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Metals Analysis Results for the Structural Qualification Test Series (SQTS) 01 - 05.

David Zalk

May 17, 2006

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UCRL-TR-221448

HAZARDS CONTROL DEPARTMENT

ES&H Team 1

June 27, 2001

ES&H 1-01-056

TO: Kent Haslam, Albert Lee, Jack Lowry, and Rick Visoria

FROM: David M. Zalk, MPH, CIH

SUBJECT: Metals Analysis Results for the Structural Qualification Test Series (SQTS) 01 - 05.

Enclosed is the report summarizing the metals analysis results at the Contained Firing Facility (CFF), during SQTS 01 - 05. This metals analysis includes evaluation of a bulk dust and surface swipe sampling protocol during the testing series that obtained samples at 3 primary locations in the CFF chamber area. The sampling protocol for each of the bulk dust samples involves an assessment of the concentration for 20 different metals, the oxidation state of selected metals, a particle size selective analysis, and morphological information. In addition, surface swipes were taken during SQTS 05 on the equipment and personnel door frames to indicate the characteristics of airborne metals due to leakage past the gasket seals. The bulk dust metals analysis indicates a nearly complete conversion of the aluminum casing to an oxide form with an even split between spherical and non-spherical morphology. Size selective analysis shows 83% of the particulates are in the inhalable size range of less than 100 microns and 46% are in the respirable range of less than 10 microns. Combining metals analysis and leakage results indicate the potential for a problematic personal exposure to metals external to the chamber unless modifications are made. Please feel free to call me at 2-8904 if you have any questions or if I may be of further service.

cc: Jerry Bardecker L-871
Carl Ingram L-170
Jim Lyle L-874
Lloyd Multhauf L-170
Dewey Ravenscroft L-874
Larry Sedlacek L-871
Larry Simmons L-871
Mark Sueksdorf L-871

University of California



**Lawrence Livermore
National Laboratory**

**METALS ANALYSIS RESULTS
FOR THE
STRUCTURAL QUALIFICATION TEST SERIES 01 - 05**

At

Site 300

Building 801

Contained Firing Facility

Prepared for:

Kent Haslam, Albert Lee, Jack Lowry, and Rick Visoria

Site 300

Lawrence Livermore National Laboratory

Prepared by:

**David Zalk
2-8904**

June 27, 2001

**METALS ANALYSIS RESULTS
FOR THE
STRUCTURAL QUALIFICATION TEST SERIES 01 - 05**

ABSTRACT

Industrial Hygienist: David M. Zalk, MPH, CIH
Facility: Building 801,
Contained Firing Facility
Dates: March 27 - April 26, 2001

GENERAL FINDINGS:

Bulk dust samples and surface swipe samples were taken from March 27 through April 26, 2001 at the Contained Firing Facility (CFF) firing chamber area during the Structural Qualification Test Series (SQTS) 01 through 05. The sampling protocol includes an assessment of the concentration for 20 different metals, the oxidation state of selected metals, a particle size selective analysis, and morphology.

Bulk Dusts; The bulk dust metals analysis indicates a nearly complete conversion of the aluminum casing to an oxide form and the creation of a variety of aluminum-containing compounds. Morphological information is available for SQTS 02 - 05 and it indicates an even split in the creation of spherical and non-spherical particulates. Size selective analysis for the entire test series shows 82.7% of the particulates are in the inhalable size range of less than 100 microns and 46.3% of the particulates are in the respirable range of less than 10 microns.

Surface Swipes; Surface swipe samples were taken after SQTS 05 on both the equipment door and personnel door frames to indicate the characteristics of airborne metals that leaked past the gasket seals. A mean of 88.4% of the particulates on the door frames were of a non-spherical morphology which indicates leakage quickly after shot time. The personnel door frame also had 6 times more particulates larger than 100 microns in width than did the equipment door, reflecting well on efforts to improve the equipment door seal.

Combined Analyses; Previous carbon monoxide monitoring indicates a leak potential at five locations. Metals analysis results show that virtually all metals within the chamber, even trace levels as alloys or residuals on the chamber surfaces, can be accounted for in the dust analyses and therefore may be present wherever leaks may occur as both airborne and surface particulate. Preliminary estimates indicate that approximately 10 milligrams of beryllium, per each kilogram of mass within the

chamber, may lead to personal exposures during personnel door re-entry procedures above the $0.2 \mu\text{g}/\text{m}^3$ Action Level for beryllium.

1.0 Introduction

After each test of the Structural Qualification Test Series (SQTS) at the Contained Firing Facility (CFF), there was a debriefing session. After SQTS 03 the Industrial Hygienist for CFF was tasked with performing and assessing a complete metals analysis on bulk dust samples that had already been taken, and a similar assessment for bulk dust samples that would be taken for the remainder of the SQTS. The sampling protocol for each of the bulk dust samples involves combined assessment methods for the concentration of 20 different metals, the oxidation state of selected metals, a particle size selective analysis, and obtaining morphological information. In addition, surface swipe sampling was performed during SQTS 05 on the equipment and personnel door frames to indicate the characteristics of airborne metals due to leakage past the gasket seals. The SQTS was also an opportunity to obtain other information covered in previous reports, such as area and personal sampling for airborne metals¹⁻⁶ and the potential for leakage at various locations external to the CFF chamber¹.

