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DOE TECHNICAL STANDARD

Management of Items and Areas Containing Low Levels of Beryllium

U.S. Department of Energy   AREA (SAFT-0103)
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TABLE OF CONTENTS

ACRONYMS

AA	atomic absorption
ACGIH	American Conference of Governmental Industrial Hygienists
ACS	American Chemical Society
AIHA	American Industrial Hygiene Association
ASTM	American Society for Testing and Materials
ATSDR	Agency for Toxic Substances and Disease Registry
BWP	beryllium work plan
CBD	chronic beryllium disease
CBDPP	Chronic Beryllium Disease Prevention Program
CFR	Code of Federal Regulations
CI	confidence internal
CRV	critical value
DHHS	Department of Health and Human Services
DOD	Department of Defense
DOE	Department of Energy
DQO	data quality objective
EPA	Environmental Protection Agency
EQL	estimated quantitation limit
ES	electron spectrometry
FLAA	flame atomic absorption
GFAA	graphite furnace atomic absorption
GM	geometric mean
GSD	geometric standard deviation
ICP	inductively coupled plasma
IDE	interlaboratory detection estimate
IH	industrial hygiene (or hygienist)
IQE	interlaboratory quantitation estimate
ISO	International Standards Organization
IUPAC	International Union of Pure and Applied Chemistry
K-M	Kaplan-Meier
LCL	lower confidence limit
LCS	laboratory control sample
LOD	level of detection
LOQ	level of quantitation
MARSSIM	Multi-Agency Radiation Survey and Site Investigation Manual
MDA	Military D aperture
MDL	method detection limit
MDV	minimum detectable value
MLE	maximum likelihood estimate
MS	mass spectrometry
NIOSH	National Institute for Occupational Safety and Health
NMAM	NIOSH Manual of Analytical Methods
OEL	occupational exposure limit

OGC	Office of General Counsel
OSHA	Occupational Safety and Health Administration
PAT	proficiency analytical testing
PDF	portable document format
PPE	personal protective equipment
QA	quality assurance
QC	quality control
RL	reporting limit
SRM	standard reference material
TLV	Threshold Limit Value
TWA	time-weighted average
UCL	upper confidence limit
UTL	upper tolerance limit

1. BACKGROUND

On December 8, 1999, the Department of Energy (DOE) published title 10 CFR 850 (the Rule) to establish a chronic beryllium disease prevention program (CBDPP) to:
- reduce the number of workers currently exposed to beryllium in the course of their work at DOE facilities managed by DOE or its contractors,
- minimize the levels of, and potential for, exposure to beryllium, and
- establish medical surveillance requirements to ensure early detection of the disease.

On January 4, 2001, DOE issued DOE G 440.1-7A, *Implementation Guide for use with 10 CFR 850, Chronic Beryllium Disease Prevention Program,* to assist line managers in meeting their responsibilities for implementing the CBDPP. That guide describes methods and techniques that DOE considers acceptable in complying with the Rule.

Since that time, as DOE and its contractors have implemented the various provisions of the Rule, numerous issues concerning how to manage items and areas that contain low levels of beryllium have been raised that were not included in the Rule or the implementation guide. In response, DOE formed a release criteria workgroup and held a working meeting to develop guidance for situations not included in the Rule concerning the control of items and areas that contain low levels of beryllium.

The best practices and lessons learned from throughout the DOE complex for managing items and areas that contain low levels of beryllium have been compiled in this Technical Standard under three broad categories:

- Control Levels
- Characterization of Beryllium Levels
- Remediation and Handling

2. CONTROL LEVELS

2.1 Control Levels for Items and Areas

The Rule specifies
- an action level for the concentration of beryllium in air that triggers numerous Rule provisions,
- a housekeeping surface level to ensure adequate process control in areas known to contain beryllium, and
- surface levels as one of the criteria for releasing beryllium-contaminated "equipment or other items."

The Rule [e.g., §850.11(b)(3), §850.35(a)(1)(ii)] also requires exposures be kept as low as possible. The Rule does not, however, specify control levels for conditions in which:
- concentrations of beryllium in air are below the Rule action level,
- surfaces of facilities and areas, or equipment or other items (including waste), contain, or may contain, low levels of beryllium.

DOE sites need to establish the controls they will apply in order to manage the health and safety of their workers and their communities where beryllium levels are found to exceed background but do not exceed the explicit control levels in the Rule. Controls that are appropriate for areas are given in Table 2.1. Similar controls for equipment and building systems and spaces where beryllium is most likely to accumulate over time are given in Table 2.2.

Table 2.1. Controls for Beryllium Control Areas

	Beryllium Regulated Area	Beryllium Controlled Area	Beryllium Buffer Area	Background Area[1]
	Airborne or the potential for airborne beryllium contamination at or above 0.2 µg/m^3 as an 8-hr time-weighted average (TWA)	Surface contamination[2] at or above 0.2 µg/100 cm^2 and/or reasonably expected to have airborne greater than or equal to 0.1µg/m^3 but less than 0.2 µg/m^3 as an 8-hr TWA	Surface contamination at or above 0.05 µg/100 cm^2 but below 0.2 and/or reasonably expected to have airborne greater than or equal to 0.05µg/m^3 but less than 0.1 as an 8-hr TWA[3]	Surface contamination below 0.05 µg/100 cm^2 and airborne below 0.05µg/m^3 as an 8-hr TWA[3] **NO REQUIREMENTS**
POSTING				
DANGER BERYLLIUM REGULATED AREA BERYLLIUM CAN CAUSE LUNG DAMAGE CANCER HAZARD AUTHORIZED PERSONNEL ONLY	X			
CAUTION BERYLLIUM CONTROLLED AREA CONTAMINATED WITH BERYLLIUM BERYLLIUM CAN CAUSE LUNG DAMAGE CANCER HAZARD AUTHORIZED PERSONNEL ONLY		X		

[1]Background includes areas determined to have no expectation of beryllium contamination after conducting the 10 CFR 850.20 *Baseline Beryllium Inventory*.

[2]Surface contamination refers to removable contamination as defined in 10 CFR 850.3 "Removable contamination means beryllium contamination that can be removed from surfaces by nondestructive means, such as casual contact, wiping, brushing or washing.

[3] The most common analytical reporting limit (RL) for beryllium is 0.05 µg per sample. (See Sect. 3.8.2.5.) The preferred level of beryllium to consider as background in air is 0.01 µg/m^3. This preferred level could be measured in an 8-hour sample using ICP-MS but that is costly and highly subject to matrix interference (See sect. 3.8.2.4.) This level also could be measured with a sample of five cubic meters of air. High volume or long term sampling to obtain five cubic meters of air is appropriate for measuring background levels when the purpose is to determine background but not to obtain a personal exposure level.

	Beryllium Regulated Area	Beryllium Controlled Area	Beryllium Buffer Area	Background Area[1]
	Airborne or the potential for airborne beryllium contamination at or above 0.2 $\mu g/m^3$ as an 8-hr time-weighted average (TWA)	Surface contamination[2] at or above 0.2 $\mu g/100$ cm^2 and/or reasonably expected to have airborne greater than or equal to 0.1$\mu g/m^3$ but less than 0.2 $\mu g/m^3$ as an 8-hr TWA	Surface contamination at or above 0.05 $\mu g/100$ cm^2 but below 0.2 and/or reasonably expected to have airborne greater than or equal to 0.05$\mu g/m^3$ but less than 0.1 as an 8-hr TWA[3]	Surface contamination below 0.05 $\mu g/100$ cm^2 and airborne below 0.05$\mu g/m^3$ as an 8-hr TWA[3] **NO REQUIREMENTS**
NOTICE BERYLLIUM BUFFER AREA THIS BUILDING MAY CONTAIN BERYLLIUM SAFETY ASSESSMENT REQUIRED PRIOR TO INTRUSIVE WORK ACTIVITIES BERYLLIUM PERSONNEL WITH BERYLLIUM MEDICAL RESTRICTIONS NOT ALLOWED			X	
AUTHORIZATION/ QUALIFICATION				
Beryllium Workers Only	X			
Workers Medically Screened and Trained for Potential Intrusive Beryllium Work		X		
No Access to Beryllium Medically Restricted Workers No additional requirement for non-intrusive work			X	

4

[1]Background includes areas determined to have no expectation of beryllium contamination after conducting the 10 CFR 850.20 *Baseline Beryllium Inventory*.
[2]Surface contamination refers to removable contamination as defined in 10 CFR 850.3 "Removable contamination means beryllium contamination that can be removed from surfaces by nondestructive means, such as casual contact, wiping, brushing or washing.
[3] The most common analytical reporting limit (RL) for beryllium is 0.05 μg per sample. (See Sect. 3.8.2.5.) The preferred level of beryllium to consider as background in air is 0.01 $\mu g/m^3$. This preferred level could be measured in an 8-hour sample using ICP-MS but that is costly and highly subject to matrix interference (See sect. 3.8.2.4.) This level also could be measured with a sample of five cubic meters of air. High volume or long term sampling to obtain five cubic meters of air is appropriate for measuring background levels when the purpose is to determine background but not to obtain a personal exposure level.

	Beryllium Regulated Area	Beryllium Controlled Area	Beryllium Buffer Area	Background Area[1]
	Airborne or the potential for airborne beryllium contamination at or above 0.2 µg/m^3 as an 8-hr time-weighted average (TWA)	Surface contamination[2] at or above 0.2 µg/100 cm^2 and/or reasonably expected to have airborne greater than or equal to 0.1µg/m^3 but less than 0.2 µg/m^3 as an 8-hr TWA	Surface contamination at or above 0.05 µg/100 cm^2 but below 0.2 and/or reasonably expected to have airborne greater than or equal to 0.05µg/m^3 but less than 0.1 as an 8-hr TWA[3]	Surface contamination below 0.05 µg/100 cm^2 and airborne below 0.05µg/m^3 as an 8-hr TWA[3] **NO REQUIREMENTS**
ENGINEERING CONTROLS				
Ventilation for Personnel Protection	X			
Testing and Use of Laboratory-Type Hoods	X			
Areas where beryllium-contaminated personal protective equipment is removed is maintained under negative pressure or are located so as to minimize dispersion of beryllium into clean areas	X			
MONITORING				
Personal air monitor	X	X	X	
Surface wipes	X	X	X	
HYGIENE				
No eating, drinking, chewing gum, use of tobacco products, applying cosmetics, taking medication, or storing food	X	X	X	
HOUSEKEEPING				

[1]Background includes areas determined to have no expectation of beryllium contamination after conducting the 10 CFR 850.20 *Baseline Beryllium Inventory*.

[2]Surface contamination refers to removable contamination as defined in 10 CFR 850.3 "Removable contamination means beryllium contamination that can be removed from surfaces by nondestructive means, such as casual contact, wiping, brushing or washing.

[3] The most common analytical reporting limit (RL) for beryllium is 0.05 µg per sample. (See Sect. 3.8.2.5.) The preferred level of beryllium to consider as background in air is 0.01 µg/m^3. This preferred level could be measured in an 8-hour sample using ICP-MS but that is costly and highly subject to matrix interference (See sect. 3.8.2.4.) This level also could be measured with a sample of five cubic meters of air. High volume or long term sampling to obtain five cubic meters of air is appropriate for measuring background levels when the purpose is to determine background but not to obtain a personal exposure level.

	Beryllium Regulated Area	Beryllium Controlled Area	Beryllium Buffer Area	Background Area[1]
	Airborne or the potential for airborne beryllium contamination at or above 0.2 $\mu g/m^3$ as an 8-hr time-weighted average (TWA)	Surface contamination[2] at or above 0.2 $\mu g/100\ cm^2$ and/or reasonably expected to have airborne greater than or equal to 0.1$\mu g/m^3$ but less than 0.2 $\mu g/m^3$ as an 8-hr TWA	Surface contamination at or above 0.05 $\mu g/100\ cm^2$ but below 0.2 and/or reasonably expected to have airborne greater than or equal to 0.05$\mu g/m^3$ but less than 0.1 as an 8-hr TWA[3]	Surface contamination below 0.05 $\mu g/100\ cm^2$ and airborne below 0.05$\mu g/m^3$ as an 8-hr TWA[3] **NO REQUIREMENTS**
Housekeeping methods (such as the use of a wet mop, sticky tack cloth, or a vacuum cleaner equipment with the HEPA air filter) are used to suppress the formation of aerosols	X	X	X	
WORK CONTROLS				
Work control document, including hazard controls	X	X	X	
Be entry log	X	X	X	
Beryllium-labeled cleaning equipment is controlled and dedicated for hazardous material use only	X	X	X	
Structural barriers or structural boundary identifiers (i.e., blue tape, flagging, plastic chains, or painted strips) are used to adequately alert workers to the boundaries of such areas	X	X	X	
Ensure that workers shower at the end of a work shift	X	X		

6

[1]Background includes areas determined to have no expectation of beryllium contamination after conducting the 10 CFR 850.20 *Baseline Beryllium Inventory*.

