

## **ARGONNE NATIONAL LABORATORY- WEST'S APPROACH TO FILTER CHARACTERIZATION**

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### **ABSTRACT**

Like other DOE facilities, ANL-W uses a variety of nuclear grade, industrial grade, or furnace-type particulate filters to control airborne radioactivity and hazardous contaminants in radiological containment structures or processes. As designed, these filters entrain and ultimately concentrate contaminants in the media. Toxic metal contaminants include cadmium, chromium, lead, and mercury present in sufficient concentrations to exhibit the hazardous waste characteristic of toxicity as defined in 40 CFR 261.24. Radionuclide contaminants deposited in the media may at times accumulate in sufficient quantity to classify the filter as transuranic or remote-handled waste. Upon their removal from the ventilation system, these particulate filters become wastes, which must be characterized to determine their hazardous and radioactive classifications. A well defined filter characterization process is essential for the proper/consistent waste characterization and minimization and for maintaining personnel radiological exposures as-low-as-reasonably-achievable (ALARA) (1,2). ANL-W has developed an approach to filter sampling and characterization to meet these needs.

The ANL-W filter sampling and characterization process is designed to ensure representative sampling and/or process knowledge is utilized in characterizing the filters. The data obtained through sampling and/or process knowledge is used to show compliance with the Resource Conservation and Recovery Act (3) and Treatment/Storage/Disposal Facility Waste Acceptance Criteria.

The ANL-W filter characterization involves the collection of process information, filter handling and sampling, sample analysis, data management filter characterization, and waste handling. Each element of the process is streamlined to ensure proper characterization while minimizing radiological exposure to maintenance workers, samplers, laboratory personnel, and waste handlers.

## **PROCESS INFORMATION**

The following background information is routinely used to accurately and efficiently characterize spent filters: ventilation configuration, potential contaminants, past sampling results, and activities in the area or process materials handled.

Information related to the ventilation configuration includes: manufacturer and facility descriptions, drawings related to the layout of the filters in each bank, filter stages (i.e., one pre-filter and two HEPA filters in series), and filter type (pre- or HEPA filter, metal or wood frame, media with or without aluminum separators, etc.). Potential contaminants include radionuclides that are transuranic, fissile, or beta emitters; toxicity characteristic metals such as cadmium, chromium, lead, or mercury; and listed waste constituents from the management of listed hazardous waste. Past sampling results are utilized to determine potential correlations between filters within a single bank, a filter's radiation dose rates versus its radioactive contaminant levels, and between a filter's total analysis concentrations and the contaminant's leach rates. The activities performed in the area or process materials involved (e.g., lead or cadmium operations) are used to determine the contaminants of concern.

Documentation of this process information provides the basis for sample collection, contaminant analysis, analytical methods, and a defensible final waste determination. This information is archived for future reference.

## **FILTER HANDLING AND SAMPLING**

Complementing the general process information, filter specific waste characterization information must also be collected and documented. For example, the location of the filter within the bank and the date the filter was installed and removed is necessary information in an effort to minimize sampling. In most cases, second stage HEPA filters (those behind first stage HEPA filter) have less contaminant loading than the pre-filter or first stage HEPA filter and may allow for a reduced sampling frequency or less complicated analysis (i.e., total vs TCLP). The time period the filter was in operation may indicate fewer (or more) contaminants of concern. Radiation dose rates and filter weights are also important factors often used to determine which filters are potentially the most contaminated.

A joint Nuclear Regulatory Agency / Environmental Protection Agency (NRC/EPA) guidance (4) was published November 20, 1997 (62 FR 62079) to eliminate unnecessary or redundant waste testing and reduce occupational radiation exposure to individuals conducting waste analysis. Although it was written for commercial mixed waste generator, the preamble stated that "...the guidance may also be useful for Federal Facilities that generate mixed waste". The focus of the guidance is on RCRA Subtitle C compliance in characterizing the hazardous component(s) of mixed low-level radioactive waste (MLLW). The guidance stresses the value of waste knowledge in making hazardous waste determinations and the flexibility allowed in testing mixed wastes in order to minimize radiation hazards. This guidance also offers several strategies to help maintain exposures as-low-as-reasonably-achievable if testing is required. These include samples less than 100 gram in size as well as surrogate samples. ANL-W routinely collects less than 100 gram

samples from filters exceeding 100 mR/hr. Sample size of less than 100 grams is allowable as long as the resulting test is sufficiently sensitive to measure the constituents of interest at the regulatory levels prescribed in the TCLP. Surrogate sampling has not been used in ANL-W filter characterization. As promulgated in this NRC/EPA guidance, the sampling and characterization described in this paper have been tailored to maximize the use of process knowledge and minimize the number and size of the samples collected and analyzed.