The metals analysis protocol includes a determination of the distribution of the dust and debris created by each of the tests. It was determined that the three best sampling locations to determine size distribution would be based on the dust's heaviest to lightest fractions. The heaviest fraction was presumed to be the dust remaining on the chamber floor after each shot. The lightest fraction was best reflected as the dust collected on a pre-filter surface as long as each sampled pre-filter was dedicated to one individual test. The dust that settled on horizontal surfaces on the chamber walls, such as the bolts on the inside of the equipment door, served as the collection area for the medium weight fraction. Previous metals evaluations occurred to determine the potential for personal exposures and the efficiency of the ventilation within the chamber determined in earlier personal sampling reports¹⁻⁵.

2.0 Operational Description

Hazards Control ES&H Team 1 employees, Ron Cadiz, Jeff DeBisschop, and David Zalk collected bulk dust samples and surface swipe samples from March 27 through April 26, 2001. The bulk dust sampling was performed at various locations in and around the CFF firing chamber located in Building 801 (B-801). Bulk dust sampling was systematically collected, when possible, from the chamber floor, the upstream side of a pre-filter dedicated to an individual test, and from the horizontal surfaces of bolts on the inside of the equipment door. Additional bulk dust samples were taken from the camera room, after a leak in a gasket during SQTS 03 or 04 became apparent, and from the collection bag of a chamber floor vacuum after SQTS 02. Surface swipe sampling was performed during SQTS 05 on the hinge and open side of the equipment door and on the open side of the personnel door frames. These surface samples were taken of a

white, powdery residue that formed across the inside frame of the door and past the gasket seal. These surface swipe samples were taken to indicate the characteristics of airborne metals due to leakage past the gasket seals. This metals analysis sampling protocol occurred during the five experiments that were part of the originally scheduled SQTS.

The SQTS for the Contained Firing Facility (CFF) chamber included five planned experiments. The five experiments had different amounts of C-4 explosive in an aluminum casing. The first experiment, SQTS 01, contained 15 Kilograms (Kg) of C-4 and occurred on March 26, 2001. SQTS 02 had 30 Kg of C-4 and occurred on April 3rd. SQTS 03 had 60 Kg of C-4 and occurred on April 11th. SQTS 04 had 75 Kg of C-4 (125% overtest of the operational limit) and occurred on April 19th. SQTS 05 had 35 Kg of C-4 and occurred on April 26th. The experiments for SQTS 01 through 04 were placed in the middle of the chamber at a point 4 feet above the chamber floor. Both the location and size of SQTS 05 were the most indicative of future testing with the experiment placed in front of the FXR target on the beam center line.

The bulk dust samples were taken the day following a test when possible. Chamber floor samples were taken at random locations on the north side. Pre-filter samples were taken after the removal of a single pre-filter dedicated to an individual test and were taken from the upstream side in the center of the filter. Equipment door bulk samples were taken from a powdery gray dust that settled on the top horizontal surface of twelve bolts facing the chamber side of the equipment door at a height of approximately four feet above the chamber floor. The camera room bulk sample was taken after SQTS 05, but the bulk dust sampled was shaken loose from a previous leak in a gasket around a portal that occurred during SQTS 03 and possibly SQTS 04. The surface swipe samples were taken, over an area of approximately 100 square centimeters, of a white, powdery residue that formed across the inside frame of the equipment and personnel door frames and across their gasket seals. The locations of all bulk dust samples, surface swipe samples, as well as area and personal sampling for metals, are outlined within the map listed as Attachment A at the end of this report.

3.0 Sampling and Analytical Methodologies

Metals analysis was performed on the bulk dust samples by digesting the samples using the Environmental Protection Agency (EPA) SW-846 Method 3050A and analyzing for 20 metals using EPA SW-846 Method 6010A. Sample analysis for these metals was by Inductively Coupled Argon Plasma - Atomic Emission Spectrometry (ICP-AES) and was performed by the LLNL Hazards Control Analytical Laboratory, an American Industrial Hygiene Association (AIHA) accredited laboratory. This method is considered semi-quantitative, but is essential for analyzing aluminum in the presence of all the other Industrial Hygiene series of metals sampled. The one environmental sample for metals within a digested pre-filter was sampled according to the Total Threshold Limit Concentration (TTLIC) metals analysis by the EPA SW-846 Method

3050A, then analyzed for metals with Method 6010A. Analysis for mercury was performed in accordance with Method 7471A by the LLNL C&MS Environmental Services, accredited by the Environmental Laboratory Accreditation Program (ELAP).

For the analysis of bulk dust and surface swipe samples, particle characterization, size distribution, and morphology was performed with Scanning Electron Microscopy (SEM). A portion of each sample was mounted on a conductive carbon tab on an SEM stub. To minimize extraneous peaks, the samples were not coated. Each sample was analyzed in the SEM at 20kV accelerating voltage and a magnification of 100 - 2500 times. Elemental compositions were determined using energy dispersive x-ray spectroscopy (EDX) which can detect elements from boron (atomic number 5) and up in concentrations above approximately 0.5 percent. Laboratory analysis was performed by Forensic Analytical, an American Industrial Hygiene Association (AIHA) accredited laboratory.