[2]Surface contamination refers to removable contamination as defined in 10 CFR 850.3 "Removable contamination means beryllium contamination that can be removed from surfaces by nondestructive means, such as casual contact, wiping, brushing or washing.

[3] The most common analytical reporting limit (RL) for beryllium is 0.05 μg per sample. (See Sect. 3.8.2.5.) The preferred level of beryllium to consider as background in air is 0.01 $\mu g/m^3$. This preferred level could be measured in an 8-hour sample using ICP-MS but that is costly and highly subject to matrix interference (See sect. 3.8.2.4.) This level also could be measured with a sample of five cubic meters of air. High volume or long term sampling to obtain five cubic meters of air is appropriate for measuring background levels when the purpose is to determine background but not to obtain a personal exposure level.

	Beryllium Regulated Area	Beryllium Controlled Area	Beryllium Buffer Area	Background Area[1]
	Airborne or the potential for airborne beryllium contamination at or above 0.2 µg/m^3 as an 8-hr time-weighted average (TWA)	Surface contamination[2] at or above 0.2 µg/100 cm^2 and/or reasonably expected to have airborne greater than or equal to 0.1µg/m^3 but less than 0.2 µg/m^3 as an 8-hr TWA	Surface contamination at or above 0.05 µg/100 cm^2 but below 0.2 and/or reasonably expected to have airborne greater than or equal to 0.05µg/m^3 but less than 0.1 as an 8-hr TWA[3]	Surface contamination below 0.05 µg/100 cm^2 and airborne below 0.05µg/m^3 as an 8-hr TWA[3] NO REQUIREMENTS
Use appropriate labels, tags, and forms	X	X	X	
PERSONAL PROTECTIVE EQUIPMENT				
Respiratory protection	X	X		
Personal protective equipment	X	X		
PPE, regardless of measured exposure levels, may be used voluntarily if it is determined not to introduce new hazards	X	X	X	
Segregate non-disposable company-issued protective clothing into properly labeled containers before transfer to the laundry	X	X	X	
TRAINING				
General beryllium awareness training	X	X	X	
Beryllium hazard recognition and control training for workers that may access areas that contain beryllium		X	X	
Beryllium hazard recognition and control training for beryllium workers	X			

7

[1]Background includes areas determined to have no expectation of beryllium contamination after conducting the 10 CFR 850.20 *Baseline Beryllium Inventory*.
[2]Surface contamination refers to removable contamination as defined in 10 CFR 850.3 "Removable contamination means beryllium contamination that can be removed from surfaces by nondestructive means, such as casual contact, wiping, brushing or washing.
[3] The most common analytical reporting limit (RL) for beryllium is 0.05 µg per sample. (See Sect. 3.8.2.5.) The preferred level of beryllium to consider as background in air is 0.01 µg/m^3. This preferred level could be measured in an 8-hour sample using ICP-MS but that is costly and highly subject to matrix interference (See sect. 3.8.2.4.) This level also could be measured with a sample of five cubic meters of air. High volume or long term sampling to obtain five cubic meters of air is appropriate for measuring background levels when the purpose is to determine background but not to obtain a personal exposure level.

Table 2.2. Potential Beryllium-contaminated Equipment, Building Systems, and Spaces

	Potential Beryllium Contaminated Systems and/or Spaces 1	Potential Beryllium Contaminated Systems and/or Spaces 2	Potential Beryllium Contaminated Systems and/or Spaces 3
	No external Be surface contamination measured. Verifiable records or verbal accounts of past use of equipment and/or systems and/or space	No external Be surface contamination measured. Records or verbal accounts of past use of equipment and/or systems and/or space alleged but not verifiable	No external Be surface contamination measured. No verifiable records or verbal accounts of past use of equipment and/or systems and/or space
POSTING			
DANGER BERYLLIUM REGULATED AREA BERYLLIUM CAN CAUSE LUNG DAMAGE CANCER HAZARD AUTHORIZED PERSONNEL ONLY			
CAUTION BERYLLIUM CONTROLLED AREA CONTAMINATED WITH BERYLLIUM BERYLLIUM CAN CAUSE LUNG DAMAGE CANCER HAZARD AUTHORIZED PERSONNEL ONLY			

	Potential Beryllium Contaminated Systems and/or Spaces 1	Potential Beryllium Contaminated Systems and/or Spaces 2	Potential Beryllium Contaminated Systems and/or Spaces 3
	No external Be surface contamination measured. Verifiable records or verbal accounts of past use of equipment and/or systems and/or space	No external Be surface contamination measured. Records or verbal accounts of past use of equipment and/or systems and/or space alleged but not verifiable	No external Be surface contamination measured. No verifiable records or verbal accounts of past use of equipment and/or systems and/or space
NOTICE BERYLLIUM BUFFER AREA THIS BUILDING MAY CONTAIN BERYLLIUM SAFETY ASSESSMENT REQUIRED PRIOR TO INTRUSIVE WORK ACTIVITIES PERSONNEL WITH BERYLLIUM MEDICAL RESTRICTIONS NOT ALLOWED	X	X	X
AUTHORIZATION/ QUALIFICATION			
Beryllium Workers Only	X		
Beryllium Screened Workers		X	
No Access to Beryllium Medically Restricted Workers. No additional requirements for non-intrusive work			
No requirements			X
ENGINEERING CONTROLS			
Ventilation for Personnel Protection			
Testing and Use of Laboratory-Type Hoods			

	Potential Beryllium Contaminated Systems and/or Spaces 1	Potential Beryllium Contaminated Systems and/or Spaces 2	Potential Beryllium Contaminated Systems and/or Spaces 3
	No external Be surface contamination measured. Verifiable records or verbal accounts of past use of equipment and/or systems and/or space	No external Be surface contamination measured. Records or verbal accounts of past use of equipment and/or systems and/or space alleged but not verifiable	No external Be surface contamination measured. No verifiable records or verbal accounts of past use of equipment and/or systems and/or space
Areas where beryllium-contaminated personal protective equipment is removed is maintained under negative pressure or are located so as to minimize dispersion of beryllium into clean areas			
MONITORING			
Personal air monitor	X	X	
Surface wipes	X	X	
HYGIENE			
No eating, drinking, chewing gum, use of tobacco products, applying cosmetics, taking medication, or storing food	X		
HOUSEKEEPING			
Housekeeping methods (such as the use of a wet mop, sticky tack cloth, or a vacuum cleaner equipment with the HEPA air filter) are used to suppress the formation of aerosols	X	X	
WORK CONTROLS			
Work control document, including hazard controls	X	X	
Be entry log	X	X	

	Potential Beryllium Contaminated Systems and/or Spaces 1	Potential Beryllium Contaminated Systems and/or Spaces 2	Potential Beryllium Contaminated Systems and/or Spaces 3
	No external Be surface contamination measured. Verifiable records or verbal accounts of past use of equipment and/or systems and/or space	No external Be surface contamination measured. Records or verbal accounts of past use of equipment and/or systems and/or space alleged but not verifiable	No external Be surface contamination measured. No verifiable records or verbal accounts of past use of equipment and/or systems and/or space
Beryllium-labeled cleaning equipment is controlled and dedicated for hazardous material use only	X		
Structural barriers or structural boundary identifiers (i.e., blue tape, flagging, plastic chains, or painted strips) are used to adequately alert workers to the boundaries of such areas	X	X	
Ensure that workers shower at the end of a work shift	X		
Use appropriate labels, tags, and forms	X	X	
PERSONAL PROTECTIVE EQUIPMENT			
Respiratory protection	X		
Personal protective equipment	X		
PPE, regardless of measured exposure levels, may be used voluntarily if it is determined not to introduce new hazards	X	X	
Segregate non-disposable company-issued protective clothing into properly labeled containers before transfer to the laundry	X	X	
TRAINING			
General beryllium awareness training	X		

	Potential Beryllium Contaminated Systems and/or Spaces 1	Potential Beryllium Contaminated Systems and/or Spaces 2	Potential Beryllium Contaminated Systems and/or Spaces 3
	No external Be surface contamination measured. Verifiable records or verbal accounts of past use of equipment and/or systems and/or space	No external Be surface contamination measured. Records or verbal accounts of past use of equipment and/or systems and/or space alleged but not verifiable	No external Be surface contamination measured. No verifiable records or verbal accounts of past use of equipment and/or systems and/or space
Beryllium hazard recognition and control training for workers that may access areas that contain beryllium		X	
Beryllium hazard recognition and control training for beryllium workers	X		

2.2 Applicability of 10 CFR 850 to Various Forms of Beryllium

2.2.1 Forms of Beryllium Included in Rulemaking Justification

A key consideration for a site's CBDPP is determining if a beryllium-containing material is included within the scope of the Rule. Beryllium materials that are covered are beryllium metal, oxides, and alloys. Studies linking exposures to these materials and beryllium disease provided the justification for promulgating the Rule.

2.2.2 Naturally Occurring Forms of Beryllium

Naturally occurring forms of beryllium are the trace amounts of beryllium found in very common materials such as soil, clay, sand, coal, and vegetation. Many similar forms of beryllium have been modified from their natural state by human activity but still can be included in the term "naturally occurring" for this guidance because they also contain trace amounts of beryllium found in very common materials (e.g., cement, concrete, acoustic ceiling tiles, abrasive blasting agents, brick, mortar, cinderblock, and fly ash). The beryllium in some of these materials is in the form of extremely insoluble silicates and alumino-silicates, which are believed to be biologically inert and therefore have no health impact.

2.2.3 Beryllium in Background Soil

The Rule does not apply to the beryllium in background soil, which is perhaps the most common naturally occurring form of beryllium. The definition of beryllium in 10 CFR 850.3, *beryllium means elemental beryllium and any insoluble beryllium compound or alloy containing 0.1 percent beryllium or greater that may be released as an airborne particulate*, suggests that the Rule is applicable to any form of beryllium that may become airborne, but the Rule contains an explicit exception. The exception is found in 10 CFR 850.31(b)(1), Release Criteria, in reference to releasing beryllium-contaminated equipment or items. That section requires the employer to ensure that *the removable contamination level of equipment or item surfaces does not exceed the higher of 0.2 µg/100 cm^2 or the concentration level of beryllium in soil at the point of release, whichever is greater*. Therefore, beryllium in background soil is not included in the Rule's release criteria provision.

2.2.4 Naturally Occurring Forms of Beryllium Other Than the Forms in Soil

Questions have been raised regarding a link between a number of extremely common naturally occurring forms of beryllium and beryllium disease. No documented cases of beryllium disease resulting from exposures to these forms of beryllium were identified during the rulemaking procedure. Section C, *Health Effects*, of the preamble to 10 CFR 850 (http://www.eh.doe.gov/be/docs/berule.pdf) provides a comprehensive discussion

of the occurrences of chronic beryllium disease (CBD) and beryllium sensitivity known of at that time. The exception for beryllium in background soil was included in the Rule in response to commenters' concerns that background soil could otherwise unintentionally be included in the Rule.

The issue of unintentional inclusion of common naturally occurring forms of beryllium other than those in background soil was not raised during the rulemaking process. Consequently, those forms of beryllium are not explicitly excluded from the Rule. A determination that those forms of beryllium were not considered in the rulemaking process and therefore are not covered by the Rule would require a formal interpretation by DOE's Office of General Counsel (OGC). OGC is the only DOE office that is authorized to interpret DOE regulations. OGC may determine that, even if it was unintentional, common naturally occurring forms of beryllium other than those in background soil are covered by the Rule. In that eventuality, a revision of the Rule would be required to exclude those forms of beryllium from the Rule.

2.2.5 Applicability of Other Standards to Naturally Occurring Forms of Beryllium

Standards other than 10 CFR 850 may apply to common naturally occurring forms of beryllium depending on the provisions of the governing DOE contract, regardless of DOE's ultimate policy concerning the applicability of the Rule to these materials. Many DOE sites incorporate compliance with Occupational Safety and Health Administration (OSHA) regulations and American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Values (TLVs) under their contract. Both OSHA and the ACGIH have indicated that their current exposure limits may not be sufficiently protective. For those reasons, the 10 CFR 850.23 action level of 0.2 $\mu g/m^3$ of beryllium should be applied to airborne exposures to naturally occurring forms of beryllium. Neither OSHA nor ACGIH has surface level limits for any form of beryllium.

2.2.6 Less Than One-Tenth Percent Beryllium in Source Material

The definition of beryllium in the Rule includes compounds or alloys "containing 0.1 percent beryllium or greater that may be released as an airborne particulate." This does not guarantee that work involving a compound or alloy with concentrations of beryllium less than 0.1 percent will not generate exposures that exceed the control levels in the Rule. DOE sites have had a number of experiences in which beryllium airborne levels exceeded the Rule's action level of 0.2 $\mu g/m^3$ when the concentration of beryllium in the source material was well below 0.1 percent.

2.3 Areas to Include in Beryllium Inventory

Areas to include in the beryllium inventory are land, structurs, and areas for which a qualified individual has determined it to be contaminated with removable beryllium:
- greater than 0.2 µg/100 cm^2 based on the results of prior diagnostic sampling or presumed from process knowledge, or
- based on the results of a scoping survey (see Sect. 3.3) using 0.2 µg/100 cm^2 as the specified limit when berylllium is not from soil accumulation.