ANL-W emphasizes the use of process knowledge in the characterization of radioactive filters, particularly those filters exceeding 200 mR/hr beta-gamma at contact. The key to effective utilization of process knowledge is to obtain adequate, documented data to justify filter characterization with reduced or no sampling. The most frequently used method at ANL-W is the use of sampling data from previously generated filters. Previous data is used to evaluate potential toxicity constituents of concern and contaminant loading within a given ventilation system. Previous data from a single sample analyzed by both total and TCLP is often used to identify expected leach rates for hazardous constituents in a given system.

The regulations require the waste generator to use process knowledge (documented), representative sampling, or a combination of these to characterize waste. ANL-W continually evaluates personnel radiological exposure and waste generated during sampling concerns involved with sampling 100% of the generated filters against the reliance of process knowledge which may involve increased characterization uncertainty. In most cases, a combination of process knowledge and representative sampling yields the greatest reduction in personnel exposures and sample waste generation while minimizing characterization uncertainty.

Unless otherwise determined, the following concepts are accepted in the characterization of ANL-W filters. First, a single core sample obtained by coring through the entire depth of the media is viewed as representative of the filter's media. Second, filters located side-by-side in the same bank often have similar contaminant loading (toxic metals, radionuclides, dirt/dust), which if validated through past sampling, allows for reduced sampling. Third, used filter weights and radiation dose rates (alone or in combination) in these adjacent filters are often excellent indicators of which filter has received the highest contaminant loading. These concepts are utilized to reduce sampling to less than 100% of the filters generated as well as to characterize unsampled filters. Sampling data base updates, review of these data, and comparison of previously obtained data is necessary to validate these typical concepts. A database of this information is a valuable tool in minimizing the number of samples collected and the constituents analyzed.

Media samples are collected using a simple two-inch coring device shown in Figure 1. Unless otherwise indicated through multi-core sampling/analysis of a single filter or by engineering evaluation it is assumed that particulate loading is homogeneous both across the face and through the depth of the filter media. A two-inch diameter core of an eleven-inch deep filter media generally yields 20-40 grams of sample material. This sample mass is generally adequate for both metal and radionuclide analysis.