For area monitoring of the Industrial Hygiene series of metals¹, SKC air sampling pumps were used for the personal sampling with cassettes in line pre-calibrated at 2.0 liters per minute (l/min) with a precision rotameter previously calibrated against a primary standard. Cassettes were 37 millimeter, 0.8 micron pore size, mixed cellulose ester fiber (MCEF) filter and were connected inline with Tygon tubing. A post-survey flow rate was performed to check for change in sampling rate due to build-up on the cassette filter. Sample analysis for IH metals was by Inductively Coupled Argon Plasma - Atomic Emission Spectrometry (ICP-AES) utilizing NIOSH Method 7300. The analysis was performed by the LLNL Hazards Control Analytical Laboratory, an American Industrial Hygiene Association (AIHA) accredited laboratory.

4.0 Results

This section is divided into three parts to reflect the three different types of sampling methods used to obtain metals analysis information. The three different sampling methods, reflecting the three parts of this section, are bulk dust analysis, surface swipe analysis, and airborne dust analysis. The first part of this section on bulk dust analysis begins with the metals concentrations for 20 different metals across SQTS 01 - 05 which are listed in Table 1. The particle size selective analysis begins in Table 2 showing a graphic representation for the bulk dust, divided into four different Industrial Hygiene-related size categories, across SQTS 01 - 05. Morphology of the bulk dust, as well as some identified oxidation states for certain metals and compounds are presented.

The information presented in Table 3 through Table 9 is in the form of percentages. Each compound listed is shown as a percentage composition of its original sample constituents. Therefore Tables 3 - 9 may have a grouping of percentages that do not add up to hundred, this is because there are other compounds within the same sample that constitute its total composition. Table 3

shows the fate of aluminum and aluminum oxide and its morphological state, representing a particle being seen as either spherical or non-spherical. Table 4 shows additional spherical/non-spherical compound concentrations and Table 5 lists other non-spherical compounds. The next sub-section combines the data from SQTS 02 - 05 to show more detail regarding the size selective information for aluminum oxide as either spherical (Table 6) or non-spherical (Table 7) particulates. Pictures from some of the SEM analysis protocol are included for your viewing pleasure and include the images used determining this combined data for the chamber floor after SQTS 04 and 05.

Surface swipe sampling results are from SQTS 05 and were taken on the hinge and open side of the equipment door and on the open side of the personnel door frames. Results that detail aluminum oxide size selective information as spherical aluminum oxide are in Table 8 and non-spherical in Table 9. Airborne monitoring is then presented and is divided into area and personal sampling efforts discussed in previous reports^{1, 5, 6}. Results for area monitoring of the Industrial Hygiene series of metals taken at the hinge side of the equipment door, and at 15 feet away, are presented in Table 10. Table 11 shows personal breathing zone metals results at the personnel door during re-entry for SQTS 04 and SQTS 05. Locations of bulk dust samples, surface swipe samples, as well as area and personal sampling for metals, are outlined within the map listed as Attachment A at the end of this report.

