively high concentrations of ferrous iron [Fe(II)], which is subsequently oxidized by exposure to air to form ferric ion [Fe(III)]. Recently, Grau and Bisang (1995) have also shown that iron felt rapidly removes soluble mercury from solutions containing chloride ion. Other active zero-valent metals [including copper (Tyson 1993) and zinc (Pedroso et al., 1994)] can be used as the cementation agent for mercury recovery, but these metals are more expensive and produce replacement salts that are generally more toxic than the corresponding iron salts in aqueous systems (Luckey et al., 1975).

Granulated corrosion-resistant ASTM A-518 high-silicon steel was not effective for removal of soluble mercury, suggesting that galvanic reduction of mercuric ion is coupled to the oxidation of an iron metal substrate.

Mercury Recovery: Packed-Column Breakthrough Studies

Based upon the batch equilibrium sorption studies described above, steel wool was selected for column breakthrough testing. This material was removed from an Eastman Kodak Chemical Recovery Cartridge II (Fig. 6); this canister, used for recovery of silver from photographic waste solutions, is prepackaged to contain ~3.4 kg steel in a nominal 20-L active volume, for a nominal packing density of ~0.17 g steel/cm³. With a strand diameter of 93 μm, the nominal surface area for this material is ~0.006 m²/g. Vendor-supplied literature (Kodak Technical Publication J-9) indicates that optimum cementation with silver from waste solution occurs under slightly acidic conditions (pH4–6.5), which allows for a controlled rate of steel corrosion concurrent with reduction of pollutant metal ions. The insoluble reduced form of the metal of interest remains in the canister for later reclamation. Assuming a density for steel of ~7.7 g/cm³ suggests that the packed canister contains ~98% void volume, which is ample allowance for the volumetric increase that occurs as iron corrodes into iron oxides. Operation of the unit with waste feed solution at pH values between 4 and 6 minimizes the deposition of iron hydroxide sludge in the active treatment zone. At pH values less than 4, steel corrosion may be excessive, limiting the effective operational lifetime for the treatment canister. At pH values greater than ~9.5, the steel surface becomes passivated (Pourbaix 1974) and is no longer effective as a reducing agent; this condition is illustrated

Fig. 6. Chemical recovery cartridges, containing steel wool reagent: Rotex steel wool in translucent cannister on left; Kodak cannister on right.

Figure 7. Removal of soluble technetium-99 from carbonate solution by zero-valent iron (Kodak steel wool) and selected anion-exchange resins as a function of equilibrium solution pH value. (0.01 g as received sorbent/mL simulated Oak Ridge National Laboratory newly-generated low-level liquid waste process water). From Bostick et al., 1995b.

in Figure 7 for the reduction of the radionuclide technetium-99 (zero-valent iron reduces soluble Tc(VII), as pertechnetate ion, to insoluble Tc(IV) oxyhydroxide; Del Cul and Bostick, 1995; Bostick et al., 1995b). Thus, ^{99}Tc (if present in the leachate) would also be removed by treatment with steel wool. Recent work by Cantrell et al. (1995) demonstrates that metallic iron is effective for removal of both uranyl ion (UO_2^{2+}) and pertechnetate ion.

Packed-Column Breakthrough Studies

For laboratory-scale studies, Kodak steel wool was packed into glass columns, and radiologically-traced mercuric chloride solution was fed to the column by upflow. The initial concentration, C_0 , for total Hg was $\sim 3,000$ mg/L in 7.5 wt % NaCl solution, pH ~ 6 . Table 4 summarizes the experimental conditions and performance for incipient breakthroughs (defined here as $C/C_0 \sim 0.01$). Breakthrough data are plotted in Figure 8. There appears to be an optimal range for liquid flow through the steel wool. At low liquid flow rates, gases formed by the corrosion of iron, or entrained air, can coalesce, forming bubbles entrapped in the metal mesh; this prevents solution from wetting the metal surface, creating channelized flow and decreased treatment efficiency. At sufficiently high flow rates, small droplets of liquid mercury reduction products may be transported from the column before they can coalesce.

Table 4. SUMMARY OF CONDITIONS FOR PACKED-COLUMN BREAKTHROUGH STUDIES USING KODAK STEEL WOOL

	Run 1	Run 2	Run 3
Initial Active Zone Dimensions	5.25 cm ³ [1.0 cm (D) × 6.83 cm (L)]	6.32 cm ³ [1.0 cm (D) × 8.26 cm (L)]	2.75 cm ³ [0.7 cm (D) × 7.14 cm (L)]
Mass of Steel Wool (g)	4.89	4.89	2.00
Initial Wool Packing Density (g steel/cm ³)	0.93	0.78	0.73
Solution Feed Rate	0.41 mL/min (0.13 gpm/ft ²) ^a	0.91 mL/min (0.29 gpm/ft ²) ^a	1.1 mL/min (0.67 gpm/ft ²) ^a
Nominal Residence Time (minute)	12.8	6.9	2.6
Loading of Mercury on Steel at Incipient Breakthrough (g Hg/g Fe) ^b	~ 1.25	> 1.70	~ 1.23
Total Experimental Run Time (days)	~ 3.2	~ 2.0	~ 0.6

^aGallons per minute per square foot cross-sectional area.

^bGrams of mercury removed per gram of original steel wool packing, at soluble mercury $C/C_0 \sim 0.01$ (i.e., 1% breakthrough).

Figure 8. Breakthrough of complexed mercury ($C_0 \sim 3,000$ mg Hg/L in 7.5 g/L NaCl solution, initial pH ~ 6) from glass columns packed with steel wool (see Table 3 for data summary).

As noted from Table 4 and Figure 8, at favorable dynamic flow rates, it is possible to remove a greater mass of mercury than the original mass of steel present in the active zone (>1.2 g Hg per g Iron) before appreciable breakthrough of soluble mercury occurs. Thus, in principle, a single Kodak canister (~ 5 -gal capacity), containing 3.4 kg steel, could remove ~ 4.1 kg Hg under optimal conditions before an appreciable breakthrough. This would correspond to the total mercury content of $\sim 120,000$ 48-inch fluorescent lamps (or >200 55-gallon drums of crushed lamps). In practice, cementation reactions with contaminant metal ions must compete with the consumption of steel due to simple corrosion mechanisms. For the incipient breakthrough data presented in Table 4, with a high concentration of mercury in the feed solution, $\sim 1/3$ of the initial amount of steel had been consumed by reaction with mercury (i.e., a yield of >1.2 g-Hg/g-Fe at $C/C_0 = 0.01$, versus ~ 3.6 g-Hg/g-Fe at equilibrium), with most of the balance of the initial mass of steel being consumed by simple corrosion in the salt water medium.

A small packed column of steel wool was tested using untraced (nonradiological) mercury to facilitate examination of the column contents after a breakthrough of soluble mercury. Void spaces in the corroded steel fiber "mush" were observed to contain beads of elemental mercury (Figure 9). By X-ray diffraction, some insoluble calomel (Hg_2Cl_2) was also detected. Iron phases in the mush were predominantly magnetite (Fe_3O_4), with hematite (Fe_2O_3) and elemental iron also present.