2.4 Beryllium Risk Assessment

Qualified individuals perform beryllium risk assessments to assess the foreseeable potential risk of exposure to beryllium associated with equipment and other items, real property and buildings, bulk material, and waste. The qualified individual should consider all relevant information such as the types of information listed in Sect. 4.2.1.1, "Records Review," of DOE G 440.1-7A, *Implementation Guide for use with 10 CFR 850, Chronic Beryllium Disease Prevention Program* (http://www.eh.doe.gov/be/policy/g4401-7.pdf). The qualified individual need only consider an amount of information that is sufficient to make a supportable conclusion about the risk of beryllium exposure. The risk assessment becomes the basis for determining the actions necessary to comply with the requirements of 10 CFR 850 or otherwise protect workers and the public from harmful exposures.

3. CHARACTERIZATION

3.1 Statistical Methods for Investigating and Surveying Beryllium Contamination

This section describes the statistical methods that are used for investigating and surveying beryllium surface contamination.

3.1.1 Statistical Methods

The statistical methods used were adapted from the AIHA *Strateg[1]y.* Instructions for downloading statistics software and documentation are posted at http://www.csm.ornl.gov/~frome/aoed. This software produces the metrics described in the AIHA *Strategy[3]* for lognormal and non-lognormal data and censored[2] and non-censored data. The metrics are:

mu	The natural log of the maximum likelihood estimate (MLE) of the geometric mean
se.mu	Standard error of mu
sigma	The natural log of the MLE of the geometric standard deviation
se.sigma	Standard error of sigma
GM	The MLE of the geometric mean
GSD	The MLE of the geometric standard deviation
EX	The MLE of the arithmetic mean
LCLc-95	The MLE of the 95% lower confidence limit of the arithmetic mean
UCLc-95	The MLE of the 95% upper confidence limit of the arithmetic mean
KMmean	The Kaplan-Meier (KM) estimate of the arithmetic mean (distribution-free)
KLCL-95	The KM estimate of the 95% lower confidence limit of the arithmetic mean
KUCL-95	The KM estimate of the 95% upper confidence limit of the arithmetic mean
Obs%95	The observed 95th percentile of the data set
Est%95	The MLE of the 95th percentile
UTLa9595	The MLE of the geometric 95-95 upper tolerance limit (UTL)
z_OEL-xx	The MLE of the standard normal quantile (Zp) of the specified limit
NpUTL9595	The distribution-free estimate of the 95-95 UTL
Maximum	Largest value in the data set
NonDet%	The percentage of results that were <LOQ
n	The number of samples collected
Rsq	The square of the Pearson product moment correlation coefficient for the data and standard normal

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[1] Mulhausen, JR and Damiano, J, *A Strategy for Assessing and Managing Occupational Exposures,* Second Edition , AIHA Press, Fairfax, VA, 1998.

[2] Censored data means that the exact value of each sample measurement is not known. Left censoring is when the measurement is less than some value and right censoring is when the measurement is greater than some value.

m   The number of samples >LOQ

Fex-xx   The MLE of the percent of values exceeding the specified limit

FeLCL-95   The MLE of the 95% lower confidence limit of the percent exceeding the specified limit

FeUCL-95   The MLE of the 95% upper confidence limit of the percent exceeding the specified limit

Fnp-xx   The distribution-free estimate of the percent of values exceeding the specified limit

FnLCL-95   The distribution-free estimate of the 95% lower confidence limit of the percent exceeding the specified limit

FnUCL-95   The distribution-free estimate of the 95% upper confidence limit of the percent exceeding the specified limit

3.1.2 Use of Metrics

The NonDet% and Rsq metrics are used to decide whether MLE (parametric) or distribution-free (nonparametric) statistics will be used. If NonDet% is greater than 70%, the MLE is not reliable and distribution-free metrics should be used. Similarly, an Rsq less than 0.95 indicates that the data do not fit the MLE model well and distribution-free estimates should be used. When they can be used, MLEs provide more confident estimates and are preferred over distribution-free estimates.

1. Sigma and mu and their standard errors are used to calculate the MLE metrics. They are also the slope and intercept of the fitted line used in the log probability plot that can be generated using R software (instructions can be obtained at http://www.csm.ornl.gov/~frome/oed). This plot (lognormal Q-Q plot) provides visual evidence of goodness of fit of the data to the lognormal model.

2. The MLE and KM estimate of arithmetic means and confidence limits are used to compare two or more sets of data to determine if they are significantly different. These might be useful during scoping surveys to guide decisions on whether surfaces should be combined into a single survey unit or kept separate. Means and confidence intervals might also be useful in characterization surveys to compare part-per-million or metal-ratio data to determine if the survey unit is different from background.

3. The various UTL and percent exceedance statistics are used to guide judgments on whether a survey unit is in compliance with specified limits.

    Compliance is indicated if:
    *   The 95-95 UTL is less than the specified limit.
    *   The upper confidence limit of the percent exceeding the specified limit is less than 5%.

Non-compliance is indicated if:
- The lower confidence limit of the percent exceeding the specified limit is 5% or greater.

Additional sampling may be justified if the confidence interval of percent exceedances (the interval between FeULC-95 and FeLCL-95 or between FnLCL-95 and FnUCL-95) includes 5%.

3.1.3 Examples

3.1.3.1 Contaminated Survey Unit

Survey unit = smelter elevated surfaces
31 surface wipe samples with 3 <LOQ
LOQ = 0.015 µg/100 cm^2
Specified limit = 0.2 µg/100 cm^2

Censored: 0 = Yes, 1 = No

Data	Censored	Metrics		
0.015	0	mu	-2.291	
0.015	0	se.mu	0.231	
0.015	0	sigma	1.276	
0.025	1	se.sigma	0.175	
0.025	1	GM	0.101	
0.040	1	GSD	3.582	
0.040	1	EX	0.228	MLE of the arithmetic mean,
0.040	1			Lognormal
0.045	1	LCLc-95	0.134	
0.050	1	UCLc-95	0.390	
0.050	1	KMmean	0.203	Kaplan-Meier estimate of the mean,
0.095	1			Non parametric
0.070	1	KLCL-95	0.125	
0.075	1	KUCL-95	0.281	
0.100	1	Obs%95	0.650	
0.125	1	Est%95	.825	Lognormal
0.125	1	UTLa9595	1.526	Lognormal
0.145	1	z_LR-0.2	0.534	Lognormal
0.145	1	NpUTL9595	NA	Non-parametric
0.150	1	Maximum	1.140	
0.150	1	NonDet%	**9.70**	
0.165	1	n	31	
0.270	1	Rsq	**0.983**	
0.290	1	m	28	
0.345	1	Fex-0.2	29.670	Exceedance fraction, Lognormal
0.395	1	FeLCL-95	**19.470**	
0.395	1	FeUCL-95	41.790	

0.420	1	Fnp-0.2	29.030 Exceedance fraction, Non-parametric
0.495	1	FnLCL-95	16.060
0.840	1	FnUCL-95	45.190
1.140	1		

In this example, 9.7% of samples are <LOQ and the Rsq (0.983) is greater than 0.95, so MLE estimates are preferred. The lower confidence limit of the exceedance fraction is much greater than 5% (19.47%), so no additional sampling is needed to support a decision that the survey unit is contaminated.

3.1.3.2 Borderline Survey Unit

Survey unit = shipping containers for Be components

Specified limit = 0.2 µg/100 cm^2

In a scoping survey, the investigator decides to divide the survey unit into two strata: A, which has been used recently, and B, which has not been used in several years.

Step 1: Initial sample with n = 30 in each stratum.
Results in the first 3 results columns below indicate that A and/or B may not be clean.

Step 2: Additional sample of 30 from each stratum.
Results including the additional samples are in the last three results columns.

IS THIS SURVEY UNIT CLEAN?

| | Initial Sample | | | Add 30 to Each | | |
| | A | B | A+B | A | B | A+B |
	sA30	sB30	sAB60	sA60	sB60	sAB120
mu	-5.045	-4.671	-4.841	-4.653	-5.186	-5.001
se.mu	0.458	0.301	0.264	0.303	0.291	0.224
sigma	1.778	1.290	1.526	1.868	1.401	1.763
se.sigma	0.414	0.276	0.240	0.276	0.258	0.203
GM	0.006	0.009	0.008	0.010	0.006	0.007
GSD	5.917	3.631	4.601	6.478	4.060	5.827
EX	0.031	0.022	0.025	0.055	0.015	0.032
LCLc-95	0.011	0.012	0.015	**0.025**	**0.009**	0.019
UCLc-95	0.093	0.039	0.044	**0.120**	**0.024**	0.052
KMmean	0.028	0.023	0.025	0.051	0.018	0.034
KLCL-95	0.015	0.014	0.018	0.019	0.013	0.018
KUCL-95	0.041	0.032	0.033	0.083	0.023	0.051
Obs%95	0.106	0.051	0.105	0.158	0.047	0.105
Est%95	0.120	0.078	0.097	0.206	0.056	0.122
UTLa9595	0.329	0.160	**0.174**	0.415	**0.097**	0.195
z_OEL-0.2	1.933	2.374	2.117	1.629	2.553	1.924
NpUTL9595	NA	NA	0.161	1.120	0.149	0.195

Maximum	0.161	0.149	0.161		1.120	0.149	1.120
NonDet%	60.000	53.300	56.700		51.700	66.700	59.200
n	30.000	30.000	60.000		60.000	60.000	120.000
Rsq	**0.915**	0.952	**0.959**		**0.965**	**0.956**	**0.974**
m	12.000	14.000	26.000		29.000	20.000	49.000
Fex-0.2	2.660	0.880	1.710		**5.170**	0.530	2.720
FeLCL-95	0.480	0.080	0.470		**2.270**	0.070	1.280
FeUCL-95	10.110	5.580	**5.110**		10.430	**2.750**	5.300
Fnp-0.2	0.000	0.000	0.000		1.670	0.000	0.830
FnLCL-95	0.000	0.000	0.000		0.090	0.000	0.040
FnUCL-95	**9.500**	9.500	4.870		7.660	4.870	3.890

The initial survey produced discrepant results that are hard to interpret. While overall the data appear lognormal (Rsq for the SU = 0.959), Rsq for stratum A (0.915) is less than 0.95. The MLE 95-95 UTL (0.174) supports a conclusion that the survey unit is clean when compared to the specified limit of 0.2 $\mu g/100 \, cm^2$; however, the upper confidence limit of the percent exceedance of the SU (5.110%) is larger than 5%. The qualified individual decides to collect more samples to improve confidence in the estimates.

The additional samples collected in the second survey produced results that are easier to interpret. The data are more clearly lognormal (Rsq's = 0.965, 0.956, 0.974), so the MLE estimates are preferred. The confidence intervals (CI) for the MLE arithmetic means do not overlap (stratum A CI 0.025-0.120, stratum B CI 0.009-0.024), indicating that the strata should be separated into two survey units. Both the MLE 95-95 UTL (0.097) and the upper confidence limit of the percent exceedance (2.750%) support a conclusion that survey unit B is clean. These data can be used in the final status survey to document that survey unit B containers comply with the specified limit. For survey unit A, the most likely estimate of percent exceedance (5.170%) is above 5%, indicating that it is more likely than not contaminated. However, the lower confidence limit (2.270%) is below 5%, so it is possible that additional sampling will be able to demonstrate that survey unit A is clean. The qualified individual will have to decide whether it is more cost effective to collect additional samples or begin remediation.