4.1 Results; Bulk Dust

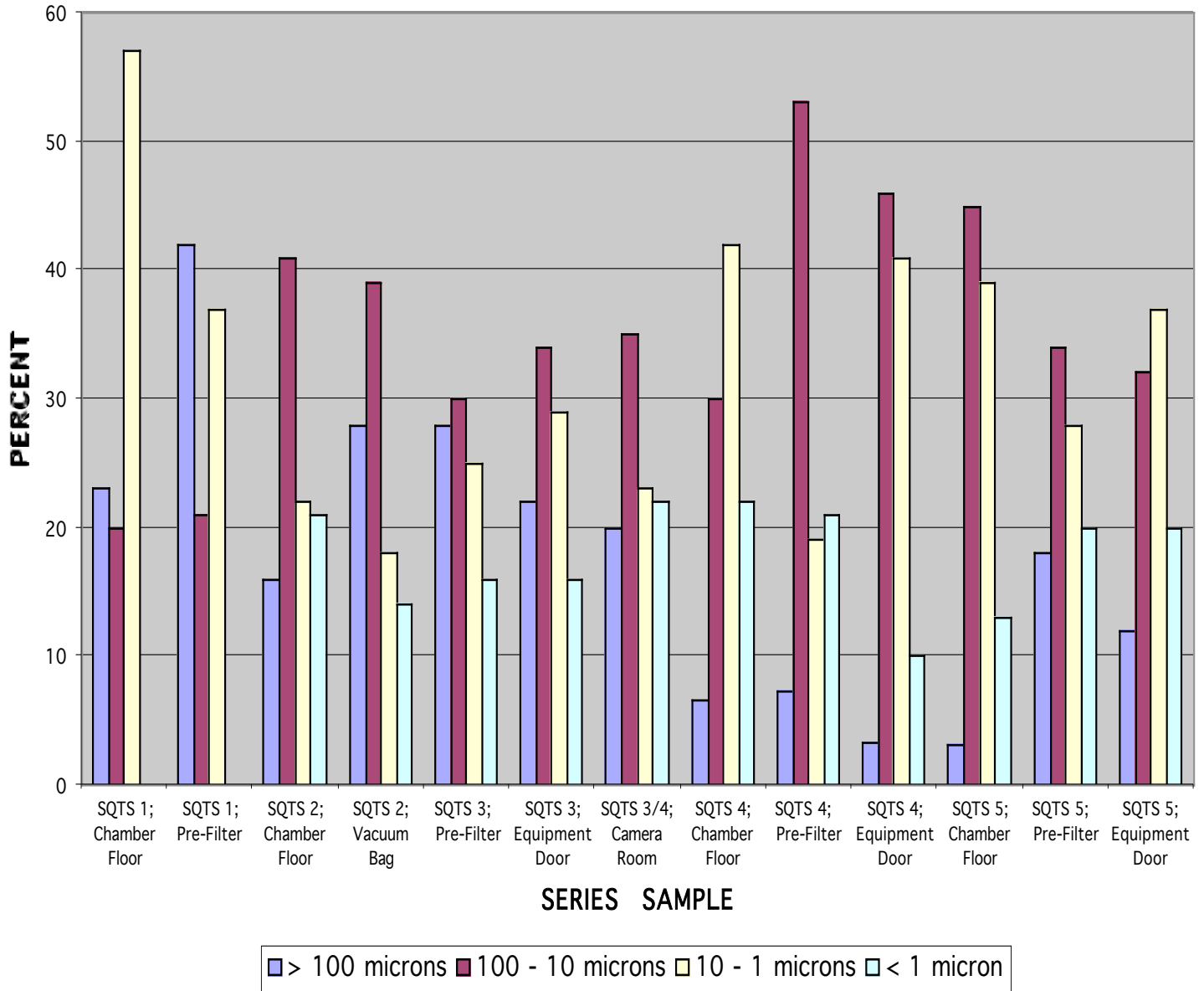
4.1.1 Metals Concentration

TABLE 1; Metals Concentrations for Bulk Dust Samples

IH METALS BULK SAMPLES	SQTS 01		SQTS 02		SQTS 03		SQTS 04		SQTS 04		SQTS 05		SQTS 05	
	Chamber (ug/g)	Floor Pre-Filter (ug/g)	Chamber (ug/g)	Floor Equipment (ug/g)	Door Pre-Filter (ug/g)	Chamber (ug/g)	Floor Equipment (ug/g)	Door Pre-Filter (ug/g)	Camera (ug/g)	Room (ug/g)	Chamber (ug/g)	Floor Equipment (ug/g)	Door (ug/g)	Pre-Filter (ug/g)
ALUMINUM	68,200	92,300	99,200	39,600	90,800	190,000	37,800	76,400	90,800	93,300	45,700	51,200		
ANTIMONY	<40	<40	<40	<40	<40	<40	<40	<40	<40	<40	<40	<40	<40	<40
BARIUM	153	173	89.3	39.1	48.5	32.2	29.6	42.8	110	38	27	545		
BERYLLIUM	0.3	0.246	0.35	0.244	<0.1	<0.1	<0.1	<0.1	0.626	<0.1	<0.1	<0.1	<0.1	<0.1
CADMIUM	6.85	8.79	3.52	<1.3	4.06	<1.3	<1.3	1.87	42	<1.3	<1.3	3.16		
CHROMIUM	583	711	837	272	691	642	199	535	393	342	305	520		
COBALT	17.5	10.4	16.3	5.69	6.54	5.97	3.42	5.95	18.9	5.11	4.93	10.9		
COPPER	2,380	2,680	3,440	1,860	5,080	2,090	1,410	3,930	>3,000	1,940	1,860	2,610		
IRON	50,300	45,500	40,000	10,000	12,700	23,600	9,130	13,900	70,600	15,600	14,500	24,100		
LEAD	54.8	135	39.4	33.6	155	28.2	11.2	103	142	20.8	27.4	79.8		
MANGANES	940	845	704	397	416	754	388	438	2,510	494	408	491		
MOLYBDENUM	26.2	38.7	16.3	<10	36.6	10.8	<10	37.8	42.8	13.4	15.1	30.4		
NICKEL	401	260	536	154	273	162	80.2	213	197	155	170	216		
SELENIUM	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100
SILVER	7.21	19.4	10.3	5.64	31.5	1.9	3.18	11.4	5.64	3.53	3.88	8.41		
THALLIUM	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30	<30
URANIUM	<70	<70	<70	<70	<70	<70	<70	<70	<70	<70	<70	<70	<70	<70
VANADIUM	35.3	47.1	30.2	19.2	32.9	37	16.9	32.8	29.5	22.5	17.9	24.9		
ZINC	1,430	2,620	737	271	2,950	206	156	3490	1,220	273	235	2,760		