At moderate flow rates, mercury removal continued to be efficient as long as some steel metal was present in the column. Recently, we have obtained packed steel wool canisters in custom-fabricated translucent plastic containers (Rotex Silver Recovery Co.), which will facilitate visual estimation of a recovery unit's operational lifetime. From Table 4, the consumption rate for steel wool before an appreciable breakthrough of soluble mercury in a corrosive salt solution was ~ 1.5 – 3.3 g/day.

Steel Wool Canister Effluent Polishing

The redox activity of the steel wool column was also evident from the various color phases observed in the glass column and translucent tubing that lead to the fraction collector. The effluent immediately downstream from the packed column had a greenish tint, suggestive of ferrous chloride, and further downstream, the effluent became orange-brown, suggestive of oxidized ferric hydroxide. The pooled, post-breakthrough effluent equilibrated with air contained predominantly soluble or colloidal ferric and mercuric ions. Filtration of the air-equilibrated effluent through 0.8- and 0.2- μm filter media did not remove appreciable mercury ($C/C_0 = 0.96$ and

Figure 9. Beads of elemental mercury after breakthrough in a packed column of steel wool.

0.93, respectively), although the effluent was progressively decolorized (attributed to the removal of colloidal iron or other material in suspension).

As discussed earlier, the on-site wastewater treatment facility uses iron co-precipitation as the primary treatment to remove soluble toxic metals and radionuclides (Bostick et al., 1992, 1995a). The effluent from the lamp leaching projects would be diluted with a large volume of other wastes (predominately saline scrubber blowdown from a mixed waste incinerator). To test for mercury removal by iron coprecipitation treatment, we used a composite column effluent which included post-breakthrough samples; this sample contained ~70 mg/L soluble mercury at an initial pH ~6.6. An aliquot was acidified (to pH ~3), and 50 mg/L of Fe(III) was added (as ferric sulfate). The solution was readjusted to pH ~8.6, and flocculated with ~15 mg/L Betz 1138 polymer. The clarified supernate was filtered before counting; removal efficiency for mercury was ~76% (i.e., $C/C_0 \sim 0.24$, with a soluble residual of ~17 mg/L). A 10-fold dilution of pooled effluent ($C_0 \sim 6.6$ mg/L) was also treated by coprecipitation, yielding a removal efficiency of ~59% (soluble residual of ~2.7 mg/L). Thus, as noted in Table 2, coprecipitation will be an efficient wastewater “polishing” step for the liquid effluent from the sorption unit operation of the proposed lamp leaching process.

3.3 RCRA Treatability Studies in Support of Proposed Treatment Flowsheet

Examination of the crushed lamps revealed two distinct types of materials: (1) shards of broken glass envelope with phosphor attached, and (2) aluminum endcap assemblies with leaded glass and filaments attached. These components were tested by the TCLP, both separately and in a mixture representing the proportionate mass ratios found in intact lamps (i.e., ~90% glass/phosphor and ~10% endcap assemblies; Foust, 1994). Testing was performed for the original (untreated) material and material that had been treated by preleaching with a solution containing ~0.5 wt % NaOCl and 75 g/L NaCl, adjusted to an initial pH value of 4. Liquid and solid phases were contacted in sealed jars placed on a rolling mill for 6 hours. Solids were contacted first with a bleach/salt “wash” solution. Phases were then separated by either filtration or decantation. Next, distilled water was added to the solid phase as a rinse solution, before again contacting phases by mixing on a rolling mill followed by a final phase separation to recover the treated solids.

Testing with Glass/Phosphor Component

As noted previously, the glass envelope and its associated phosphor component should have the greatest proportion of mercury in the crushed lamps. Using untreated glass/phosphor from the inventoried crushed lamps,

the concentration of mercury in the TCLP leachate averaged (0.14 ± 0.01) mg/L.

In the first series of treatment evaluation, 100 g of glass/phosphor shards were initially contacted with ~860 g of bleach/salt solution, resulting in a liquid/solid (L/S) mass ratio of ~8.6. The glass and leach solution were contacted by rolling the mixture in a sealed cylindrical 1-gallon jar for 6 hours. The phases were separated by coarse filtration (hardened Whatman 54 paper, nominal 20–25 mm-pore), backed by a 1 mm-pore membrane filter medium. The solids were returned to the jar, together with 1000 mL of distilled water rinse solution (L/S = 10), mixed by rolling for 2–4 hours, and then refiltered. Duplicate samples of washed solids were submitted for TCLP extraction (see Table 5), both yielded no detectable mercury in the TCLP leachate (<0.001 mg/L).

Table 5. TCLP LEACHATE TESTING FOR CRUSHED FLUORESCENT LAMP COMPONENTS

Sample Description	Metal in TCLP Extract (mg/L)	
	Mercury (Hg)	Lead (Pb)
Glass/Phosphor Component		
Untreated	0.14	0.12
Treated ^a : Series 1 (L/S ~ 8.6) ^b	<0.001	0.17
Treated ^a : Series 2 (L/S ~ 2.0) ^b	≤0.0015	0.19
Endcap/Leaded Glass Component		
Untreated	0.023	0.016
Treated ^a (L/S ~ 7.8) ^b	0.012	0.011
Glass/Phosphor (~90%) + Endcap/Leaded Glass (~10%)		
Untreated	0.055	0.062
Treated ^a (L/S ~2.9) ^b	0.027	0.044
Historic Data for As-Stored Crushed Lamps ^c	0.076 ± 0.05	<0.50

^aTreatment consisted of washing with solution containing ~0.5 wt % NaOCl and 75 g/L NaCl, adjusted to an initial pH value of 4, followed by a water rinse.

^bLiquid/Solid mass ratio for bleach/salt wash step (see text for additional detail).

^cSee Table 1.

The aqueous wash and rinse solutions from this test were submitted for analysis to verify that mercury was solubilized from the solid debris. Assuming an average total mercury content of ~80 mg/g on the glass/phosphor solids (from the direct analysis), a 100-g sample should contain a total of ~8 mg Hg ($\pm 30\%$). Replicate samples indicated total recovery of solubilized mercury in the wash and rinse solutions of ~3–7.5 g, or nominal recoveries of ~38–96%. This data is similar to the results reported by Gates et al. (1995 A,B) for the solubilization of elemental mercury from glass/phosphor component by a large excess of oxidant/complexing reagent.