3.2 Beryllium Surface Contamination Surveys

The decision to conduct surveys can be triggered by review of historical records or other information indicating that beryllium surface contamination is possible. The first steps in the planning process are to define the objectives of the survey, the surfaces that will be included, and the specified limits used to interpret data. The objective of the survey can be categorized as scoping, characterization, remedial action support, or final status. Figure 3.1 provides an overview of the strategy that will be described in this section.[3]

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[3] The terminology, concepts and figure used in this section are adapted from the *Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM),* which is posted on the web at http://www.epa.gov/radiation/marssim/index.html.
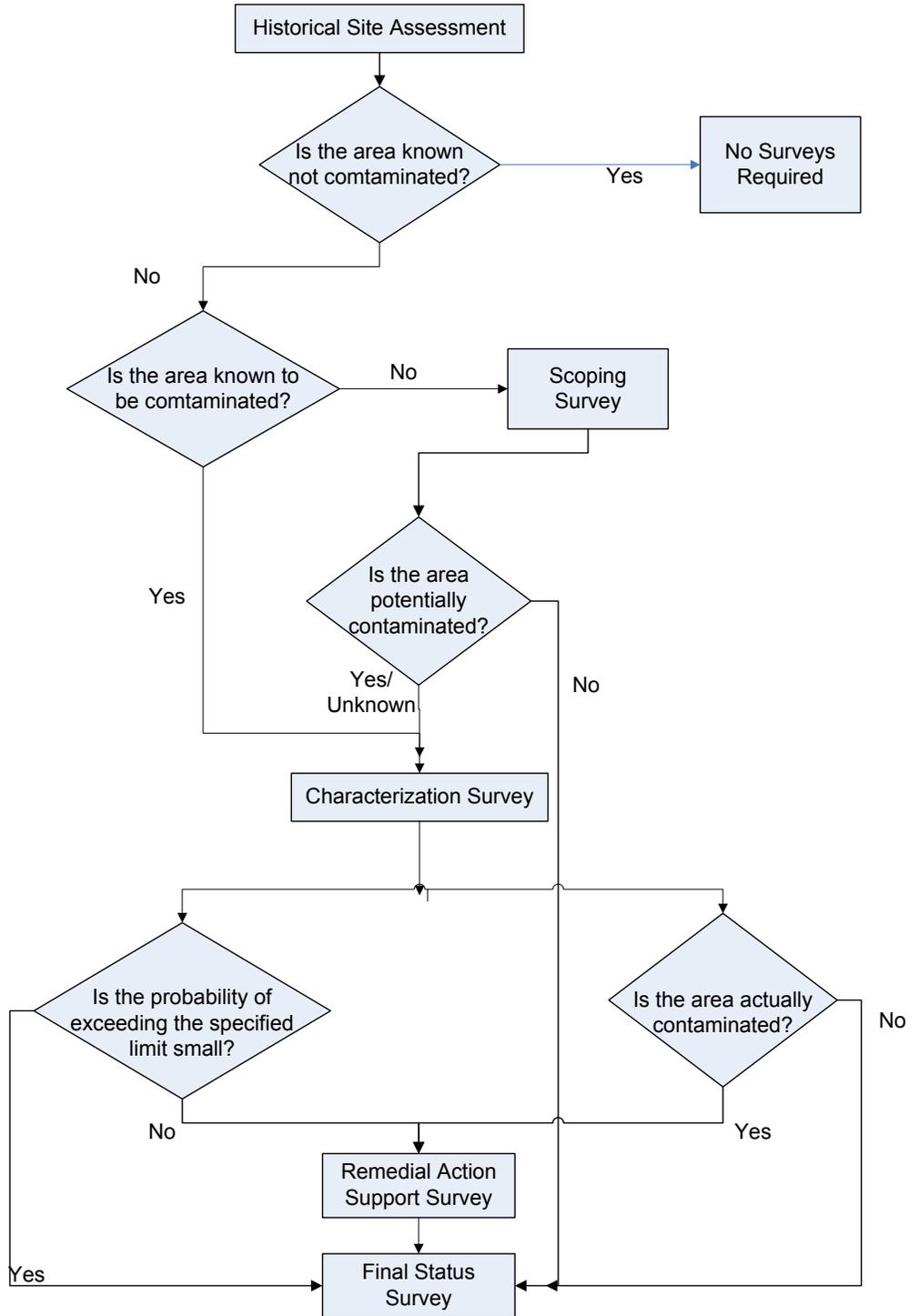
3.2.1 Survey Unit

A survey unit is the area or population of articles from which statistically planned samples are being drawn. The survey strategies described in this section are used for making decisions about large survey units, typically on the order of 100 m^2 or larger, or more than 100 articles. A survey unit should contain surfaces thought to have similar probabilities of being contaminated. They would be a building, rooms in a building, or equipment that share a common history of beryllium use or other characteristics thought to determine contamination levels. Only a small percentage of the available surface area will be sampled to make decisions about all the surfaces in the survey unit. The survey unit should not be so large that unintentional misclassification occurs by combining areas or articles with different uses or histories of use. There are no definite upper limits on the size of a survey unit. Survey units larger than 1000 m^2 or containing more than 1000 articles would be unusual if the unit had undergone remediation.

The person selecting survey units obtains information on the potential for contamination through a historical site assessment using techniques described in DOE's implementation guide for its beryllium rule.[4] If no previous survey or other quantitative information is available, the person will have to make initial selections of survey units based on judgment and qualitative information and then make adjustments as survey data develop.

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[4] DOE G 440.1-7A, *Implementation Guide for Use with 10 CFR Part 850, Chronic Beryllium Disease Prevention Program*, http://www.eh.doe.gov/be/policy/g4401-7.pdf

Figure 3.1 The Beryllium Surface Contamination Survey Process

3.2.2 Scoping Survey

A scoping survey provides initial quantitative data on contamination when no other reliable data exist. If the survey unit is known to be contaminated or if there are records indicating the survey unit is not contaminated, a scoping survey is not needed. The objectives of a scoping survey are twofold: to determine if contamination is present and to provide information on the characteristics of contaminated surfaces.

A stratified random sampling strategy can accomplish both objectives efficiently. The strata share characteristics that are hypothesized to be potential determinants of beryllium contamination and are based on qualitative information or judgment. Examples of strata might be: routinely cleaned surfaces, infrequently cleaned surfaces, process equipment, ventilation systems, and surfaces near reported locations of historical beryllium use. A stratified random sampling plan ensures that at least some samples come from each stratum. The strata should be mutually exclusive: every surface must be assigned to only one stratum. The strata should also be collectively exhaustive; no surface can be excluded. Surfaces selected for sampling within each stratum should not be intentionally biased towards best or worst case. Random selection is the usual method of ensuring that neither intentional nor unintentional bias is introduced.

If more than a few (5 or 6) strata can be hypothesized, the value of a stratified random strategy is diminished. In this circumstance, the strategy would change to total random sampling.

Sample locations should be recorded so that results above specified limits or other unusual results can be investigated.

Surface wipe sampling from uncontaminated space is expected to produce results mostly or completely less than the limit of quantitation (<LOQ), and the data are most often interpreted by comparing results to a specified limit. This leads to the selection of distribution-free 95% upper tolerance limit of the 95th percentile (95-95 UTL) since it is a statistic that can be determined even if all samples are <LOQ. Achieving distribution-free 95-95 UTL requires collection of a minimum of 59 samples. Appendix VIII of the American Industrial Hygiene Association (AIHA) *A Strategy for Assessing and Managing Occupational Exposures*[5] provides a discussion of this method. If more than 30% of data are >LOQ and appear to be lognormally distributed, it is possible to estimate geometric 95-95 UTL or percent exceedance and confidence interval. Appendix VII of the AIHA *Strategy*[3] discusses methods for calculating these statistics. A computerized method is discussed in section 3.1 above.

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[5] Mulhausen, JR and Damiano, J, *A Strategy for Assessing and Managing Occupational Exposures,* Second Edition , AIHA Press, Fairfax, VA, 1998.

Initial plans should include collecting a minimum of 59 samples from each survey unit. One possible outcome of a scoping survey is that no contamination above specified limits is found. In this case, the scoping survey can also be the final status survey, since this is sufficient evidence to support a conclusion that the survey unit is not contaminated. A finding of 59 samples below the limit also supports a conclusion that the characteristics used to define a stratum as suspicious were not determinants of beryllium contamination after all. Rather, it supports the null-hypothesis that all surfaces in the survey unit have an equally low probability of being contaminated.

The other possible outcome of the scoping survey is that one or more sample results will be above the limit. This finding would require a change in strategies, usually the initiation of a characterization survey, since CBDPPs require that surface levels do not exceed specified limits. Because only one sample above the limit will require an adjustment in plans, investigators may wish to collect the random samples in the order they judge to be from most to least likely above the limit, which may reduce the number of scoping samples needed. The decision to collect samples sequentially or all in one campaign should be based on cost and schedule.

3.2.3 Characterization Survey

Any sample result indicating contamination above limits usually requires additional investigation with a characterization survey. The objectives of a characterization survey are to determine the nature and extent of contamination so that remedial actions can be planned. This is diagnostic rather than random sampling. Once investigators have determined the characteristics of contaminated surfaces, survey units would be adjusted and planning can begin for surveys of contaminated surfaces undergoing remedial actions and final status surveys for the surfaces that are no longer contaminated.

Bulk sampling of settled dust will allow for analysis of the part-per-million (ppm) content of beryllium. Differences in the concentrations of beryllium on different surfaces support conclusions about different causes or sources of contamination. A conclusion that the two means are different is supported if their confidence intervals do not overlap. Appendix VI of the AIHA book discusses methods for calculating means and confidence intervals. Computerized methods for calculating the mean and 90% confidence interval are discussed in section 3.1 above. Also, the ratio of beryllium mass to the mass of other metals in the sample can be used to characterize likely sources of contamination.

Showing that the concentration of beryllium in bulk samples of surface dust from a survey unit is the same as in a background reference area requires a slightly different approach. A finding that that mean beryllium concentration of settled dust in the survey unit is less than or equal to the concentration in a reference area supports a conclusion that contamination is due to background. One must take

enough samples to minimize the chance of falsely concluding that contamination is equal to background because of overly large confidence intervals. This is called a type I error.

The chance of making a type I error can be minimized through the use of a derived limit. The derived limit specifies the amount that the upper confidence interval of the survey unit can exceed the lower confidence limit of the mean of the reference area. In this Technical Standard, 1 ppm has been selected as the value to be added to the 95% lower confidence limit of the reference area to establish the derived limit.

An alternative approach is to use a default limit. The Agency for Toxic Substances and Disease Registry (ATSDR) Toxicological Profile for Beryllium reports that the mean concentration of beryllium in soil is 0.6 ppm (see section 6 page 151 http://www.atsdr.cdc.gov/toxprofiles/tp4-c6.pdf.) In this Technical Standard, 0.6 ppm has been selected as the default limit. If the 95% upper confidence of the mean concentration in the survey unit is less than 0.6 ppm, then sampling in a reference area to establish a derived limit is not required.

3.2.3.1 Examples

In the following examples all data and metrics are ppm. The Kaplan-Meier (KM) estimates of the mean and 95% lower (KLCL_95) and upper (KUCL_95) confidence limits were calculated using the R software and routines described in section 3.1.2 above. The abbreviations for the metrics are defined in section 3.1.1.

	Reference Area	Survey Unit 1	Survey Unit 2	Survey Unit 3
Value 1	0.29	0.25	1.60	0.63
Value 2	0.32	0.6	0.89	0.36
Value 3	0.14	0.34	1.10	0.13
Value 4	0.32	0.17	3.30	**2.47**
Value 5	1.54	0.28	4.90	0.21
Value 6	0.63	0.64	0.68	0.66
Value 7	2.06	<0.09	1.00	0.59
Value 8	1.11	<0.09	2.80	0.57
Value 9	0.81	0.47	0.45	0.55
Value 10	0.57	0.29	4.70	0.23
Value 11	0.73	0.33	3.60	0.22
Value 12	0.45	0.26	1.50	0.96
Value 13	0.18	<0.09	3.10	0.21
Value 14	0.26	0.22	3.00	0.77
Value 15	0.36	0.33	4.70	1.04
Metrics				
EX	0.65	0.31	2.59	0.64
LCLc_95	0.44	0.21	1.76	0.43
UCLc_95	0.95	0.46	3.80	0.93
KMmean	0.65	0.30	2.49	0.64
KLCL_95	**0.40**	0.22	**1.78**	0.38
KUCL_95	**0.90**	**0.38**	**3.20**	**0.90**
Rsq	0.98	0.86	0.92	0.95

3.2.3.2 Interpretation

The derived limit at this site is the KLCL_95 value for the reference area plus 1 ppm, which in this case is 0.40 ppm + 1 ppm = 1.40 ppm.

Survey unit 1 is an example of data supporting a conclusion that the beryllium contamination found is due to naturally occurring background dust. The KUCL_95 of 0.38 ppm is less than the derived limit of 1.40 ppm and the default limit of 0.6 ppm. A finding of values well below background is not uncommon since the source of settled dust inside a facility is not just soils infiltrating or tracked into the building from outside but will also include dust generated in the building. If samples from this survey unit had been collected and analyzed first, no sampling of a reference area would have been required.

Survey unit 2 is an example of data supporting the conclusion that the beryllium contamination found is due to an industrial process rather than background. The KUCL_95 of 3.2 ppm is greater than the derived limit of 1.40 ppm. In fact, the KLCL_95 of 1.78 ppm is greater than 0.90 ppm, the KUCL_95 of the reference

area, providing a high level of confidence that this survey unit is different from background.

Survey unit 3 is an example of data where the confidence intervals overlap those of the reference area. This is the situation where the derived limit plays its role of assuring enough samples have been collected to minimize the chance of making a type I error. The KUCL_95 value of 0.90 ppm is less than the derived limit of 1.40 ppm. The statistical analysis supports a conclusion that contamination is due to naturally occurring background dust. However, value 4 of 2.47 ppm is well above the derived limit, raising suspicion that there might be a cause other than chance. The characteristics of the location of this sample should be investigated to determine if they suggest a plausible cause. If a plausible cause can be identified, judgmental samples from locations with similar characteristics should be collected to see if they are also elevated. If the excursion can't be repeated and explained, one would conclude that it was a chance occurrence.

3.2.3.3 Sample Size

In the examples, a sample size of 15 was sufficient to reach confident conclusions. Beryllium concentrations in the data sets from reference areas and clean survey units that we have seen tend to be less variable then data sets from survey units where industrial contamination is present. This is a recommended sample size for initial planning of survey costs and schedules. If schedule and ready access to the site allow, one might start with as few as 6 samples from each reference area and survey unit and collect additional samples if needed.