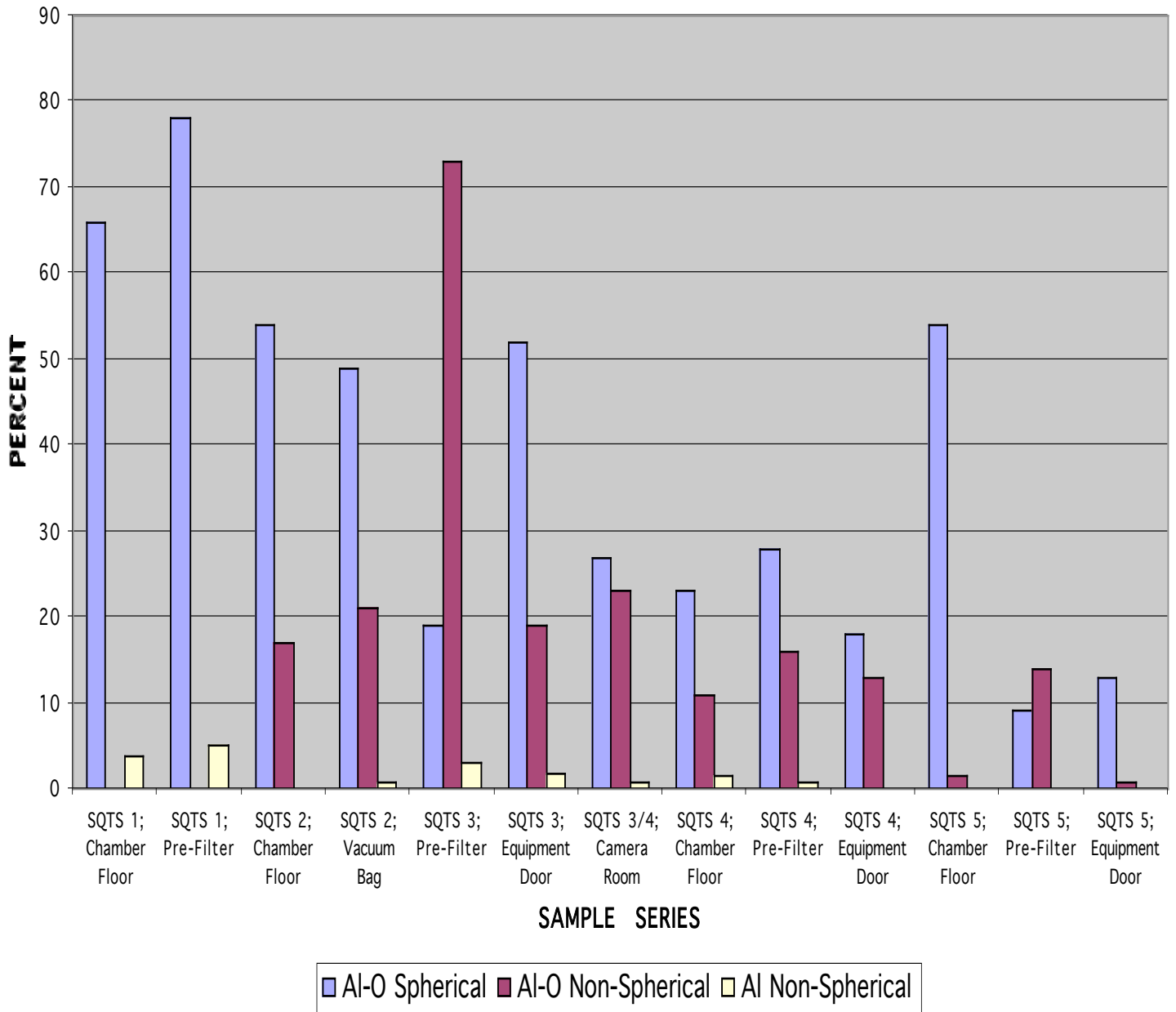
4.1.2 Particle Size Selective Analysis (SQTS 01 - 05)

TABLE 2; Bulk Dust - Size Selective Analysis



4.1.3 Morphology And Oxidation States (SQTS 01 - 05)

TABLE 3; Aluminum morphology & Oxidation States*



* Each compound is shown as a percentage distribution within its own dust sample.

TABLE 4; Other Compounds and Their Morphology**

BULK SAMPLE INFORMATION	Al-Si-Ca-Fe-O		Al-Si-Ti-Fe-O		Al-Fe-O	
	Spherical (%)	Non-Spherical (%)	Spherical (%)	Non-Spherical (%)	Spherical (%)	Non-Spherical (%)
SQTS 1; Chamber Floor	16	*	6.3	*	0	0
SQTS 1; Pre-Filter	4.1	*	1.5	*	0	0
SQTS 2; Chamber Floor	6.3	7.8	4.7	7	0	0
SQTS 2; Vacuum Bag	0.7	1.4	2.8	9.9	0	0.7
SQTS 3; Pre-Filter	0	0	0.5	1.5	0	0
SQTS 3; Equipment Door	0	0	17	0	0	3.5
SQTS 3/4; Camera Room	5.2	17	5.2	7	0	10
SQTS 4; Chamber Floor	15	11	10	3.3	20	0
SQTS 4; Pre-Filter	2.9	8.8	12	7.3	0	19
SQTS 4; Equipment Door	17	14	19	10	0	0
SQTS 5; Chamber Floor	3.2	0.8	4	4.8	0	25
SQTS 5; Pre-Filter	11	25	7.2	9.9	2	15
SQTS 5; Equipment Door	1.7	5	13	9.2	26	30

* Not Determined for SQTS 01 Samples

** Each compound is shown as a percentage distribution within its own dust sample.

TABLE 5; More Non-Spherical Compounds*

BULK SAMPLE INFORMATION	Si-O (%)	Al-Ca-O (%)	Fe-O (%)	Si-Al-O (%)	Cu-Al-O (%)	Al-Cl-O (%)	Al-Si-Ca-O (%)	Al-Ca-Fe-O (%)
SQTS 1; Chamber Floor	0.6	2	0.6	3	1.2	0	0	0
SQTS 1; Pre-Filter	1	0.52	0	7.3	0	0	0	0
SQTS 2; Chamber Floor	0	0	0	2.3	0	0	0	0.78
SQTS 2; Vacuum Bag	0	0	0	13	0	0	0	0
SQTS 3; Pre-Filter	0	0	0	3	0	0	0	0
SQTS 3; Equipment Door	0	0	0	2.6	0	1.7	2.6	0
SQTS 3/4; Camera Room	0	0.87	2.6	1.7	0	0	0	0
SQTS 4; Chamber Floor	0	0	0.55	4.4	0	0	0	0
SQTS 4; Pre-Filter	0	0.73	0	4.4	0	0	0	0
SQTS 4; Equipment Door	0	0	0	6.7	0	2.2	0	0
SQTS 5; Chamber Floor	0	0.8	0.8	4.8	0	0	0	0
SQTS 5; Pre-Filter	0	0.66	0	5.3	0	0	0	0
SQTS 5; Equipment Door	0	0	0.55	4.4	0	0	0	0

* Each compound is shown as a percentage distribution within its own dust sample.