A second series of washing was conducted in a manner similar to the first, except that (1) 400 g of glass/phosphor and 860 g (~820 mL) of bleach/salt wash solution were contacted (i.e., L/S = 2.1), followed by a water-rinse at L/S = 2.5, and (2) phase separation was by simple decantation (versus filtration). Mercury in the TCLP extract of washed lamp shards (<0.0013 mg/L) was well below the UTS success criterion (<0.025 mg/L). However, analysis of mercury in the filtered wash and rinse solutions suggested that the mercury actually solubilized was <10% of the nominal total amount present. This data suggests that oxidant concentration may become a limiting reagent and should be monitored to ensure that it remains in excess during the treatment. It also suggests that it may not be necessary to solubilize the mercury, but that it may be possible to remove the phosphor and associated mercury particulate by rinsing and filtration alone (*vide infra*).

Testing with Endcap Assembly Component

As shown in Table 5, the TCLP extract for untreated endcap assemblies contained mercury at only 0.023 mg/L. Treatment of the endcap assemblies with the aggressive bleach/salt solution (L/S = 7.8) and subsequent water-rinse (L/S = 9.1) yielded solids that gave approximately half this concentration of mercury in the TCLP leachate (see Table 5).

Testing with Proportionate Mixtures of Glass/Phosphor and Endcap Assemblies

Solids tested consisted of 20 g (6.8 wt %) endcap assemblies plus 274 g (93.2 wt %) glass/phosphor. Treatment was with ~860 g bleach/salt solution (L/S ~2.9), in a sealed jar placed on a rolling mill for 6 hours. This wash solution was decanted and replaced with 1000 g distilled water rinse (L/S = 3.4), and the jar was again placed on a rolling mill. The washed, rinsed solids were separated by filtration through coarse and fine media. The TCLP extract for the isolated solids yielded a mercury concentration of 0.027 mg/L, which is slightly above the UTS criterion of 0.025 mg/L. It is likely that the UTS criterion could be consistently met with use of a higher liquid-to-solid ratio and/or a higher concentration of the oxidant reagent.

Treatment of crushed lamp components containing endcap assemblies with a solution of bleach and salt produces a dark-colored colloidal suspension in the unfiltered supernate solution, perhaps due to leached iron or other corrosion products. It is probable that iron and aluminum in the endcap assembly consume the hypochlorite in the wash solution (by corrosive reaction), rapidly depleting its availability for solubilizing mercury.

3.4 Preliminary Investigations in Support of an Alternate Treatment Flowsheet

Preliminary Testing: Rinse Only

It may be possible to achieve the treatment goals by removal of the phosphor phase, with its associated elemental mercury, without the requirement for mercury dissolution. This would make a greatly simplified process flowsheet (see Figure 10), and would avoid the use of corrosive oxidant/complexing agent solutions. Rinsing with dilute mild reducing agents should help to minimize the solubilization of mercury during processing.

In initial testing of this concept, 1922 g of glass/phosphor (90.7 wt %) and 198 g of endcap assemblies (9.3 wt %) were contacted in a sealed jar with 2750 g process water containing 23 g granular sodium bisulfite (NaHSO_3 , from J.T. Baker Chemical Co.), for an L/S of 1.3. This L/S ratio simulates transferring the contents of a as-stored 55-gallon drum (~75% filled with crushed lamps) to an 80-gallon drum, which is filled to ~94% total capacity by the addition of 58-gallon of ~0.08 mol/L bisulfite/metabisulfite solution before sealing the drum and contacting phases by roll mixing. In the laboratory-scale testing, phases were contacted by rolling for 4 hours. The jar contents were allowed to stand overnight (~16 hours) before separating phases by draining the contents through nylon screen. The drained solids were rinsed by the addition of 2750 g of process water, and again mixed by rolling for 4 hours before again draining the liquid phase. The drained bulk solids were submitted for TCLP analysis, which yielded a value of 0.012 mg Hg/L in the leachate, which is well below the UTS criterion.

In a second test, 1030 g of glass/phosphor (95.5 wt %) and 49 g of endcap assemblies (4.5 wt %) were contacted with 2750 g of ~0.08 mol/L bisulfite solution, for L/S ~2.5. As before, the contents were mixed by rolling, the liquid drained and replaced by process water for a final rinse. The TCLP analysis for the drained solids again yielded a value of 0.012 mg Hg/L in the leachate.

Some preliminary tests were performed to confirm that mercury was actually removed by the rinse-only process. In order to harvest sufficient "phosphor" phase to permit direct analysis of its mercury content, seven unused intact lamps were crushed (total mass, 1987 g); the crushed solids were contacted with 2750 mL of ~0.09 mol/L bisulfite solution (L/S ~1.4) by rolling in a sealed jar for 4 hours. Phosphor and some very fine glass shards were isolated from the larger solids by filtering the jar contents through plastic screen (equivalent to Tyler 14 mesh), and the fine solids in the filtrate slurry were harvested by centrifugation. On a dry-weight equivalent basis,

Figure 10. Proposed process flowsheet to remove mercury-containing phosphor particulate from crushed fluorescent lamps by rinsing with water containing a mild reducing agent.

a total of 37.5 g of phosphor/fines (1.9% of total lamp mass) were collected, having a mercury content of about 2,400 µg/g. Thus, a large proportion of the mercury in the lamps tended to follow the phosphor/fines in the rinse solution. Mercury solubility in the rinse solution was minimal (<0.05 mg/L) [a saturated solution of Hg (liq) in water at 20°C contains 0.02–0.03 mg/L soluble mercury (Burrows et al., 1974)].

3.5 Proposed Apparatus for Pilot-Scale Treatability Demonstration

The flowsheet depicted in Figure 11 could be used to scale up either of the proposed redox treatment processes but is presented in terms of the bisulfite solution displacement of phosphor and other fine particulate (see previous section). The initial step is coarse screening to remove incidental large debris (e.g., respirator cartridges) that may have been placed in the drums.

Next, the solids and wash solution would be contacted; this could be accomplished with use of a tilt mixer. A small mobile cement mixer could process the contents of one drum per batch, whereas a stationary Mina-Mixer (Munson Machinery Co., Utica, NY 13503) could process 2–3 drums of crushed lamps per batch. After contacting the phases, the mixer contents could be transferred to a high-efficiency vibratory screen platform (available from, e.g., Smico Manufacturing Co., Oklahoma City, OK 73127). The unit can be configured to simultaneously produce several different size fractions. A coarse mesh screen could separate relatively large components, such as endcap assemblies, from intermediate size material (washed glass). The washed glass represents the largest mass fraction of the crushed lamps and can be tested against the Waste Acceptance Criteria for the Y-12 Sanitary Landfill. Alternately, engineers for the Transportable Vitrification System have expressed interest in the low mercury washed glass component, without endcap assemblies, for use as a waste silica source for co-immobilization of low-level waste in a glassy matrix (L. R. Dole, Lockheed Martin Energy Systems, personal communication to W. D. Bostick, Lockheed Martin Energy Systems, June 20, 1995). Wash water with fine particulate, including phosphor slurry, is collected in a sump for recycle or further processing.