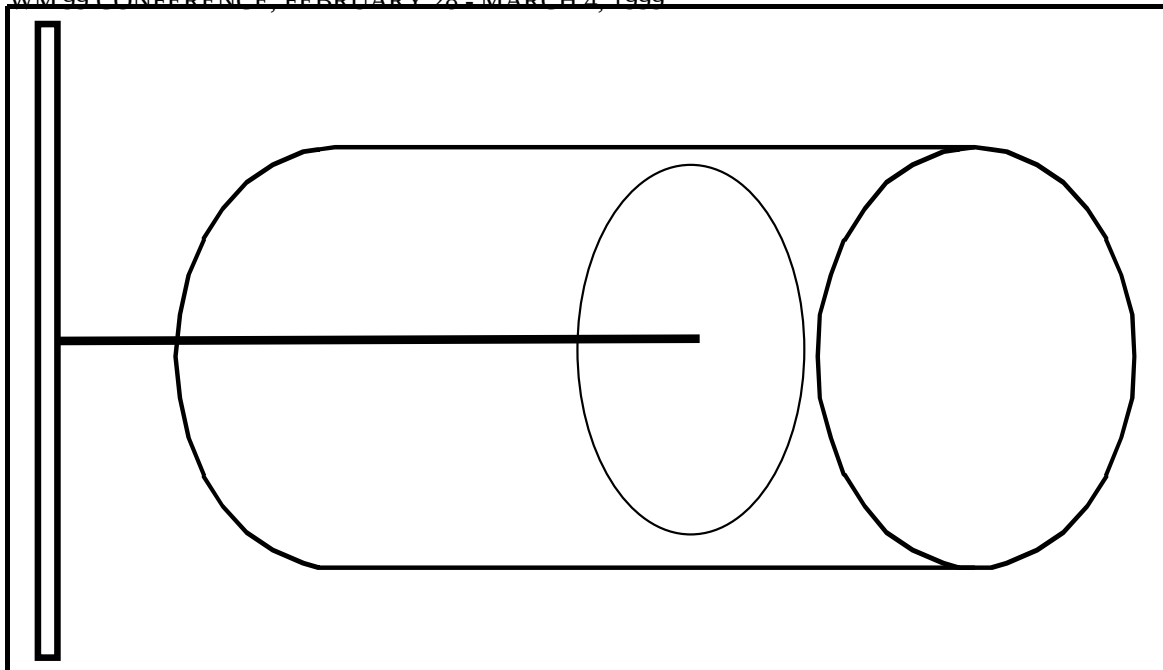


Figure 1. ANL-W 2" Core Sampler

Sampling of filters greater than 1 R/hr at contact may require a slightly modified sampling method. In this case a non-reusable coring device, such as a one-inch diameter thin gauge pipe, may be utilized to collect a smaller sample. The sample is left in the coring device for transfer to the analytical laboratory. The disposable core sampler provides beta shielding and minimizes the spread of contamination while transferring the sample from core sampler to sample container or from sample container to sample preparation container.

Collection of samples using the two-inch core sampler is a proven method for collecting samples without spreading radioactive contamination. As a precaution, most radioactively contaminated filters are sampled in radiological control containment with personnel wearing personnel protective equipment (PPE) including respiratory protection. The two-inch coring device allows the collection of samples without sampling personnel touching the sample or disturbing the media pleats. The one-inch disposable sampler provides an additional measure of radiation and contamination control.

Once the sample is cored from the media it is containerized either in wide mouth polyethylene bottles, Ziplock bags, or polyethylene sleeve. The sample container is weighed before use and after the sample has been collected to determine the mass of the core sample. The core sample weight is required for calculating the mass of the filter media. Chain-of-custody is implemented from the time of sample collection through sample analysis.

Good practices would dictate that sampling should be performed on the day the filters are removed from service ("generated"). Radiological considerations often lead to delays in this practice. Filter change-outs are often performed in "campaigns" where a bank of filters is replaced over a period of time lasting several shifts to several weeks. Once the change-out campaign is completed the filters are sampled in a similar campaign format. Campaign sampling

is very effective in reducing personnel exposure and minimizing PPE waste.

An additional consideration to reduce exposure and waste generation is to establish a documented sampling sequence. Filters may be group together based on such things as operational time, weight, dose rate, system contaminant carryover, stages within the system, etc. The filter sets can then be placed in an appropriate sampling order. Sampling, analysis, and characterization of one set of sampled filters should lead to a reduction in sampling/analysis for subsequent sampling sets.

In some cases filters may be generated which have very high contact dose rates (greater than 10 R/hr at contact) combined with inadequate process knowledge to make a waste determinations. Radiological exposure to personnel associated with the sampling and analysis of these types of filters is often deemed unacceptable. Knowledge of the potential radionuclides in the filter media can substantially reduce personnel exposures. Often it is determined that the best practice is to store the filters for a period of time to allow for radioactive decay of the particulates entained in the filter medial. Storage of uncharacterized, potentially mixed waste filters may create a regulatory liability that should be discussed with your local regulatory agency. Figure 2 illustrates the basic steps incorporated into sampling and characterization activities.

### **SAMPLE ANALYSIS**

Total analysis allows for a smaller sample mass and generates significantly less waste than does the TCLP procedure, for this reason ANL-W commonly requests total analysis first followed by TCLP analysis if constituents are present in sufficient quantity. As stated in SW-846 Method 1311 (5), the TCLP need not be run if a total analysis demonstrates that the metals are not present or are present "...at such low concentrations that the appropriate regulatory levels could not possibly be exceeded...". Based on this statement, a portion of the 20 – 40 gram core sample is analyzed first by total then if the whole filter is judged to have a potential to fail TCLP the TCLP analysis will be performed. If TCLP is necessary, a less than 100 gram sample is used and the laboratory need only analyze for the detected constituent(s).

Table I summarizes the Universal Treatment Standards for the Twelve Metal Constituents (non-wastewater) and the associated Toxicity Characteristic TCLP and UHC limits. The heading titled "Total Concentration Thresholds" identifies the total metal thresholds at which a constituent would fail TCLP if 100% of the constituent leached.

Many of the filters used at ANL-W have filter media with aluminum separators between each pleat. This aluminum is difficult for the Analytical Laboratory (AL) to dissolve in a total analysis. As an alternative, the AL removes the aluminum prior to dissolution. Analysis is performed only on the dissolved media, results are calculated and reported with the mass of the separators included. TCLP analysis is performed with the separators included. The sampler does not remove the separators during sampling.