3.2.4 Remedial Action Support Survey

If a survey unit is determined to be contaminated above specified limits, the unit can be managed as contaminated or a remedial action plan can be prepared. If remediation is chosen, a remedial action support survey is performed while remediation is being conducted, and it guides the cleanup in a real-time mode. Remedial action support surveys are conducted to:
- support remediation activities and
- determine when a site or survey unit is ready for the final status survey.

3.2.5 Final Status Survey

The final status survey provides data to demonstrate that survey units satisfy the specified limits. Although the final status survey is discussed as if it were an activity performed at a single stage of the site investigation process, this does not have to be the case. Data from the scoping or other statistically planned surveys conducted during the site investigation process can be used, provided that they are of sufficient quality.

The final status survey utilizes an unbiased approach to achieve representative sampling consistent with the application of statistical tests. Each survey unit is considered to be homogeneous, with all surfaces having an equal chance of being contaminated. The initial hypothesis is either that the survey unit has never been contaminated or that all contamination has been cleaned to levels below a specified limit. The usual method of minimizing the chance for introducing bias is random selection of sampling locations.

The 95-95 UTL is a commonly used criterion for determining the number of samples needed to demonstrate compliance with exposure limits. It is intended to minimize the probability of contaminated surfaces erroneously appearing to be clean. The 95-95 UTL was first suggested by the National Institute for Occupational Safety and Health (NIOSH)[6] as the criterion for use of even a single monitoring result near a permissible exposure limit to justify a conclusion that exposure monitoring and control programs were needed. This recommendation was adopted by OSHA as the basis for action levels in expanded standards. Subsequently, Tuggle[7] suggested that this criterion be used by employers to demonstrate compliance with exposure limits. The authors of the NIOSH publication endorsed this concept in the 3rd edition of the textbook *Patty's Industrial Hygiene and Toxicology*[8].

3.2.6 Small Areas or Single Article

If the surface area of the survey unit is small or if only a small number of articles are being surveyed, the collecting and analyzing the number of samples required for statistically planned sampling is not feasible. One strategy involves sampling either the entire or a significant percentage of the available surface with a single sample or with a few samples. The concept is similar to compositing samples, which is used to reduce analytical costs. Compositing usually involves mixing several grab samples for a single analysis rather than analyzing each separately and taking an average. For beryllium contamination surveys, the compositing would occur at the sample collection stage. A single wipe sample might be collected from the entire surface of a small article or from several areas of a larger article (larger article greater than 400 cm^2). For articles with areas less than 100 cm^2, the single result is compared directly to the specified limit. For a larger article (greater than 400 cm^2), two or more samples may be preferable to provide information on the location of contamination (e.g., interior and exterior, top and bottom, or front and back surfaces). For these larger articles, a goal might be to include at least 10% of the available surfaces in the wipe samples.

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[6] Leidel, N.A., K.A. Busch, and W.E. Crouse: *Exposure Measurement Action Level and Occupational Environmental Variability* (DHHS [NIOSH] 76–131). Cincinnati, Ohio: National Institute for Occupational Safety and Health, 1975.

[7] R.M. Tuggle: "Assessment of occupational exposure using one-sided tolerance limits." *AIHAJ,* Vol. 43, pp 338 – 341, 1982.

[8] Leidel, N.A., and K.A. Busch: "Statistical design and data analysis requirements." In R.L. Harris, L. J. Cralley, and L.V. Cralley, editors, *Patty's Industrial Hygiene and Toxicology,* 3rd ed., vol. III, part A, pp. 453–583. New York: John Wiley & Sons, 1994.

The analysis result of a single composite sample that has been used to wipe an entire area larger than 100 cm^2 is equal to the mean of all the possible 100-cm^2 grab samples that could have been taken on that area. A drawback to a single composite sample is that the single result does not provide the data that is needed to calculate a 95-95 UTL for comparison to specified limit. An approach that can be used if the area included in the wipe is less than 400 cm^2 is to assume that all the contamination on the wipe came from a single 100-cm^2 grab sample and the single result is compared directly to the specified limit. That would be a worst case scenario; the entire surface could be declared to be below the specified limit if the presumed worst case 100-cm^2 is below the limit. An approach that can be used for areas that are larger than 400 cm^2 is to use the mathematical relationship between the mean and 95th percentile in a lognormal distribution in which the 95th percentile cannot exceed the mean by more than a factor of 4.[9] Therefore, log normally distributed results of composite samples that are converted to µg/100 cm^2 in which the mean is less than 4 times the specified limit support a conclusion that there is a <5% chance of any single 100-cm^2 surface area being above the specified limit. For example, if a single wipe sample was collected from a 1 square meter (m^2) surface it would be a composite of 100, 100-cm^2 surface samples. Analytical results indicating that less than 5 µg of beryllium was collected on the composite sample would be evidence that none of the 100-cm^2 surfaces is above 0.2 µg/100 cm^2. A mathematical expression for this is:

$$(< 5 \text{ µg/m}^2)/100 < 0.05 \text{ µg/100 cm}^2 < (0.2/4) \text{ µg/100 cm}^2$$

3.3 Conducting the Baseline Inventory

Title 10 CFR 850.20(b)(1-4), "Baseline beryllium inventory," specifies four actions that can be taken in conducting the baseline inventory. Any or all of the four actions may be appropriate to a situation; all four actions are not necessarially needed. For example, bulk sampling and worker interviews are not necessary if monitoring data exist documenting routine airborne release of beryllium.

3.4 Decision Logic for Area Assessments

Figure 3.2 provides the decision logic for determining which areas should be included in the beryllium inventory.

3.5 Identification of Natural Sources of Beryllium

The most common analytical techniques for determining the beryllium content of a sample begin with digesting all the beryllium into ions. These techniques do not distinguish the form that the beryllium was in before the digestion step. Qualified individuals can make the determination that beryllium in and around a shop or process

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[9] Rappaport, SM. "Assessment of long-term exposures to toxic substances in air." *Ann. Occup. Hyg.* 1991 Feb;35(1):61-121.

area is in a metal, oxide, or alloy form based on process knowledge and matching the composition of the sample with the composition of the original material. An analytical technique used with some success at DOE sites to determine the levels of beryllium in background soil is based on matching the ratios of atoms of beryllium to the atoms of a major constituent (e.g., iron, aluminum, or manganese) of the soil. Various other analytical techniques are available to determine if beryllium is in a naturally occurring form. The methods used to determine the form of beryllium should be documented and technically defensible, particularly when the site determines that the source is (1) background soil, in which case the Rule does not apply, or (2) another natural form of beryllium, which may be excluded from consideration under the Rule as a result of an OGC interpretation or future rulemaking.

3.6 Decision Logic for Air Sampling

Figure 3.3 gives the decision logic for air sampling.

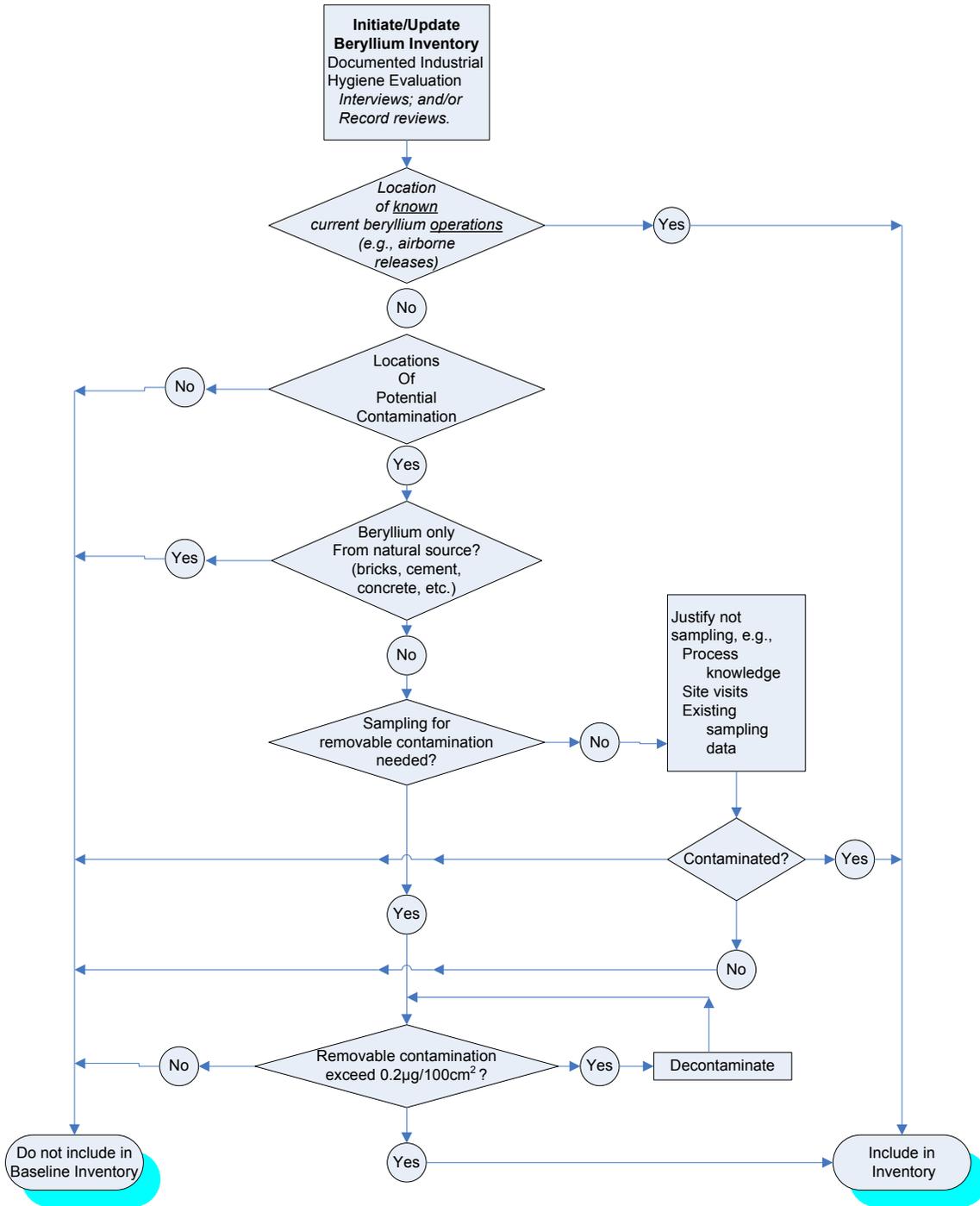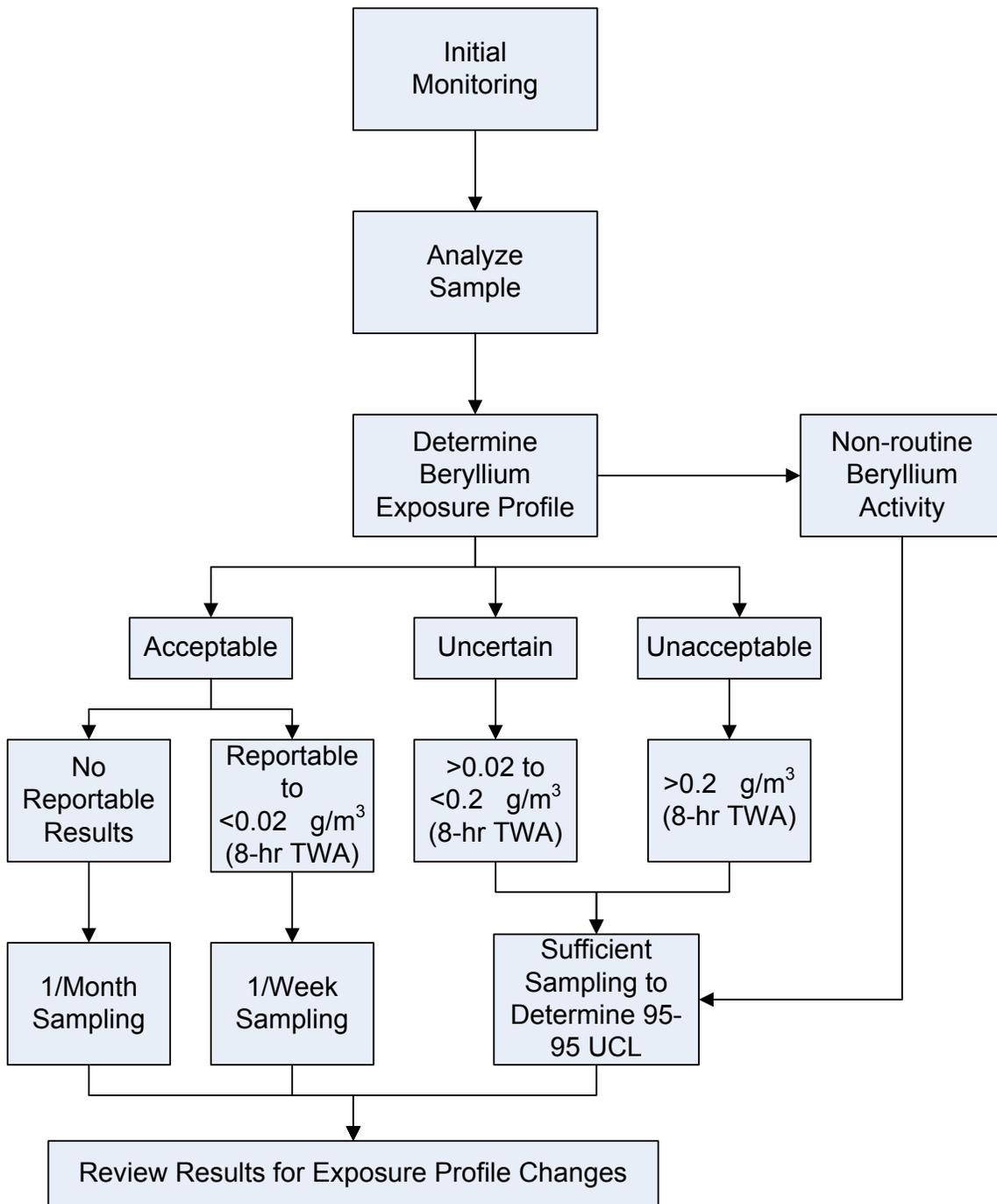**Figure 3.2. Determining Items and Areas for Beryllium Inventory**