Results presented in Section 4 suggest that mercury tracks with the phosphor phase. Removal of fine phosphor from aqueous suspension can be accomplished with the use of the V-Sep filtration unit (New Logic International, Emeryville, CA 94608). This novel continuous filtration system uses a vibratory shear force at the membrane filter surface, allowing a high filtrate flux without clogging the membrane surface. To test the performance of this novel system, we provided the vendor with a sample of virgin phosphor (production-grade material, before addition of mercury, kindly provided by General Electric Corp.). This material in slurry was demonstrated to be extremely easy to filter in the vendor test. The vendor suggests that, with use of a Trisep

Polysulfone membrane filter, the unit can yield a high-flux clear permeate (filtrate) solution and a concentrated solids stream (containing 50–60 wt % solids) (M. Monroe and B. Culkin, New Logic International, correspondence to K. T. Bowser, Lockheed Martin Energy Systems, January 1996). The small-volume concentrated solids stream can be containerized as RCRA hazardous waste or should be amenable to resource recovery (the mercury content should be more concentrated than in the original waste by a factor of >100).

Steel wool cannisters can be used as a final “polishing step” to remove any solubilized mercury before the filtrate is transported to the CNF for iron co-precipitation and ultimate discharge. The Rotex model M-24 cannister (Rotex Silver Recovery Co., Springfield, OH 45503) contains ~24 lbs of steel wool and has a recommended solution throughput of >14 gal/h.

In order to minimize the possibility of transfer of soluble mercury to the CNF, performance of the steel wool reagent cannister should be monitored to ensure that its treatment capacity has not been exceeded. Use of a translucent reagent cannister (such as illustrated in Fig. 6) facilitates visual confirmation that active reagent is still available, but use of a continuous, on-line monitor for soluble mercury would offer greater performance assurance. Colleagues at the Oak Ridge Y-12 Plant have recently evaluated an automated commercial electrochemical sensor (model 4800 on-line metals monitors, Ionics Corp., a division of Chemtronics, Ltd.) for mercury in aqueous solution. They report a reliable instrument operating range of 0.02–1.0 mg/L (Smith and Godfrey, 1995). If the concentration of mercury is determined to be above a set threshold value, a transducer signal could be generated to actuate a valve to divert the solution flow to a fresh treatment cannister; the spent cannister would be removed from service, and retained for possible resource recovery. This system would provide assurance that mercury in solution collected for transfer to the CNF is within acceptable limits.

Figure 11. Flow sheet of proposed redox treatment process for removal of mercury from crushed fluorescent lamps.

4. CONCLUSIONS

As others have noted, hypochlorite (bleach) is an effective reagent to solubilize elemental mercury. We applied this approach to solubilization of mercury in components of crushed fluorescent lamps. The glass/phosphor component contains the great majority of the total mercury. For this component, solubilization appeared successful, providing that a large stoichiometric excess of hypochlorite was available. In the presence of endcap assembly components, solubilization of mercury by hypochlorite/chloride wash solution appeared less complete. It is possible that the residual solids may contain sorbed mercury that might cause the treated solids to not consistently meet the qualifying criterion for land disposal mercury-containing waste (*viz.*, < 0.025 mg Hg/L in the TCLP test leachate). The metal components of the endcaps (aluminum casing and iron filaments, etc.) may consume the oxidant reagent (by corrosion) and may complicate the overall redox chemistry (note in Table 3 that Al, if stripped of its protective oxide coating, is a potent reducing/cementation agent). It may be possible to consistently treat crushed lamps to the land disposal criterion for mercury with use of a large excess of oxidant. Alternately, the large endcap assemblies could be separated from the smaller, lighter glass/phosphor shards and the components treated separately.

Mercury is shown to be effectively removed from aqueous solution by cementation with zero-valence iron in the form of steel wool. In our testing, steel wool performed well for the removal of complexed mercuric ion in saline solution under conditions of dynamic flow. Steel wool reagents are available from several manufacturers in the convenient form of prepackaged chemical recovery cartridges, which are used primarily for the recovery of silver ion from photographic waste.

It may also be possible to achieve the treatment goals by removal of the phosphor phase, with its associated elemental mercury, without the requirement for mercury dissolution. This concept was successfully tested by rinsing a mixture of lamp components with a solution of dilute mild reducing solution (to suppress dissolution of mercury), followed by a water rinse to remove residual reagent solution.

Effective implementation of this alternative will require removal of fine particulate with its associated mercury phase from the rinse slurry; this small volume of fine particulate waste, enriched in mercury content, should be amenable to recovery of mercury by (e.g.) thermal retorting. If bisulfite solution is used in the rinse process, excess reagent, after mercury recovery, may be converted to innocuous sulfate ion by air oxidation.

5. ACKNOWLEDGMENTS

The authors wish to thank Dr. Donald Foust, General Electric Corporation, for sharing information regarding mercury distribution in fluorescent lamp components. We thank M. J. Shelton of the K-25 Site Waste Management Organization for sponsoring these investigations.

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APPENDIX A

SAMPLE DATA

SAMPLE IDs

	ANALIS ID	Customer ID	Description
	The following two samples were analyzed for mercury instead of for mercury on the TCLP extraction solution:		
	950606-001	D	Untreated glass from jar D
	950606-002	F	Untreated glass from jar F
Test I	<p>The samples below were generated during leach tests on glass from jar D.</p> <p>(1) 100 grams of glass and a solution of 720 ml distilled water, 60 grams of salt, and 80 ml of lab-grade bleach were placed in a gallon glass jar and rolled on Gary's roller mill for 6 hours. This was then filtered through first a coarse filter (Whatman 54) and then through a fine filter (1 : membrane filter).</p> <p>(2) Next the glass was returned to the gallon jar along with 1000 ml of distilled water. This was rolled for 2-4 hours. Then the wash water was filtered through the coarse and fine filters.</p>		
	950616-042	060995-1	Leach solution for jar D
	950616-044	061295-1	Rinse water for jar D
	950616-046	061295-3	Treated glass from jar D
Test II	<p>The samples below were generated during leach tests on glass from jar F.</p> <p>(1) 100 grams of glass and a solution of 720 ml distilled water, 60 grams of salt, and 80 ml of lab-grade bleach were placed in a gallon glass jar and rolled on Gary's roller mill for 6 hours. This was then filtered through first a coarse filter (Whatman 54) and then through a fine filter (1 : membrane filter).</p> <p>(2) Next the glass was returned to the gallon jar along with 1000 ml of distilled water. This was rolled for 2-4 hours. Then the wash water was filtered through the coarse and fine filters.</p>		
	950616-043	060995-2	Leach solution for jar F
	950616-045	061295-2	Rinse water for jar F
	950616-047	061295-4	Treated glass from jar F
	The following samples were submitted to get TCLP analysis on the glass from jars B, D, F, and J.		
	950719-284	12118-127-F	Untreated glass from jar F
	950719-285	12118-127-D	Untreated glass from jar D
	950802-039	D	Untreated glass from jar D
	950802-038	121118-127-J	Untreated glass from jar J
	950802-040	12118-127-B	Untreated glass from jar B
Test III	<p>The samples below were generated during leach tests on glass from jar D.</p> <p>(1) 400 grams of glass and a solution of 720 ml distilled water, 60 grams of salt, and 80 ml of lab-grade bleach were placed in a gallon glass jar and rolled on Gary's roller mill. The liquid was decanted off the glass. This was then filtered through first a coarse filter (Whatman 54) and then through a fine filter (1 : membrane filter).</p> <p>(2) Next the glass was returned to the gallon jar along with 1000 ml of distilled water. This was rolled. Then the wash water was filtered through the coarse and fine filters.</p>		
	950719-286	070595-1	Leach solution for jar D
	950719-289	070795-1	Rinse water for jar D
	950802-041	12118-127-D1	Treated glass from jar D
	The samples below were generated during leach tests on glass from jar F.		