The following supplies will be needed to perform sampling and characterization:

- 2" diameter reusable core sampler (alternatively, 1" diameter, pre-weighed disposable core sampler)
- Sample bags, or bottles (pre-weighed)
- Sample labels
- COC seals and form(s)
- Analytical request form(s)
- Work Permits
- PPEs
- Sample Collection Worksheet(s) (see Figure 3)
- Filter Excel Characterization Spreadsheet

The following steps should be used when sampling and characterizing filters.

1. Collect and document waste generation information in a logbook.
2. Assign the filter a unique ID number. Weigh and radiation survey the filter. Label the filter with this information and complete the applicable section of the worksheet.
3. Determine which filters need to be sampled for characterization. It is generally assumed that heavier or high dose rate filters will contain a high contaminant loading if all other factors are equal. Review the potential to campaign or sequence the sampling process. Filters selected for sampling should be labeled "TO BE SAMPLED". Filters to be characterized based on other analytical results should be labeled "PENDING CHARACTERIZATION".
4. Move selected filters to sampling location. Move the non-sampled filters to a radioactive material storage area.
5. Place filter face up.
6. Cut an opening in the bag and in the screen if present.
7. Collect sample by slowly rocking, twisting, and pushing the core sampler through the media. Core sampler must be pushed completely through the media.
8. Remove the core sampler and push sample into the pre-weighed sample container. (1" core sampler functions as both the sampling device and the sample container, it must be pre-weighed)
9. Weigh sample and record information on the worksheet. Manage the sample under chain-of-custody.
10. Re-bag filter, making sure that the filter information and sample number is visible on the outermost container. Label filter: "SAMPLED: Waiting for Results/Characterization". Circle "results" or "characterization" as appropriate. Record the sample number and sampler's initials and date on the filter as well.
11. Complete sampling paperwork (worksheets and logbook entries). Submit sample(s) under COC for analysis.
12. Input the applicable information into the characterization spreadsheet. Verify information and review the preliminary calculated waste determination.
13. Review potential discrepancies in the waste determination and make a final determination as appropriate

## 14. Disposition filters.

Figure 2. General Sampling and Characterization Procedure

Table I. Universal Treatment Standards for the Twelve Metal Constituents (non-wastewater)

| Waste | Constituent | TC Limits<br>(mg/L) | Total Threshold,<br>TC Limits +<br>(11g/g) | Phase IV UTS<br>Limits<br>(mg/L TCLP) | Total<br>Threshold,<br>UTS Limits ++ |
|-------|-------------|---------------------|--------------------------------------------|---------------------------------------|--------------------------------------|
| D005  | Barium      | 100                 | 2000                                       | 21.0                                  | 420.0                                |
| D006  | Cadmium     | 1.0                 | 20                                         | 0.11                                  | 2.20                                 |
| D007  | Chromium    | 5.0                 | 100                                        | 0.60                                  | 12.0                                 |
| D008  | Lead        | 5.0                 | 100                                        | 0.75                                  | 15.0                                 |
| D009  | Mercury     | 0.2                 | 4                                          | 0.025                                 | 0.50                                 |
| D010  | Selenium    | 1.0                 | 20                                         | 5.7                                   | 115                                  |
| D011  | Silver      | 5.0                 | 100                                        | 0.14                                  | 2.8                                  |
| na    | Antimony*   | na                  | na                                         | 1.15                                  | 23.0                                 |
| na    | Beryllium*  | na                  | na                                         | 1.22                                  | 24.4                                 |
| na    | Nickel*     | na                  | na                                         | 11.0                                  | 220.0                                |
| na    | Thallium*   | na                  | na                                         | 0.20                                  | 4.0                                  |
| na    | Vanadium*   | na                  | na                                         | 1.6                                   | 32.0                                 |
| na    | Zinc**      | na                  | na                                         | 4.3                                   | 86.0                                 |

+Calculated based TCLP procedure of 100 g of sample per 2000 ml of extract (TC limit x 20 = Total threshold TC limit).

++ Calculated based TCLP procedure of 100 g of sample per 2000 ml of extract (UTS limit x 20 = Total threshold UTS limits).

\* These are UHC only, they are not TC metals.

\*\* These are not UHC.

Generators should note that effective August 24, 1998, the land disposal restrictions (LDR) treatment standards were revised in what is called the "LDR Phase IV Rule" (6). In the Phase IV rule the numerical treatment standards called universal treatment standards (UTS) are now being applied to waste containing toxic metals. As of this date, UTS apply both to the metal for which the waste is regulated as well as to any underlying hazardous constituents (UHCs) present in the waste. As in the case of a hazardous waste determination, determining if the waste is restricted from land disposal (i.e., presence of UHCs and compliant with the UTSs) may be made based on testing or knowledge of the waste.