Figure 3.3. Decision Logic for Determining Air Sampling Frequency

Initial Monitoring

↓

Analyze Sample

↓

Determine Beryllium Exposure Profile → Non-routine Beryllium Activity

Acceptable | Uncertain | Unacceptable

No Reportable Results | Reportable to <0.02 μg/m³ (8-hr TWA) | >0.02 to <0.2 μg/m³ (8-hr TWA) | >0.2 μg/m³ (8-hr TWA)

1/Month Sampling | 1/Week Sampling | Sufficient Sampling to Determine 95-95 UCL

Review Results for Exposure Profile Changes

3.7 Sampling Methods

3.7.1 Purpose of Sampling

Title 10 CFR 850 requires air, surface, and bulk sampling when necessary to implement various Rule provisions. Air sampling is required when necessary to conduct the baseline beryllium inventory (10 CFR 850.20), to determine compliance with the permissible exposure limit (10 CFR 850.22), to determine if the action level has been exceeded, and to conduct exposure monitoring (10 CFR 850.24). Surface sampling is required when necessary to conduct the baseline beryllium inventory (10 CFR 850.20), to determine compliance with the housekeeping (10 CFR 850.30) limit for operational areas, and to meet the cleanliness release criteria (10 CFR 850.31) for releasing beryllium-contaminated equipment and other items for use by others. Bulk sampling is required by the Rule's release criteria (10 CFR 850.31) provision if a site wishes to demonstrate that surface contamination levels do not exceed the levels of beryllium in the surrounding soil.

Title 10 CFR 850 is silent in both the Rule and its preamble about the air and bulk sampling methods that should be used. The preamble, however, states, "To reduce the variability in reported surface contamination across the DOE complex, DOE recommends, but does not require, the use of a single sampling method: NIOSH Method 9100, *NIOSH Manual of Analytical Methods,* 4th Edition, August 15, 1994, 'Lead in Surface Wipe Samples'."

3.7.2 Air Sampling

Air sampling should be performed using the sampling (not the analytic) components of NIOSH Method 7300, *NIOSH Manual of Analytical Methods,* "Elements by ICP (Nitric/Perchloric Acid Ashing)," http://www.cdc.gov/niosh/nmam/pdfs/7300.pdf. See section 3.8 of this Technical Standard for analytical methods.

3.7.3 Surface Sampling

Obtaining precise and accurate results with surface sampling is problematic due to the many variables inherent in the surfaces, the material being sampled, the sampling media, and the sampling technique. Nonetheless, an objective of this technical standard is to maximize the precision and accuracy of results and the consistency of results throughout DOE.

Three categories of methods are available for surface sampling. The three methods are generally referred to as wet, in which a surface is wiped with wet sampling media; dry, in which a surface is wiped with dry sampling media; and vacuuming, in which the surface is vacuumed and the sample is collected on a dry filter in the vacuum system.

Wipe surface sampling for beryllium should be performed only on relatively clean surfaces. Most of the material on heavily loaded surfaces (e.g., iron, aluminum, titanium) is likely to interfere with the analysis of the comparatively small amount of beryllium. If beryllium particles are included in the heavily loaded surface, the beryllium often will overwhelm the range of the analytic equipment, which is calibrated for small amounts of beryllium. Heavily loaded surfaces should be sampled by bulk sampling (see Sect. 3.7.4).

Wet wipe sampling is the method that should be used unless wetting the surface would change the properties of the wiped surface in a manner that would render the surface unacceptable for its intended use. The technical literature does not provide peer-reviewed studies comparing the recoveries of beryllium from surfaces sampled by wet versus dry media, but the consensus of DOE subject matter experts at this time is that sufficient experience with both methods supports the conclusion that wet wipe media recover more beryllium from surfaces than do dry wipe media. In addition, standardizing on one method whenever possible will help meet the objective of improving comparability of results throughout DOE. DOE expresses the same objective in the Rule's preamble: *In the long term, by recommending a single method (a wet method) for conducting the surface sampling, DOE believes that the variability associated with surface sampling will be reduced without specifying a particular method in the rule*.

3.7.3.1 Wet Wipe Sampling

The wet wipe sampling method that should be used is American Society for Testing and Materials (ASTM) D6966*, Standard Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Determination of Metals.* That method uses wet wipes such as those specified by ASTM E1792, *Standard Specification for Wipe Sampling Materials for Lead in Surface Dust.* The wetting agent should be de-ionized water, which is more appropriate than solvents or detergents for wiping to determine the "removable contamination level" as that term is used in the Rule.

3.7.3.2 Dry Wipe Sampling

Certain surfaces must not be exposed to any wetting agents to ensure that the characteristics of the item do not change as a result and other reasons may arise that preclude the use of wet wipes. Dry wipe sampling is acceptable in those situations. Appendix 3-1 provides the dry wipe sampling method that should be used.

3.7.3.3 Equivalency Factors

Some DOE sites have accumulated many years' worth of surface contamination data obtained using dry wipes or wipes wetted with different wetting agents.

Conducting comparison studies could allow these sites to establish equivalency factors to convert their historical results to results that would have been obtained had they used de-ionized water.

It will be technically challenging to conduct credible comparison studies. The studies would have to simulate historical surface conditions, apply amounts of beryllium in the ranges of interest, adequately control other confounding variables, and be reviewed for adequacy by competent persons who are not affiliated with the site. The data sets being compared should have a correlation coefficient of at least 0.60. A specific protocol for conducting a comparison study is not available, but a useful model can be found in Kerr's *Sampling Beryllium Surface Contamination Using Wet, Dry, and Alcohol Wipe Methods.*[10] Applying equivalency factors should only be used for converting historical data and should not justify the continued use of wipes that are dry or not wetted with de-ionized water. Also, the equivalent values should be identified as such and their derivation explained to recipients of this data to prevent the equivalent values being mistaken for the actual values.

3.7.3.4 Vacuum Surface Sampling

Vacuum surface sampling is accomplished with a portable vacuum that has a dust collection cassette just downstream of the vacuum nozzle. It has been used in studies,[11] and a method, *Standard Practice for Collecting Surface Dust by Microvacuum Sampling for Subsequent Metals Determination,* is in ASTM Committee D22 balloting. It promises to have advantages over both wet and dry sampling for some situations because it:
- is likely to have superior ability to recover beryllium from irregular and textured surfaces,
- is likely to be able to recover material from heavily loaded surfaces in sufficient amounts to allow determination of both surface contamination and surface loading, and
- may improve the beryllium recovery and consistency of sample results from relatively clean surfaces.

Vacuum surface sampling should be used only in unique situations until the ASTM Standard Practice is available.

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[10] Kerr, Kent, *Sampling Beryllium Surface Contamination Using Wet, Dry, and Alcohol Wipe Methods*, Thesis, Master of Science in Industrial Hygiene, Department of Safety Sciences, Central Missouri State University (December 2004). Accessible at http://www.osti.gov/bridge/servlets/purl/837587-M4P95G/native/837587.pdf

[11] Clark, C.S., W. Menrath et al.: "The Influence of Exterior Dust and Soil Lead on Interior Dust Lead Levels in Housing That Had Undergone Lead-based Paint Hazard Control," *J. Occup. Environ. Hyg.*, 1: 273-282 (2004)

3.7.4 Bulk Sampling

A generalized bulk sampling method is provided in Appendix 3-2.

3.8 Analytical Methods

3.8.1 Purpose of Analysis and Data Quality Objectives

3.8.1.1 Typical Problem for Which This Technical Standard Was Developed

Lessons Learned 2003-SR-WSRC-0013 was generated as a result of discrepancies in analytical results from two AIHA-accredited laboratories (a site lab and a contract lab), both of which were using the NIOSH 7300 protocol or a modified version thereof. The discrepancies resulted from matrix and spectral interferences experienced by the offsite laboratory. Different analytical lines were used by the two labs, and potentially interfering species (Cu and Ni) were present. Also, BeO could not be accounted for by either lab. Recommended actions are provided in this Technical Standard.

3.8.1.2 Summary Guidance

Samples that are taken for compliance with 10 CFR 850 must meet the Data Quality Objectives (DQO) prescribed by 10 CFR 850.24(e) (accuracy of $\pm 25\%$ with a confidence level of 95%). For samples that may not need the stringent 10 CFR 850 DQO, consideration may be given to less costly analytical methods [e.g., flame atomic absorption (AA) rather than inductively coupled plasma with electron spectrometry (ICP-ES), see below for discussion of methods]. The consideration should include the desired turnaround time, sample load demand, throughput capacity, data quality objectives (sensitivity, etc.), other analytes of interest or interferences, and specificity.

3.8.1.3 Compliance Samples

The following are examples of purposes for which samples are taken to demonstrate compliance with 10 CFR 850:
- Compliance with housekeeping limits
- Selecting personal protective equipment (PPE)
- Evaluating personal exposures
- Monitoring the efficacy of contamination control using both air and surface sampling
- Compliance with release limits for equipment or other items

Analysis of these samples should meet the DQO prescribed by 10 CFR 850. DOE sites should ensure that laboratories (both on-site and off-site) performing analyses for compliance with 10 CFR 850 demonstrate performance consistent with the guidance in this Technical Standard.

3.8.1.4 Purposes Not Related to 10 CFR 850 Compliance

The following are examples of purposes for which samples are taken that are not necessarily related to compliance with 10 CFR 850:
- Diagnosing points of emissions from process equipment
- Clearing areas for various types of occupancy
- Identifying and quantifying beryllium exposure sources
- Demonstrating environmental compliance
- Maintaining good public relations
- Maintaining good community relations by screening personal vehicles, homes, etc.
- Analyzing bulk samples to determine contribution of background beryllium to surface loading levels.

Analysis of these samples may not necessarily need to meet the DQO prescribed by 10 CFR 850. An appropriate DQO should be defined and provided to the analytical laboratory. Depending on the DQO, it may be possible to use alternative, less costly analysis methods and/or use a non-accredited laboratory.

3.8.2 Standard Methods

3.8.2.1 Summary Guidance

Currently, DOE sites and contract labs are using methods derived from a variety of published sources, including (but not necessarily limited to) the following sources:
- Environmental Protection Agency (EPA) Manuals (SW-846 and/or Contract Laboratory Program)
- NIOSH Manual of Analytical Methods (NMAM)
- OSHA Sampling and Analytical Methods
- ASTM consensus standards
- International Standards Organization (ISO) consensus standards

A committee with representation from DOE sites, NIOSH, OSHA, the Department of Defense (DOD), and other interested parties has begun developing a set of ASTM standard methods for sampling and analysis of beryllium under the auspices of ASTM International Subcommittee D22.04 on Workplace Atmospheres. Use of these methods, as they are developed and issued, is strongly encouraged. Users of alternative methods should demonstrate and document that the alternative methods provide a level of performance that is equivalent or superior to the standard methods with respect to parameters including DQO, precision, bias, method detection limit (see Sect. 3.8.2.5), and limit of quantitation (see Sect. 3.8.2.5). (Reference: EPA SW-846, Chapter 1, Quality Control.)

3.8.2.2 Sample Preparation (Digestion)

The following guidance is provided pending development of an ASTM standard method for beryllium sample preparation.

Heat/Energy Source

Acceptable heat/energy sources include hot plates, hot blocks, and microwave ovens. Hot plates and hot blocks should be used in fume hoods with sufficient airflow. Those using microwave ovens should consider digestion vessels compatible with the digestion matrix, overpressurization protection, and safety interlocks to prevent excessively high temperatures.