SAMPLE IDs

	ANALIS ID	Customer ID	Description
Test IV	<p>(1) 400 grams of glass and a solution of 720 ml distilled water, 60 grams of salt, and 80 ml of lab-grade bleach were placed in a gallon glass jar and rolled on Gary's roller mill. The liquid was decanted off the glass. This was then filtered through first a coarse filter (Whatman 54) and then through a fine filter (1 : membrane filter).</p> <p>(2) Next the glass was returned to the gallon jar along with 1000 ml of distilled water. This was rolled. Then the wash water was filtered through the coarse and fine filters.</p>		
	950719-287	071295-1	Rinse water for jar F
	950719-288	070695-1	Leach solution for jar F
	950802-042	12118-127-F1	Treated glass from jar F
Test V	<p>The samples below were generated during leach tests on just endcaps.</p> <p>(1) 110 grams of endcaps were used. The leach solution was 60 g salt, 80 ml of lab-grade bleach, and 720 ml of distilled water. After rolling on the rolling mill, the liquid was decanted off the glass. This was then filtered through first a coarse filter (Whatman 54) and then through a fine filter (1 : membrane filter).</p> <p>(2) Next, the solids were returned to the gallon jar along with 1000 ml of distilled water. This was rolled. Then the wash water was filtered through the coarse and fine filters</p>		
	950824-056 and 057*	12118-131-1	Treated endcaps
	950824-060 and 061*	12118-129-1	Leach solution for endcaps
	950824-066 and 067*	12118-131-2	Rinse water for endcaps
	950921-088	12118-132-1	Untreated endcaps
Test VI	<p>The samples below were generated during leach tests on a mixture of endcaps and glass.</p> <p>(1) 20 grams of endcaps plus 274 grams of glass were used. The leach solution was 60 g salt, 80 ml of lab-grade bleach, and 720 ml of distilled water. After rolling on the rolling mill, the liquid was decanted off the glass. This was then filtered through first a coarse filter (Whatman 54) and then through a fine filter (1 : membrane filter).</p> <p>(2) Next the solids were returned to the gallon jar along with 1000 ml of distilled water. This was rolled. Then the wash water was filtered through the coarse and fine filters</p>		
	950824-054 and 055*	12118-131-3	Treated mixture of endcaps and glass
	950824-062 and 063*	12118-131-4	Rinse water for mixture of endcaps and glass
	950824-064 and 065*	12118-130-1	Leach solution for mixture
	950921-089	12118-132-2	Untreated mixture of glass and endcaps
	<p>The following samples were generated during a test to evaluate the use of rinsing to separate the glass and the phosphor. This test was scaled to model the treatment of the</p>		

* We submitted one sample to ASO. They divided it into two samples

* We submitted one sample to ASO. They divided it into two samples.

* We submitted one sample to ASO. They ran the TCLP and divided the resulting extraction solution into two samples.

SAMPLE IDs

ANALIS ID		Customer ID	
Test VII	crushed bulbs and endcaps from a 3/4 full 55 gallon drum in a 80 gallon overpack.		
	(1) Added 1922 g of crushed glass (628.4 g from jar D and 1294 g from jar B) and 198 g of endcaps to a gallon jar. Wash solution was 2750 ml of process water and 23 g of sodium bisulfite (0.1 N sodium bisulfite solution). This was rolled on Gary's roller mill. Then, the glass and endcaps were separated from the solution and phosphor by pouring through plastic screening. The solution was then filtered through coarse and fine filters. The glass and endcaps were returned to the jar.		
	(2) 2750 ml of process water was added. The jar was rolled again.		
	950928-048	12118-142-2	First rinse
	NA	12118-144-3	Coarse filter from first rinse
	NA	12118-145-1	Membrane filter from first rinse
Test VIII	950928-049	12118-145-2	Second rinse
	950928-051	12118-145-3	Treated solids
	The following samples were generated during a test to evaluate the use of rinsing to separate the glass and the phosphor. This test used the same amount of the same solution as the test described above, but only 1/4 as much glass and endcaps as the test above.		
	(1) Added 1030 g of crushed glass (94.8 g from jar B and 936 g from jar J) and 49.4 g of endcaps to a gallon jar. Wash solution was 2750 ml of process water and 23 g of sodium bisulfite (0.1 N sodium bisulfite solution). This was rolled on Gary's roller mill. Then, the glass and endcaps were separated from the solution and phosphor by pouring through plastic screening. The solution was then filtered through coarse and fine filters. The glass and endcaps were returned to the jar.		
	(2) 2750 ml of process water was added. The jar was rolled again.		
	950922-087	12118-142-1	First rinse
Test IX	NA	12118-144-1	Coarse filter from first rinse
	NA	12118-144-2	Membrane filter from first rinse
	950928-050	12118-145-5	Second rinse
	950928-052	12118-145-4	Treated solids
	The following samples were generated during a test to evaluate leaching with bleach solution.		
	(1) Used 180 g salt, 2160 ml process water, 240 ml lab-grade bleach, 49 grams endcaps, and 870 g glass.		
Test IX	(2) (3) (4) Rinsed three times, each time with 2700 ml process water.		
	951011-060	12252-8-1	Leach solution
	951011-061	12252-14-1	First rinse
	951101-075	12252-14-2	Treated solids
	951101-077	12252-16-1	Second rinse
	951101-076	12252-17-1	Third rinse
The samples below were generated during a test to produce enough phosphor to be tested for mercury by cold vapor AA.			
(1) Seven bulbs (grams) were mixed with 2750 ml of process water and 23 ml of sodium bisulfite.			