4.1.4 Combined Aluminum Information (SQTS 02 - 05)

TABLE 6; Size Selective; Spherical Aluminum Oxide *

* Each compound is shown as a percentage distribution within its own dust sample.

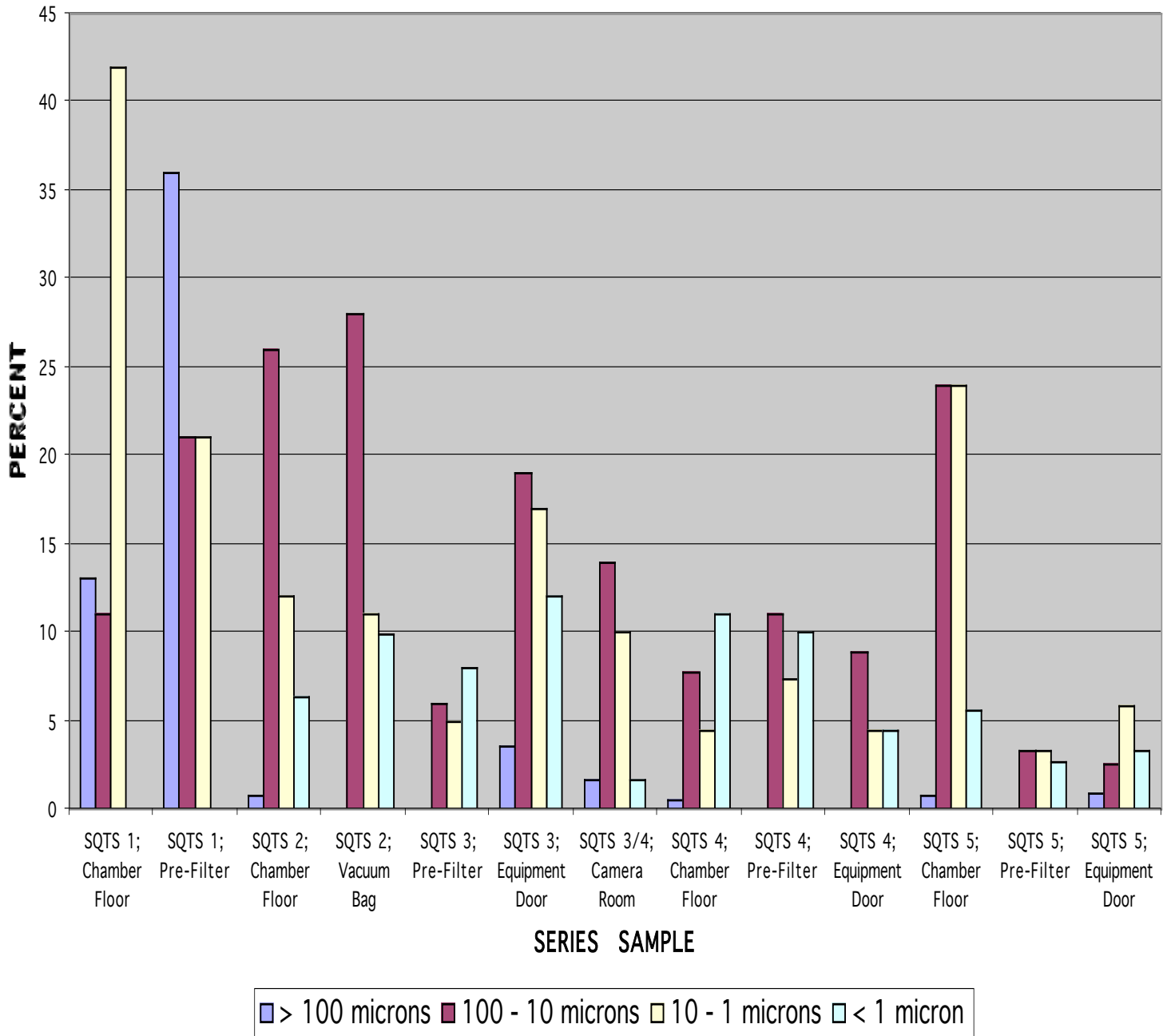
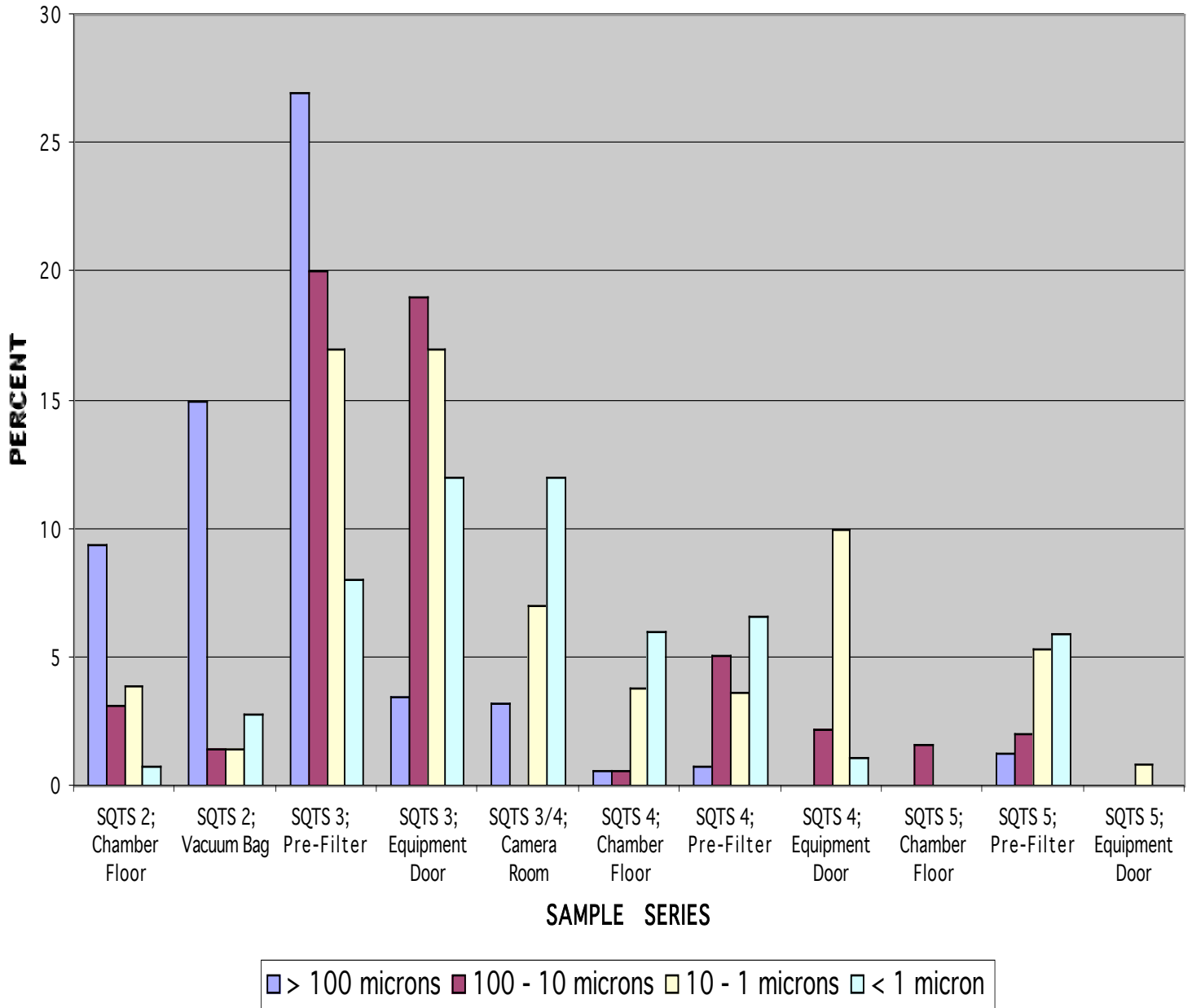
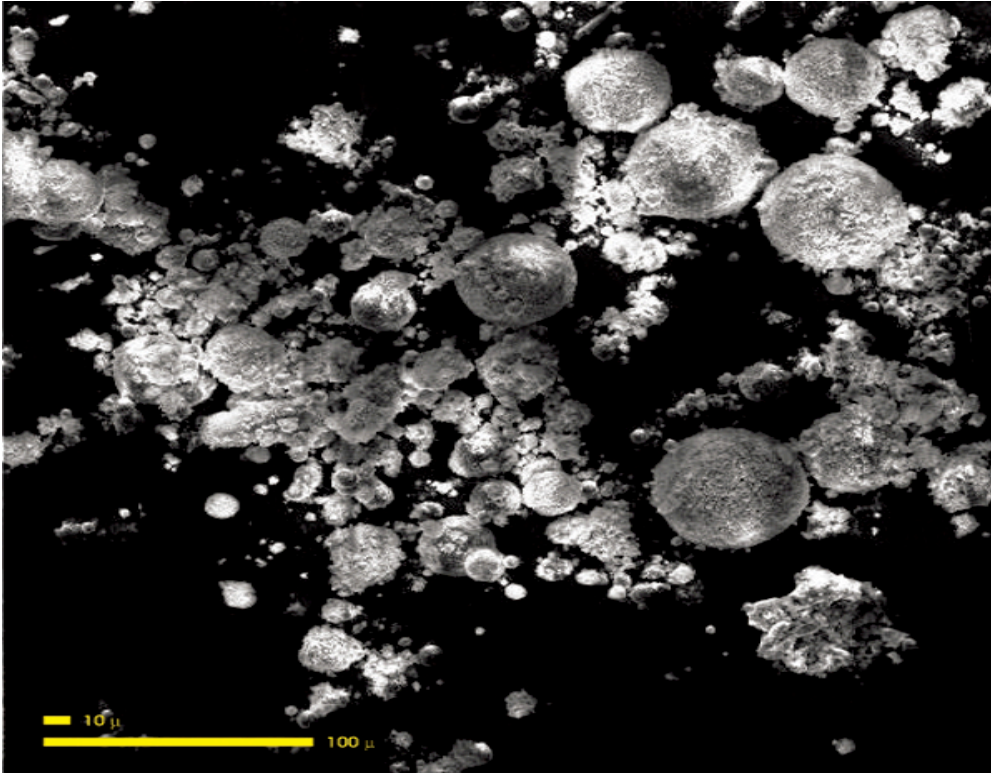