Digestion Reagents

It is essential that the selected digestion protocol be capable of digesting the desired forms of beryllium that will be encountered in the sample matrix. It usually is desired to digest all the beryllium in the sample, but it may be possible to speciate specific forms of beryllium by selecting appropriate digestion reagents. The digestion protocol should also adequately digest the sample matrix (wipe or filter) to minimize the risk of matrix effects during the analysis.

Of particular concern is digestion of beryllium oxide (BeO). BeO is readily formed on airborne beryllium particles and on beryllium metal objects, so all beryllium samples will include beryllium oxide. This form of BeO is relatively easy to digest. Also, "low-fired" manufactured BeO is relatively easy to digest but is rarely, if ever, manufactured. Of greater concern is "high-fired" BeO, which is manufactured today and is more difficult to digest because it has a lower surface-to-mass ratio. Note, also, that a digestion protocol suitable for beryllium metal, oxide, and alloys may not necessarily digest naturally occurring forms of beryllium (such as silicates and aluminates) that may be found in soil samples. Conversely, digestion protocols used for silicates may not digest BeO. Consideration should be given to the species of beryllium that needs to be analyzed, to ensure that the correct analytical methods are employed. As mentioned above, organizations currently are developing standard methods that will include digestion reagents for different beryllium species.

Experience to date suggests that reliable digestion of BeO requires the use of sulfuric acid and/or hydrofluoric acid. Other reagents, such as ammonium bifluoride, are being tested, but definitive data are not yet available. It should be noted that this experience is based on BeO as encountered in the field, not on a BeO reference material (see Sect. 3.8.3.2 for status of BeO reference material).

Laboratories should perform recovery studies that demonstrate acceptable performance of the selected digestion method(s). Such studies may still be

somewhat qualitative until necessary standard reference materials are available (see Sect. 3.8.3.2).

3.8.2.3 Instrumentation

Analytical instruments routinely used for compliance with 10 CFR 850 include inductively coupled plasma emission spectrometers (ICP-ES), inductively coupled plasma mass spectrometers (ICP-MS), and graphite furnace atomic absorption (GFAA). For samples that do not support compliance with 10 CFR 850 (see Sect. 3.8.1.3 above), instruments such as flame atomic absorption (FLAA) may also be appropriate.

- ICP-ES is currently the most common instrument used for beryllium samples because of its ability to meet the required DQO, relative affordability, and versatility for other analytical work.
- ICP-MS generally offers a lower detection limit but is more expensive to purchase and operate, and it can be subject to problematic matrix interferences.
- GFAA provides results that are comparable to ICP-ES but typically does not provide the same throughput capacity.
- FLAA is less expensive but typically does not meet the DQO for 10 CFR 850 compliance because its Reporting Limit (see sect. 3.8.2.5) is too high.

3.8.2.4 Standard Analysis Methods

The following are ASTM standard analysis methods that are recommended for beryllium analysis. Alternative methods, if used, should demonstrate a level of performance that is equivalent or superior to the standard methods.

Inductively Coupled Plasma-Emission Spectrometry (ICP-ES)

The recommended method is the ASTM method D7035, *Standard Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry*.

Special considerations that apply to ICP-ES analysis include:
- Laboratories should evaluate and properly account for spectral interferences from concomitant species. The evaluation should be documented, and appropriate interference correction actions (if any) should be incorporated in lab procedures. This is more of an issue for wet wipes than for dry wipes or air filters because wet wipes generally collect more material (both beryllium and concomitants). Additional measures may be required for radiologically-contaminated samples since both uranium and plutonium can also interfere with beryllium emission lines. ICP-ES instruments with high-resolution capabilities may be less subject to spectral interferences.

- Care should be given as to selection of appropriate emission lines used for the analysis. Use of more than one emission line will typically help ensure that there are no interferences, or that appropriate interference corrections are applied when necessary. However, only one value (typically from the most sensitive emission line) should be reported. Emission lines typically used include 313.042 nm, 313.107 nm, and 234.861 nm.

Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

The recommended method is ASTM D5673, *Standard Test Method for Elements in Water by Inductively Coupled Plasma – Mass Spectrometry.* Dilution factors cited in D5673 may need to be adjusted based on what is optimal for a given ICP-MS instrument.

Special considerations that apply to ICP-MS analysis:
- Laboratories should evaluate and properly account for isobaric interferences. The evaluation should be documented, and appropriate interference correction actions (if any) should be incorporated in lab procedures.
- ICP-MS is more susceptible to matrix interferences than ICP-ES. Laboratories should evaluate and properly account for such interferences. The evaluation should be documented. Some wipes that comply with ASTM D6966, *Standard Practice for Collection of Settled Dust Samples Using Wipe Methods for Subsequent Determination of Metals*, using wipes such as those specified in ASTM E1792, have been found to have background levels of metals (such as aluminum and arsenic) and binding agents that may constitute interferents.

Graphite Furnace Atomic Absorption (GFAA)

A specific method is not recommended at this time (see Sect. 3.8.2.1). The following special considerations apply:
- GFAA is susceptible to matrix interferences. Laboratories should evaluate and properly account for such interferences. The evaluation should be documented.
- Appropriate background correction techniques should be incorporated in lab procedures.

Fluorescence

A specific method is not recommended at this time (see Sect. 3.8.2.1). The following special consideration applies:
- Fluorescence is susceptible to quenching effects due to temperature, high dissolved oxygen, or impurities. Laboratories should evaluate and properly account for such interferences. The evaluation should be documented.

3.8.2.5 Reporting Limits

The reporting limit (RL) is "the lowest concentration of analyte in a sample that can be reported with a defined, reproducible level of certainty" (*AIHA Laboratory Quality Assurance Policy Document,* 1/1/04). It is the lowest numerical value that a laboratory will report to the customer without flags or qualifiers.

Acceptable reporting limits

Laboratory reporting limits should be no higher than 0.05 μg per wipe or per cubic meter (one-half the 10 CFR 850 limits); this is considered minimally acceptable. A reporting limit of 0.02 μg (or lower) per wipe or per cubic meter is preferred.

Terminology

The following terminology is used in the balance of this section to describe determination of reporting limits.
- *Method Detection Limit (MDL)* is defined in 40 CFR 136, Appendix B, as follows: "The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte." This term is comparable to the following terms in various standard methodologies:
  - *Limit of Detection (LOD)* – American Chemical Society
  - *Interlaboratory Detection Estimate (IDE)* – ASTM D6091
  - *Critical Value (CRV)* – International Standards Organization/International Union of Pure and Applied Chemistry (ISO/IUPAC)
  - *Minimum Detectable Value (MDV)* – ISO/IUPAC
- *Limit of Quantitation (LOQ)*, known in SW-846 as the Estimated Quantitation Limit (EQL). The LOQ is a statistically derived value, as described below. The term LOQ is used by ACS and ISO/IUPAC, and is comparable to the following terms in various standard methodologies:
  - *Minimum Level of Quantitation (ML)* – EPA
  - *Interlaboratory Quantitation Estimate (IQE)* – ASTM D6512

Determination of RL

The first step in this process is determination of the MDL. There are a number of published methodologies, several of which are described and evaluated by EPA (*Technical Support Document for the Assessment of Detection and Quantitation Approaches,* EPA-821-R-03-005, February 2003). This evaluation endorses the SW-846 MDL approach. Because that approach has been successfully used in the environmental regulatory context, it is also recommended for 10 CFR 850

compliance purposes. Alternative methods, if used, should demonstrate equivalent or superior performance.

The next step is determination of the LOQ. This value takes into account uncertainties in the analytical measurement system, such as using balances for mass, using dispensers for volumes, the effect of temperature variations on instrument readings, etc., by accepted methods such as those found in the *EURACHEM/CITAC Guide, Quantifying Uncertainty in Analytical Measurement, Second Edition* (Editors: S. L. R. Ellison, M. Rosslein, and A. Williams). The LOQ is generally found to be 5 to 10 times the MDL and is highly matrix-dependent. Again, there are a number of published methodologies, also described and evaluated in EPA-821-R-03-005. The EPA and ACS methodologies are said to be functionally equivalent but are calculated differently. Either should be equally acceptable for compliance with 10 CFR 850. Alternative methods, if used, should demonstrate equivalent or superior performance.

Setting the RL equal to the LOQ is the preferred procedure because it provides the most sensitive results with reproducible certainty. This is particularly useful for monitoring or clearance situations in which the levels are very low because it maximizes the data with actual values rather than assigning the RL to some of those data. This, in turn, increases the power of the statistical analyses of those data. However, setting an RL that is higher than the LOQ for the purpose of having a constant, non-fluctuating value, is a common practice because the LOQ is periodically (typically monthly) re-evaluated. The RL should be set as low as possible, and the IH should have the option of requesting the LOQ for sets of data in which it is important to maximize the data with values other than the RL.

Example Calculations

An illustration of how MDL, LOQ, and RL may be calculated:

A laboratory digests a set of beryllium samples and brings the digested samples volumes up to 25 mL for analysis by ICP-ES. It conducts an MDL procedure and determines MDL to be 0.155 µg/L.

On a per-sample basis, the lab calculates an MDL of 0.155 µg/L × 0.025 L/sample = 0.004 µg/sample. The MDL of the beryllium in a sample therefore is 0.004 µg.

To obtain the LOQ, the laboratory conducts an estimation of uncertainty procedure and determines its combined uncertainty to be 7.0. This includes the uncertainties inherent in analyzing this specific sample matrix.

It calculates an LOQ of 0.004 µg/sample × 7.0 = 0.028 µg/sample. The LOQ of the beryllium in a sample therefore is 0.028 µg for the specified sample matrix.

Qualified individuals typically submit sample volumes along with air samples to laboratories for analysis, and the laboratories return results in concentrations. The laboratories divide the mass of beryllium in the sample by the sample volume to calculate the concentration of beryllium in air.

When sample results are below the LOQ but above the LOD, laboratories typically report a result of "less than (LOQ value)." For instance, referring to the above example, such a result might be reported as "less than 0.028 μg" for the sample. In the case of air samples collected on a filter medium, laboratories would typically divide their LOQ by the sample volume to calculate a concentration result. For instance, if the sample volume is 1000 L (or 1 m^3), the result reported to the Registry would be "less than 0.028 μg/m^3." This concentration value is the value required by the DOE's beryllium registry, *Beryllium-Associated Worker Registry Data Collection and Management Guidance,* http://www.eh.doe.gov/health/worksurv/berylliumspecs.pdf, when a result is less than the LOQ.

Laboratories avoid reporting numbers for results between their MDL and LOQ because recipients often ascribe meaning to those numbers that is not technically valid. However, most laboratories will provide those numbers if the client insists and will flag the values to indicate that they are below the LOQ. For example, a laboratory might report a result of 0.014 μg for the sample or 0.014 μg/m^3 for a 1-m^3 volume sample and flag it as "less than the LOQ, LOQ = 0.028 μg" or "less than the LOQ, LOQ = 0.028 μg/m^3." Note that DOE's beryllium registry requires the LOQ, not a number between the MDL and LOQ.

Laboratories typically characterize results as "non-detect" for samples for which the beryllium mass result is less than the laboratory's MDL. They avoid providing numbers for those results because recipients often ascribe meaning to those numbers that is not technically valid. However, most laboratories will provide those numbers if the client insists and will flag the values to indicate that they are below the MDL. For example, a laboratory might report a number such as 0.002 μg for the sample or 0.002 μg/m^3 for a 1-m^3 volume sample and flag it as "less than the MDL, MDL = 0.004 μg" or "less than the MDL, MDL = 0.004 μg/m^3." Note that the DOE registry requires the LOQ, not the MDL.

3.8.3 Quality Assurance/Quality Control (QA/QC)

3.8.3.1 Accreditation versus Equivalence

Title 10 CFR 850.24(f) requires that samples be analyzed in a laboratory accredited for metals by the AIHA or "a laboratory that demonstrates quality assurance for metals analysis that is equivalent to AIHA accreditation." Although use of an accredited laboratory is typically preferred, use of a lab with "equivalent QA" may be desirable or necessary in some instances, especially for new labs in the process of obtaining accreditation and for labs analyzing radioactively-

contaminated samples. For a laboratory to be considered "equivalent", a documented evaluation of the laboratory QA program should be conducted, comparing the QA program to the AIHA *Policy Manual* or other recognized accrediting organization. This evaluation should be followed by a documented determination of "equivalence" by the appropriate site authority. Laboratories seeking initial accreditation should consult the AIHA web site (www.aiha.org) for information, including checklists used by AIHA evaluation teams.

3.8.3.2 Proficiency Analytical Testing (PAT) Samples

Currently, AIHA accreditation for metals includes beryllium and requires participation in the metals IHPAT sample program. AIHA offers a separate beryllium PAT (BePAT) program; however, participation in this program is not required for accreditation for metals (including beryllium). A separate beryllium accreditation program is under development, which will require BePAT participation. Although BePAT participation is not currently required, it is strongly encouraged.