SAMPLE IDs

	ANALIS ID	Customer ID	Description
Test X	This was rolled for 4 hours; then stored in the refrigerator over night. The solids and liquids were separated by screening.		
	(2) The solids were then mixed with 2700 ml of process water. This was rolled for 4 hours; then stored in the refrigerator over the weekend. The solids and liquids were separated by screening.		
	(3) The solids were then mixed with another 2700 ml of process water. This was rolled for 4 hours; then stored in the refrigerator overnight.		
	The liquids from the steps above were centrifuged instead of filtered.		
	951114-078	12252-24-1	First rinse - solids
Test XI	951114-099	12252-24-2	Second rinse - solids
	951115-021		First rinse - supernate
	95115-022		Second rinse - supernate
	(1) The samples below were generated during a test during which a new bulb (284.9 g) was crushed and treated with a mixture of 107.2 g salt, 850 ml (937 g) bleach, 560 ml (556.9 g) process water, and 580 g 1N HCl. The pH of this solution was 4.0.		
Test XII	(2) After rolling on rolling mill, this was kept in refrigerator overnight, filtered through plastic porch screen and rinsed with 1425 ml process water. After rolling on rolling mill, this was kept in refrigerator overnight and then, filtered through plastic porch screen.		
	—	12252-33-1	Leach solution
	—	12252-37-2	Rinse water
	951129-150	12252-37-1	Treated solids
Test XII	This was the bisulfite rinse, bleach leach, rinse, rinse, rinse test.		
	(1) The samples below were generated during a test in which a new bulb (284.7 g) was crushed and treated with a mixture of 12.1 g sodium bisulfite in 1365. ml process water. The pH of this solution was 4.0. After rolling on rolling mill, kept in refrigerator overnight. Filtered through plastic porch screen.		
	(2) Next leached with a solution of 108.1 g salt, 574 ml process water, 850 ml (977.6 g) bleach , and 425 g 1N HCl. This solution had a pH of 4. After rolling on rolling mill, this was kept in refrigerator overnight and filtered through plastic porch screen.		
	(3) This was then rinsed with 1444 ml process water and rolled. This was kept in the refrigerator over the long weekend and filtered through the plastic screen.		
	(4) 1449 ml of process water was added ,and the jar was rolled. This was kept in refrigerator overnight and filtered through plastic porch screen.		
	(5) The solids were then rinsed with 1420 ml of water. After rolling on rolling mill, this was kept in refrigerator overnight and filtered through plastic porch screen.		
	(6) The solids were then rinsed with 1434 ml of water. After rolling on rolling mill, this was kept in refrigerator over the weekend and filtered through plastic porch screen.		
	—	12252-33-2	First rinse solution
	—	12252-37-3	Bleach leach solution
	—	12252-45-1	Second rinse water
	—	12252-47-2	Third rinse water

SAMPLE IDs

	ANALIS ID	Customer ID	Description
	–	12252-53-2	Fourth rinse water
	–	12252-55-2	Fifth rinse water
	951206-135	12252-55-1	Treated solids
Test XIII	This was the bleach leach, rinse, rinse, rinse test.		
	(1) The samples below were generated during a test in which a new bulb (281.5 g) was crushed and treated with a mixture of 107 g salt, 570 ml process water, 870 ml (913.2 g) bleach, and HCl. The pH of this solution was 4.0.		
	(2) After rolling on rolling mill, this was kept in refrigerator overnight, filtered through plastic porch screen and rinsed with 1467 ml process water.		
	(3) After rolling on rolling mill, this was kept in refrigerator overnight, filtered through plastic porch screen, and rinsed with 1523 ml of process water.		
	(4) This was kept in the refrigerator over the long weekend, filtered through the plastic screen and rinsed with 1495 ml process water.		
	–	12252-34-1	Leach solution
	–	12252-38-1	First rinse
	–	12252-45-2	Second rinse
	–	12252-48-1	Third rinse
	–	12252-54-2	Fourth rinse
	951206-136	12252-54-1	Treated solids
Test XIV	This was the low bleach leach, rinse, rinse, rinse test.		
	(1) The samples below were generated during a test in which a new bulb (281.4 g) was treated with 107.3 g salt, 1286 ml process water, 140 ml (158.72 g) of bleach, and 104 g 1N HCl. The pH of this solution was 3.8.		
	(2) After rolling on rolling mill, this was kept in refrigerator overnight, filtered through plastic porch screen and rinsed with 1427 ml process water.		
	(3) After rolling on rolling mill, this was kept in refrigerator overnight, filtered through plastic porch screen, and rinsed with 1429 ml of process water. This was rolled, then stored in the refrigerator over the long weekend.		
	(4) This was filtered through the plastic screen and washed with 1477 ml process water.		
	(5) This was kept in the refrigerator overnight, filtered with the plastic screen, and rinsed with 1417 ml process water.		
	–	12252-35-1	Leach solution
	–	12252-38-2	First rinse
	–	12252-46-1	Second rinse
	–	12252-48-2	Third rinse
	–	12252-54-4	Fourth rinse
	951206-138	12252-54-3	Treated solids
	(1) The samples below were generated during a test in which a new bulb (286.2 g) was treated with 12.1 g sodium bisulfite and 1457 ml process water.		
	(2) After rolling on rolling mill, this was kept in refrigerator overnight, filtered through plastic		

SAMPLE IDs

	ANALIS ID	Customer ID	Description
Test XV	porch screen and rinsed with 1426 ml process water and 1.8 g sodium bisulfite.		
	(3) After rolling on rolling mill, this was kept in refrigerator overnight, filtered through plastic porch screen, and rinsed with 2 g sodium bisulfite in 1412 g process water. After rolling on rolling mill, this was kept in refrigerator overnight, and filtered through plastic porch screen.		
	(4) This was then washed with 2 g sodium bisulfite in 1370 ml of process water. After rolling on rolling mill, this was kept in refrigerator over the long weekend, and filtered through plastic porch screen.		
	(5) This was then washed with 2 g sodium bisulfite in 1451 ml process water. After rolling on rolling mill, this was kept in refrigerator overnight, and filtered through plastic porch screen.		
	(6) This was then washed with 2.3 g sodium bisulfite in 1368 ml process water. After rolling on rolling mill, this was kept in refrigerator overnight, and filtered through plastic porch screen.		
	—	12252-35-2	First rinse
	—	12252-39-1	Second rinse
	—	12252-46-2	Third rinse
	—	12252-49-1	Fourth rinse
	—	12252-54-6	Fifth rinse
	951206-137	12252-54-5	Treated solids
Test XVI	(1) The samples below were generated during a test in which a new bulb (284.2 g) was treated with 107.2 g salt, 575 ml process water, 870 ml (973 g) bleach, and 517 g 1N HCl. The pH of this solution was 4.0.		
	(2) After rolling on rolling mill, this was kept in refrigerator overnight, filtered through plastic porch screen and rinsed with 1427 ml process water. After rolling on rolling mill, this was kept in refrigerator over the long weekend, and filtered through plastic porch screen.		
	(3) The solids were then treated with 107 g salt, 583 ml process water, 850 ml (937 g bleach), and 418 g HCl. The pH of the solution was 4. After rolling on rolling mill, this was kept in refrigerator overnight, and filtered through plastic porch screen.		
	(4) The solids were then treated with 108.6 g salt, 540 ml process water, 850 ml (929.5 g) bleach, and 532.7 g 1 N HCl. The pH of this solution was 4. After rolling on rolling mill, this was kept in refrigerator overnight, and filtered through plastic porch screen.		
	(5) The solids were washed with 1354 ml process water. After rolling on rolling mill, this was kept in refrigerator over the weekend, and filtered through plastic porch screen.		
	(6) The solids were washed with 1349 ml process water. After rolling on rolling mill, this was kept in refrigerator overnight, and filtered through plastic porch screen.		
	—	12252-36-1	First leach
	—	12252-44-1	First rinse
	—	12252-47-1	Second leach
	—	12252-53-1	Third leach
	—	12252-55-1	Second rinse
	—	12252-56-2	Third rinse

SAMPLE IDs

	ANALIS ID	Customer ID	Description
	951208-067	12252-56-1	Treated solids

Samples marked by "NA" in the "ANALIS ID" column are for Material Sciences.