#### DATA MANAGEMENT, FILTER CHARACTERIZATION

Each filter is weighed and receives a radiation survey upon removal from the ventilation system. The filters are labeled with a unique identification number, weight, pull date, and radiation level. This information is recorded in facility or waste management logs. For sampled filters, a sample

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collection worksheet (Figure 3) is completed to ensure that the necessary characterization information is collected. This worksheet is a supplement to the required, bound sampling logbook.

|                                                 |       |                             |
|-------------------------------------------------|-------|-----------------------------|
| <b>FILTER NUMBER</b> _____                      |       | <b>OLD FILTER #s:</b> _____ |
| <b>USED FILTER WT</b> _____                     |       |                             |
| <b>MEDIA DEPTH</b> (e.g., 5, 8, or 11) _____ in |       |                             |
| <b>MEDIA FACE DIMENSIONS</b> (check applicable) |       |                             |
| 22" x 22" for standard filters _____            |       |                             |
| 22"x 20" for 3 piece media filters _____        |       |                             |
| other _____                                     |       |                             |
| <b>SAMPLE NUMBER</b> _____                      |       | <b>SAMPLE DATE</b> _____    |
| <b>SAMPLE WEIGHT</b> _____ g                    |       |                             |
| <b>SAMPLE DIAMETER</b> (check applicable)       |       |                             |
| 1" _____ 2" _____ 6" _____                      |       |                             |
| <b>FILTER CONSTRUCTION:</b>                     |       |                             |
| model # (if available): _____                   |       |                             |
| description (check applicable)                  |       |                             |
| HEPA                                            | _____ | face screen _____           |
| pre-filter                                      | _____ | back screen _____           |
| metal frame                                     | _____ | metal screen _____          |
| wood frame                                      | _____ | nylon screen _____          |
| aluminum separators                             | _____ |                             |
| no separators                                   | _____ |                             |
| <b>FILTER PULL DATE</b> _____                   |       |                             |
| <b>FILTER DOSE RATE</b> _____ mR/hr             |       |                             |
| <b>SAMPLE DOSE RATE</b> _____ mR/hr             |       |                             |
| <b>COC NUMBER</b> _____                         |       |                             |
| <b>ANALYSIS REQUESTED</b> (check applicable)    |       |                             |
| gross alpha (nCi/g) _____                       |       |                             |
| gross beta (uCi/g) _____                        |       |                             |
| gamma scan (uCi/g) _____                        |       |                             |
| uranium (ug/g) _____                            |       |                             |
| TRU alpha (nCi/g) _____                         |       |                             |
| <b>Totals:</b>                                  |       |                             |
| Ag (ug/g)                                       | _____ | <b>TCLP:</b>                |
| As (ug/g)                                       | _____ | Ag (mg/L) _____             |
| Ba (ug/g)                                       | _____ | As (mg/L) _____             |
| Cd (ug/g)                                       | _____ | Ba (mg/L) _____             |
| Cr (ug/g)                                       | _____ | Cd (mg/L) _____             |
| Hg (ug/g)                                       | _____ | Cr (mg/L) _____             |
| Ni (ug/g)                                       | _____ | Hg (mg/L) _____             |
| Pb (ug/g)                                       | _____ | Ni (mg/L) _____             |
| Se (ug/g)                                       | _____ | Pb (mg/L) _____             |
| TCLP as required                                | _____ | Se (mg/L) _____             |

Figure 3. Filter Sampling Worksheet

Independent of the filter's efficiency or design complexity, all filters consist of two primary components, the filtration media and the support framework. The filtration media is generally constructed of fiberglass and often contains aluminum pleats for support. The support framework consists of a wood, metal, or cardboard frame with seals and media protecting screens. Based on the filter's designed purpose, contaminants accumulate primarily in the filter media while the filter frame components are significantly less contaminated with radioactive and hazardous constituents. Sampling and characterization of particulate filters is based on a determination that analysis of the media is not representative of the entire filter but can be used to characterize the entire filter.

Because the media sample only accounts for the contaminants in the media, characterization of the entire filter involves combining the mass of the frame (considered substantially uncontaminated) with the mass of the contaminated media. Leachate or total concentrations for the entire filter will be somewhat less than the media-only concentrations. Not accounting for the frame in the characterization could lead to an inaccurately high number of hazardous or transuranic waste determinations.