Additionally, DOE and others are developing a more challenging BePAT program. Current BePAT samples use beryllium acetate, which provides the beryllium ion but does not require digestion. This was a good first step in developing a beryllium analysis proficiency program, but most beryllium samples contain BeO and many contain high-fired BeO, which is difficult to digest. This current effort requires developing a high-fired beryllium oxide (BeO) standard reference material (SRM). Once the BeO SRM is available, it is expected that BePAT samples could be used to incorporate BeO proficiency samples into the BePAT program (if approved by AIHA). This will provide a more effective means of ensuring that all participating labs have sufficiently robust digestion and analysis protocols. The BeO PAT samples should be available by the end of 2006. Once these samples are available, they should be used by labs analyzing beryllium samples for 10 CFR 850 compliance.

3.8.3.3 Quality Control

Appropriate QC protocols can be found in NMAM, SW-846, ASTM test methods such as D7035, *Standard Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)*, and other analytical methods compendia. The QC protocol used by the lab should be documented in lab procedures and should include (but not be limited to) the following:
- Media blanks (at least one per batch is recommended), which should be provided to the laboratory by the customer
- QC checks, such as a laboratory control sample (LCS), within a batch of samples
- Calibration range, and actions to take when sample results fall outside the calibration range

- Expiration dating for working-level calibration standards, particularly below 1 mg/L
- Corrective actions to take to resolve difficulties with any of the above.

3.8.4 Communication Between the Qualified individual and the Laboratory

3.8.4.1 Terminology

It is important for both IH and analytical lab personnel to have a common understanding of the meaning of terms such as reporting limit, LOD, LOQ, etc. Misunderstandings may result in misinterpretation of data, which could lead to incorrect field decisions.

3.8.4.2 Chain of Custody

Procedures such as ASTM D4840, *Standard Guide for Sampling Chain-of-Custody Procedures*, for managing chain of custody from the sample collector to receipt by the lab should be in place and agreed to by all involved parties. This is especially important when off-site labs are used.

3.8.4.3 Information Needed by the Lab

Routine procedures should document "normal" requirements regarding DQO and units of measure to be reported. The lab should be notified in the event of any of the following:
- Unusual sample matrix
- Potential interferences known to be in the sample(s)
- Other unusual requirements

3.8.4.4 Information Needed from the Lab

In addition to routine reporting requirements, the lab should notify the customer in the event of any of the following:
- Unexpected concomitants detected
- Levels of concomitants higher than expected
- Problems during analysis such as interferences, LOQ problems, matrix effects, or contamination found in one or more blanks

3.8.4.5 Data Transmission

Laboratories are encouraged to automate data reporting to the extent practicable. Chain-of-custody issues may exist with electronic data files; some labs address this issue by sending Portable Document Format (PDF) files rather than editable files.

3.8.5 Field Instrumentation

There is significant interest in equipment that could perform analyses of wipe and/or air filter samples in the field, saving both time and money as compared to taking such samples to a laboratory for analysis. These field instrument methods would be most useful where meeting the DQOs for compliance with 10 CFR 850 is not an issue, such as near real-time monitoring of exposures to complement compliance monitoring, identifying work practices that contribute to exposures, and diagnosing process equipment to determine the locations of emissions. A variety of development efforts are in progress, but none are currently validated for use. Any field instrumentation should be validated before it is used for compliance with 10 CFR 850.

Appendix 3-1

DRY WIPE SAMPLING

(Adapted from Y-12 Y73-66-IH-021INS)

Purpose

The objective of a surface wipe sample is to identify surface levels of removable contamination (as defined in 10 CFR 850.3 *Definitions*) of a metal in a semi-quantitative manner. Information obtained from surface wipe samples serves as a performance indicator for housekeeping practices.

Applies To

This instruction applies to Industrial Hygiene personnel who perform surface wipe sampling for metal analyses.

Prerequisites

Personnel must be trained and competent to perform this task.

Procedure

1. Describe personal protective equipment (PPE) requirements and potential hazards.

2. Collect the equipment, materials, forms, and sample containers required for the task:
    - Whatman® No. 41 or 42 filter media
    - Disposable 100-cm^2 templates as described in ASTM D6966
    - Disposable latex gloves
    - Sample containers as described in ASTM D6966
    - Sample identification labels
    - Sealable plastic bag for waste
    - Applicable warning labels for waste bags
    - Sampling forms
    - PPE required for the location and for this procedure

3. Label a sufficient number of sample containers with identification numbers. Include a minimum of 10% or at least two field blanks per sample set.

4. Don PPE as required and enter the area where the sample is to be collected.

5. Using clean, disposable gloves, remove the filter media from its package.

6. Applying firm pressure, swipe a 100-cm^2 area (use a template or may estimate the area of an irregular surface). Use the pattern given in ASTM D6966 for a square template.

7. Without allowing the filter to contact any other surface, fold the media (half or quarters) with the sample side inward, and place the filter completely inside the identified sample container.

8. Place the template (if used) in the waste bag.

9. Remove each disposable glove and place in the waste bag.

10. Document the necessary information on the appropriate sampling forms.

11. Repeat steps 5-10 until all desired samples have been collected. Place samples in a clean, sealable plastic bag. Apply any applicable warning labels to the bag's exterior.

12. Upon completion of the task, collect all materials and documentation. Doff PPE as required to exit the area where the sample(s) were collected. Dispose of waste materials in the appropriate manner.

13. Complete "chain of custody" procedures and take samples to the laboratory for analysis. If the samples cannot be taken directly to the laboratory, they must be placed in a secure location to ensure sample integrity. The location must also preclude sample cross-contamination.

Records

- Sampling results
- Completed chain of custody forms
- Field data sheets until results are formally accepted

Appendix 3-2

BULK SAMPLING

Collecting the Ambient Samples

1. Don latex gloves.

2. Determine 16 sample locations (i.e., two locations in each of eight directions: north, south, east, west, northeast, southeast, northwest, and southwest). When feasible, the locations should be at a distance of approximately 100 yards from the area or facility that is the subject of the sampling campaign. If visibly different types of soil are apparent, additional samples may be taken to represent the various soil types.

3. Collect the 16 samples using a plastic scoop that will dig into the surface.

4. Place the materials in plastic 50-mL vials.

5. Send the samples to an AIHA-accredited (or equivalent) laboratory.

Collecting the Target Samples

1. Prior to sample collection, collect 1 g of material (using a scale to ensure that 1 g has been collected). This will give the technicians collecting the samples a visual indication of how much material is needed for each 1-g sample.

2. For each sample collected, use a clean set of latex gloves, a new brush, and new white sheet of paper.

3. Using the brush, sweep at least 1 g of material onto a blank, white sheet of paper. A large area may be needed to accumulate a 1-g sample. Move the paper as necessary.

4. Fold the sheet of paper and use it to pour the material into a 100-mL jar. Seal the jar and label it with a unique identification number that is tied to the sample location.

5. Weigh the jar before sampling. Weigh the jar after sampling and continue to sample and weigh until at least 1 g of material has been collected.

6. Send the samples to an AIHA-accredited (or equivalent) laboratory.

Sample Analysis

1. Ambient and target samples are analyzed by the same method.

2. Optional: Analyzing each size fraction of each sample separately would allow a comparison of the ambient and target beryllium concentrations in the different fractions. Using a sieve, separate the soils into four particle sizes: 1 to 2 mm, 600 microns to 1 mm, 150 to 600 microns, and <150 microns.

3. Analyze the samples using methods discussed in section 3.8.2 of this Technical Standard.

4. The analysis should yield results in mg/kg or μg/g, which are equivalent dimensional units to ppm.

4. REMEDIATION AND HANDLING

4.1 Release of Equipment and Other Items

DOE sites frequently use equipment and other items (referred to as "equipment" for the balance of this guidance) for new purposes and release equipment to others when no longer needed by the site. Sites must consider the risk of exposure to subsequent users of equipment that may be contaminated with beryllium. There are three scenarios for sites to consider. The equipment:
- is beryllium-contaminated and subject to the Rule's release criteria provisions (10 CFR 850.31),
- contain levels of beryllium that are not subject to 10 CFR 850.31 or at one time were suspected of containing higher levels of beryllium but were subsequently found to not contain those levels, or
- is not contaminated with beryllium.

4.1.1 Releases Subject to 10 CFR 850.31

The Rule's release criteria provision (10 CFR 850.31) specifies control levels and the actions that must be taken for beryllium-contaminated equipment. Beryllium-contaminated equipment is equipment:
- in a beryllium regulated area, for which it has been established through sampling that the removable contamination, either internal or external, exceeds $0.2 \mu g/100 \text{ cm}^2$;
- considered by a qualified individual (e.g., a certified industrial hygienist) to be contaminated based on process knowledge; or
- considered by a qualified individual to pose a risk of exposure to the recipient during intrusive activities on the equipment or items.

If equipment is routinely sent to a specific recipient, the written notification and recipient's commitment required by 10 CFR 850.31 may be written to encompass the multiple releases over an established period of time as long as the conditions of release remain the same. Should conditions change, the recipient's commitment should be reestablished.

4.1.2 Releases Not Subject to 10 CFR 850.31 but Needing Controls

Equipment that is not beryllium-contaminated as described in Sect. 4.1.1 is not subject to 10 CFR 850.31, but the situation may still warrant control actions. Examples of these situations are:
- The equipment had been suspected of being beryllium-contaminated but was found not to be. Appropriate controls are to inform the recipient of the reasons for the original concern and the results of the evaluation that determined that the equipment is not contaminated.

- The equipment is beryllium-contaminated as described in Sect 4.1.1 but has been designated as waste. Title 10 CFR 850.31 is not applicable but 10 CFR 850.32 is.

4.1.3 Releases Not Subject to 10 CFR 850.31 or Needing Controls

A qualified individual should document the beryllium risk assessment for any equipment that the qualified individual considered sufficiently suspect to warrant the risk assessment but subsequently determined to not be subject to 10 CFR 850.31 or in need of any controls.

4.1.4 Release of Equipment Without Sampling

Qualified individuals may determine that sampling for removable contamination from equipment that is not beryllium-contaminated as described in Sect. 4.1.1 is not necessary based on a beryllium risk assessment considering process knowledge and/or representative sampling. The following are examples of equipment that may not require sampling for removable contamination prior to release:

- Equipment that is known never to have contacted removable contamination exceeding 0.2 µg/100 cm^2. Isolation techniques may be used while equipment is in proximity to removable beryllium to eliminate contact.
- Equipment for which it can be demonstrated that it has never been in locations with airborne releases of beryllium or removable contamination based on the beryllium inventory.
- Equipment for which it has been demonstrated through representative sampling of similarly exposed equipment that removable contamination would not be expected to exceed 0.2 µg Be/100 cm^2.

4.2 Waste Disposal

Title 10 CFR 850.32, *Waste disposal,* and 10 CFR 850.38, *Warning signs and labels,* provide the beryllium waste disposal requirements for DOE contractors. DOE Offices and contractors are required to seal waste in impermeable bags, containers, or enclosures to prevent the release of beryllium dust during handling and transportation prior to disposal and to label containers to warn the recipient of the waste of the potential hazard and to caution against unsafe practices. Title 10 CFR 850.31, *Release Criteria*, is not applicable to beryllium waste. Title 10 CFR 850.32 and 850.38 are not applicable to non-DOE operations or activities such as municipal landfills.

4.2.1 Description of Beryllium Waste

Beryllium waste is both beryllium-containing material and beryllium-contaminated equipment that is designated for disposal and that meet one of the following criteria based on process knowledge, calculation and analysis, and/or sampling:

- Equipment with removable beryllium contamination (internal or external) exceeding, or potentially exceeding, 0.2 µg/100 cm^2;
- Building materials and demolition debris containing beryllium exceeding 0.1 percent (w:w) (1000 parts per million); or
- Job-associated materials such as gloves, booties, and disposable coveralls coming from a regulated area or from an area where the concentration of beryllium can reasonably be expected to exceed 0.2 µg Be/m^3 in air or 0.2 µg Be/100 cm^2 removable from surfaces (unless it can be demonstrated that the material could not become contaminated through the use of isolation techniques such as placing a clip-board inside a plastic bag for a walkthrough of the area).

4.2.2 Exclusions from Beryllium Waste

4.2.2.1 Naturally Occurring Forms of Beryllium

Naturally occurring beryllium in background soil is not considered to be beryllium in the Rule and therefore is not beryllium waste (see Sect. 2.2.3). Other naturally occurring forms of beryllium are considered to be beryllium in the Rule at this time, but that inclusion may have been unintentional. A future interpretation by DOE's OGC or a rule revision may remove or exclude these forms of beryllium from the Rule (see Sect. 2.2.4).

4.2.3 Applicability of 10 CFR 850.31, Release Criteria, to Waste

The Rule's release criteria provision, 10 CFR 850.31, does not apply to beryllium waste. Any other viewpoint (e.g., that 10 CFR 850.31 does apply to this waste) would require a formal interpretation by DOE's OGC. OGC is the only DOE office that is authorized to interpret DOE regulations.

4.2.4 Decision Logic for Release of Equipment, Other Items and Waste to General Public and DOE Non-Beryllium Areas

Figure 4.1 provides a decision logic for determining the appropriate option for release of equipment and other items.

Figure 4.1 Decision Logic for Release or Equipment, Other Items, and Waste