NOTEBOOK PAGES

Test	Notebook	Pages
I	12118	121-122
II	12118	121-122
III	12118	124-127
IV	12118	124-127
V	12118	128, 131-132
VI	12118	129-132
VII	12118	136,139-142,144-145
VIII	12118	137-142,144-145
IX	12118	147
X	12133 12252	29 22-24, 27
XI	12133 12252	30 28, 33, 37
XII	12133 12252	30 29-30, 33-34, 37, 45, 47, 53, 55, 68
XIII	12133 12252	30 30, 34, 38, 45, 48, 53, 54
XIV	12133 12252	30 31, 34-35, 38, 46, 48, 54
XV	12133 12252	30 31, 35, 39, 46, 49, 54
XVI	12133 12252	30 32, 35-36, 44, 47, 53, 55, 56

APPENDIX B

REVIEW OF ANALYTICAL DATA FOR CRUSHED FLUORESCENT LAMPS

Date: 25 August 1995

To: Beck, Bowser, Bunch, Fellows, Neff, Sellers
CC: Broussard, Gibson, Lyles, Astoria
From: Bostick

Subject: Review of Analytical Data for Crushed Fluorescent Lamps

This note is an update to my earlier memo of 27 July 1995.

1. "Policy Statement"

'RCRA Status of Spent Fluorescent and Other Lamps,' MMES Environmental Advisory (July 1992).

"(MMES's) environmental staff has tested various fluorescent lamps using the (TCLP) and has found that the majority of those lamps tested hazardous for the mercury characteristic...This finding has essentially been corroborated by an industry group and by the EPA. However, interlaboratory analytical variability (same lamps, different labs) can be great.....the variability between labs was nearly three orders of magnitude."

2. TCLP Testing by Lamp Manufactures

This information is based upon the article 'Inter-Laboratory Testing for Mercury by TCLP and Surce Reduction in the Lamp Manufacturing Industry," by V. D. Meyer (from EPA/600/R-92/105, August 1992; copy courtesy of D. Foust, G.E.).

In Phase III of the inter-laboratory study, one contract lab crushed a batch of standard 4-ft lamps (consecutively manufactured units from a single manufacturer), and each participating analytical lab received a subsample of this material for analysis. In Phase IV testing, the participating labs received intact lamps to crush and to perform TCLP testing. Results are summarized in Table 1. There was a great deal of inter-laboratory variation in the reported results, but consensus indicates that the lamps fail the toxicity criterion for mercury (0.20 mg/L in the TCLP leachate).

Table 1. Inter-Laboratory Variation in TCLP Results for Mercury in Fluorescent Lamps

Laboratory	Mean TCLP results for Hg (mg/L):	
	Crushed Lamps	Whole Lamps
1	0.010 ± 0.007	0.012 ± 0.01
2	0.40 ± 0.08	0.58 ± 0.07
3	0.49 ± 0.06	0.44 ± 0.08
4	0.51 ± 0.06	0.47 ± 0.14
5	0.94 ± 0.35	1.87 ± 0.81
6	0.105 ± 0.03	0.02 ± 0.006
7	3.09 ± 2.11	0.08 ± 0.02
8		0.58 ± 0.23
Mean	0.78 ± 1.21	0.505 ± 0.63

3. TCLP Testing at ORNL

Source: M. P. Maskarinec, "Leaching of Mercury from Fluorescent Light Bulbs," ORNL/CF-91/361 (November 12, 1991). ['Internal Use Only']

Previous testing by the EP-Tox protocol had not indicated that Fluorescent lamps were characteristically hazardous (e.g., Hg in the extract solution was consistently less than the toxicity defining criterion of 0.20 mg/L). This study was intended to compare Toxicity Characteristic testing performance for Fluorescent Lamps, as performed by the earlier (EP-Tox) and more recent (TCLP) protocols. Used bulbs delivered to lab intact, before size-reduction for TCLP. Results summarized in Table 2 (10 different bulb types, one blank):

Table 2: Analysis for Mercury in TCLP extract of Crushed Fluorescent Lamps

IAL ID	Hg (mg/L) in TCLP Extract (duplicate samples)	
911101-067,-068	0.75, 0.71	(fail)
911101-069,-070	0.31, 0.26	(fail)
911101-071,-072	0.26, 0.19	(fail)
911101-073,-074	0.86, 0.91	(fail)
911101-075,-076	0.15, 0.12	
911101-077,-078	0.18, 0.18	
911101-079,-080	0.12, 0.10	
911101-081,-082	0.17, 0.17	
911101-083,-084	0.08, 0.07	
911101-085,-086	0.06, 0.06	
(blank	<0.001)	

Naively assuming a normal distribution for the results, the average (mean/median) concentration of mercury in the extract is 0.29 ($\pm$ 0.28) mg/L; thus the median value is greater than the Hg toxicity criterion (0.20 mg/L). These data apparently are the basis of the July 1992 advisory. Maskarinec presented no data for other elements (not run?), so no guidance is provided for (e.g.) Pb toxicity characteristic.

4. TCLP Testing for Crushed Fluorescent Lamps: Results from Y-12

Table 3 summarizes data from sampling events and analyses at the Y-12 Plant (data courtesy of Ken Cooper of Y-12 and Steve Astoria of Gilbert Commonwealth). These data corroborate the data from ORNL (Section 3, above): mean concentration of mercury in the TCLP extract was 0.27 ($\pm$ 0.16) mg/L. One anomalous incidence was noted for exceedence of the toxicity criterion for cadmium (all other results for Cd were below the analytical detection limit). [note, however, that Cd-doped phosphors have been used in some manufactured lamps to achieve a higher quantum efficiency; see Soules, T. F., and Hoffman, M. V., 'Luminescent Materials (Phosphors)', in Kirk-Othmer Encyclopedia of Chemical Technology, 3rd ed., Vol. 14, pp. 527-545.]

At the bottom of the Table 3, we list the Universal Treatment Standard (UTS) criteria. Since the waste is designated 'D009' (characteristically hazardous for mercury), the waste is prohibited from land disposal until it is conditioned to meet the UTS criterion for mercury (i.e., the conditioned waste must yield a mercury concentration in the TCLP extract of <0.025 mg/L).