The ANL-W utilizes the following information to characterize the intact filter:

- Used filter weight
- Media face area (cm<sup>2</sup>)
- Sample weight
- Sample radius
- Sample contaminant concentrations.

This information is then used to calculate the following:

Note: The depth of the filter media is not identified and is not necessary when calculating the total weight of the media based on the weight of the sample. All core samples are taken through the entire depth of the media making this measurement unnecessary. Instead, sample density is based upon the weight of the sample in grams (g) divided by the diameter of the sample in square centimeters (cm<sup>2</sup>).

$$\text{Eq.1} \quad \text{SampleDensity}(g / \text{cm}^2) \equiv \text{SampleWt}(g) \div \text{SampleFaceArea}(\text{cm}^2)$$

$$\text{Eq. 2} \quad \text{UsedMediaWt}(g) \equiv \text{SampleDensity}(g / \text{cm}^2) \times \text{MediaFaceArea}(\text{cm}^2)$$

$$\text{Eq. 3} \quad \text{NonMediaWt}(g) \equiv \text{UsedFilterWt}(g) - \text{UsedMediaWt}(g)$$

$$\text{Eq. 4} \quad \text{WholeFilterConc}(\text{ug} / g) \equiv [\text{SampleConc}(\text{ug} / g) \times \text{UsedMediaWt}(g)] \div [\text{NonMediaWt}(g)]$$

These calculations are used in all intact filter calculations whether the analysis is total, TCLP or radioactivity. This characterization yields concentrations most representative of intact filter. If the media and frame are to be separated, the results of the media sample should be applied only to the media.

ANL-W utilizes a Microsoft Excel spreadsheet to quickly perform the characterization using the calculations discussed above. The information for this spreadsheet is obtained from the waste/sampling logs, sample collection worksheets, and analytical results. The generator inputs the necessary data and the spreadsheet performs the calculations. The spreadsheet provides ANL-W with a standardized tool for consistent characterization. Contaminant concentrations in the intact filter are calculated based upon the contaminant concentrations in the filter media sample. Logic formulas in the spreadsheet assist the generator in the preliminary waste characterization. If the sample has only been analyzed for total metals the spreadsheet identifies the constituents in the sample with sufficient concentrations to fail the TCLP. A decision to perform TCLP is then be made.

Final characterization is performed once the following considerations have been addressed.

- Was the sample representative of the waste as generated? This includes both the filter sampled and the use of sample results to characterize an un-sampled filter.
- Was the analysis for total metals or an extract (TCLP)?
- Was the data sufficient to make a complete waste determination?
- How will the filter be disposed, will it be dismantled?

## **WASTE HANDLING**

As a normal practice ANL-W assume that the filter will remain intact for treatment and disposal. If the frame and media are separated, the generator may inadvertently create transuranic media waste from an intact filter initially characterized as LLW. Likewise, mixed waste may be created from a non-hazardous intact filter. To perform a final characterization the ultimate disposition of the filter should be known. Filters disposed or treated intact should be characterized intact. Filters dismantled for any reason should have the media and frame characterized separately.

Excess samples are a similar situation to dismantling the filter. Samples should be characterized based on the results of the sample analysis not on the results of the intact filter characterization. For this reason samples disposed separate from the filter may have a waste classification different than the intact filter from which it was taken.

## **CONCLUSIONS**

Particulate filters generated in DOE facilities often accumulate hazardous constituents, in particular, radionuclides and toxic metals. Waste generators must characterize their waste streams using sampling or process knowledge or a combination of the two. ANL-W utilizes sampling and characterization methods discussed in this paper to characterize these particulate filters. These methods are designed to minimize personnel radiological exposure and the volume of waste generated from the sampling activities while providing the data necessary for characterization. ANL-W filter characterization is based on the premise that a sample of the filter media is not entirely representative of the waste being disposed. ANL-W therefore includes the filter's frame into the waste determination resulting in characterization more representative of the actual waste.

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## REFERENCES

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2. "DOE Radiological Control Manual", DOE/EH-0256T (1992)
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4. "Joint NRC/EPA Guidance on Testing Requirements for Mixed Radioactive and Hazardous Waste", 62 FR 62079, Nov 20 1997
5. "Test Methods for Evaluating Solid Waste", EPA Publication SW-846, current edition
6. "Land Disposal Restrictions Phase IV, Final Rule", 63 FR 28556, May 26 1998