5. TCLP Testing for Crushed Fluorescent Lamps Stored at K-25

The data in Table 4 is a compilation of TCLP results provided courtesy of Tom Broussard. All

available data for this population of waste sampled from storage drums (N=7) indicate that Hg in TCLP extract is below the TCLP toxicity criterion; labeling the storage drums 'D009' (hazardous waste, Hg-contaminated) was a management decision (justifiable, given the data from Tables 1-3).

Although the number of data are small (N=7), we can fit data to a normal distribution, and compute the theoretical % probability for exceedence of the 0.2 mg/L criterion. [Assuming all data are equally valid, the projected exceedence of criterion would be ~ 0.5% of the population]. No other regulated metal, including Cd, was determined to exceed the applicable toxicity criterion.

6. Uranium Contamination in the Crushed Lamps

Samples from the recent (1994/1995) Y-12 and K-25 sampling campaigns indicate that little or no significant uranium exists in the stored samples (Table 5). The uranium concentration in the solid debris averaged <0.4 ug/g (or < 0.4 parts-per-million, ppm). Only one sample (E950520-111, from Y-12) had sufficient uranium (0.21 ug/g) to permit an estimate of the isotopic abundance of U-235; the value of 0.30 wt% is less than the abundance in natural (nonenriched) uranium (0.71 wt% U-235).

Assuming 1 ug U per gram of lamp material (i.e., 1 ug/g, maximum background value reported in Table 4), and an isotopic abundance of 0.3 wt% (and thus a specific activity ~ 4.8E-7 Ci/g), the estimated activity in the debris solids is ~ 0.5 picocuries (pCi) per g. The Waste Acceptance Criteria for the Y-12 Industrial Landfill specifies that uranium must not be present in wastes at activities > 35 pCi/g.

Table 4. Data for TCLP Extraction of Crushed Fluorescent Lamps Stored at K-25 Site
(Results given as mg/L in extract)

AnalIS ID ^a	Hg ^b	Pb ^c	As ^c	Ba ^c	Cd ^c	Cr ^c	Ni ^c	Se ^c	Ag ^c
940608-124	0.033	1.0	<0.25	0.73	<0.015	<0.05	0.97	<0.25	<0.035
940608-123	0.11	1.4	<0.25	7.2	<0.015	0.17	14	<0.25	<0.035
940608-121	0.056	1.1	<0.25	1.1	<0.015	<0.05	1.6	<0.25	<0.035
940608-122	0.066	0.43	<0.25	0.75	<0.015	0.091	0.53	<0.25	<0.035
940608-119	0.065	<0.25	<0.25	4.2	<0.015	0.23	9.8	<0.25	<0.035
940608-120	0.031	<0.25	<0.25	1.1	<0.015	<0.05	1.3	<0.25	<0.035
910115-070	0.169	0.07	0.054	1.4	0.0097	<0.01	1.3	<0.05	<0.01
Median Value	0.076 ±0.05	≤0.64	<0.25	2.4 ±2.5	<0.015	≤0.09	4.2 ±5.4	<0.25	<0.035
TCLP Criterion ^d	0.20	5.0	5.0	100	1.0	5.0	NA	1.0	5.0
UTS Criterion ^e	0.025	0.37	5.0	7.6	0.19	0.86	5.0	0.16	0.30

^a LMES Analytical Laboratory Information System Sample Identification Number

^b Atomic Absorption Spectroscopy (EPA Method 7470)

^c Inductively Coupled Plasma Spectroscopy (EPA Method 6010)

^d Toxicity Characteristic Leaching Procedure (40 CFR 261.24)

^e Universal Treatment Standard for hazardous waste: TCLP extract (nonwastewater) (60 FR 264)

Table 3. Data for TCLP Extraction of Crushed Fluorescent Lamps Stored at Y-12 Site
(Results given as mg/L in extract)

AnaLIS ID ^a	Hg	Pb	As	Ba	Cd	Cr	Se	Ag
E950520-105	0.11	<0.50	<0.50	<10	<0.10	<0.50	<0.50	<0.50
E950520-106	0.42	<0.50	<0.50	<10	<0.10	<0.50	<0.50	<0.50
E950520-107	0.08	<0.50	<0.50	<10	<0.10	<0.50	<0.50	<0.50
E950520-108	0.25	<0.50	<0.50	<10	<0.10	<0.50	<0.50	<0.50
E950520-109	0.15	<0.50	<0.50	<10	1.4/5.7	<0.50	<0.50	<0.50
E950520-110	0.19	<0.50	<0.50	<10	<0.10	<0.50	<0.50	<0.50
E950520-111	0.13	<0.50	<0.50	<10	<0.10	<0.50	<0.50	<0.50
E950520-112	0.26	<0.50	<0.50	<10	<0.10	<0.50	<0.50	<0.50
E950520-113	0.56	<0.50	<0.50	<10	<0.10	<0.50	<0.50	<0.50
E950520-114	0.57	<0.50	<0.50	<10	<0.10	<0.50	<0.50	<0.50
E950520-115	0.37	<0.50	<0.50	<10	<0.10	<0.50	<0.50	<0.50
E950520-116	0.19	<0.50	<0.50	<10	<0.10	<0.50	<0.50	<0.50
E950520-117	0.24	<0.50	<0.50	<10	<0.10	<0.50	<0.50	<0.50
Median Value	0.27 ±0.16	<0.50	<0.50	<10	≤0.55	<0.50	<0.50	<0.50
TCLP Criterion	0.20	5.0	5.0	100	1.0	5.0	1.0	5.0
UTS Criterion ^c	0.025	0.37	5.0	7.6	0.19	0.86	0.16	0.30

^a LMES Analytical Laboratory Information System Sample Identification Number

^b Toxicity Characteristic Leaching Procedure (40 CFR 261.24)

^c Universal Treatment Standard for hazardous waste: TCLP extract (nonwastewater) (60 FR 264)

Table 5. Analysis for Uranium in Crushed Fluorescent Lamps (ug/g, or parts-per-million, by fluorometric analysis)

Sample ID	Site	U, ug/g
940608-119	K-25	<1.0
940608-120	K-25	<1.0
940608-121	K-25	<1.0
940608-122	K-25	<1.0
940608-123	K-25	<1.0
940608-124	K-25	<1.0
E950520-105	Y-12	<0.05
E950520-106	Y-12	0.06
E950520-107	Y-12	<0.045
E950520-108	Y-12	<0.053
E950520-109	Y-12	<0.047
E950520-110	Y-12	<0.048
E950520-111	Y-12	0.21 (U-235 assay: 0.3 wt%)
E950520-112	Y-12	<0.039
E950520-113	Y-12	0.16
E950520-114	Y-12	0.16
E950520-115	Y-12	<0.05
E950520-116	Y-12	<0.059
E950520-117	Y-12	<0.061
Mean Value		<0.37

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