TABLE 7; Size Selective; Non-Spherical Aluminum Oxide*

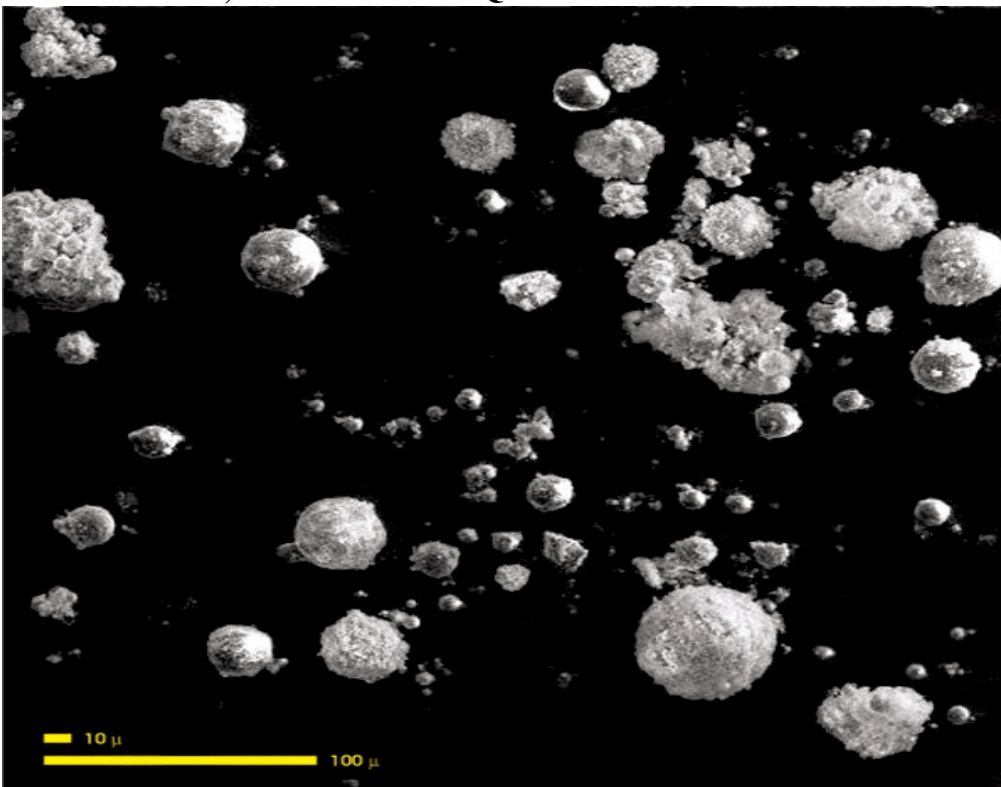


* Each compound is shown as a percentage distribution within its own dust sample.

4.1.5 SEM Pictures Used to Derive Combined Information
Picture 1; Chamber Floor SQTS 04



Picture 2; Chamber Floor SQTS 05



4.2 Results; Surface Swipes

4.2.1 Combined Aluminum Information (SQTS 05)

TABLE 8; Size Selective; Spherical Aluminum Oxide*

SQTS 5 SWIPE SAMPLE INFORMATION	Al-O Spherical > 100 microns (%)	Al-O Spherical 100 - 10 microns (%)	Al-O Spherical 10 - 1 microns (%)	Al-O Spherical < 1 micron (%)
Equipment Door; Open Side	0	8.9	8.9	4.9
Equipment Door; Hinge Side	0	0	1.9	1.9
Personnel Door	0	0	0	0.81

* Each compound is shown as a percentage distribution within its own dust sample.

TABLE 9; Size Selective; Non-Spherical Aluminum Oxide*

SQTS 5 SWIPE SAMPLE INFORMATION	Al-O Non-Spherical > 100 microns (%)	Al-O Non-Spherical 100 - 10 microns (%)	Al-O Non-Spherical 10 - 1 microns (%)	Al-O Non-Spherical < 1 micron (%)
Equipment Door; Open Side	1.6	11	28	11
Equipment Door; Hinge Side	0	0	26	1.8
Personnel Door	11	12	24	25

* Each compound is shown as a percentage distribution within its own dust sample.

4.3 Airborne Metals

4.3.1 Area Metals (SQTS 04 - 05)

TABLE 10; Area Metal s at Hinge Side of the Equipment Door for SQTS 4 and 5

Metals	SQTS 4 At Door (ug/m3)*	SQTS 4 At 15 Feet (ug/m3)	SQTS 5 At Door (ug/m3)	SQTS 5 At 15 Feet (ug/m3)
Aluminum	226	74.7		
Copper	10.7	4.43		
Iron	37.1	13.9	10.2	2.86
Manganese	0.953	0.411		

* $\mu\text{g}/\text{m}^3$ = micrograms of metal per cubic meter of air.

4.3.2 Personal Metals (SQTS 04 - 05)

TABLE 11; Breathing Zone Metals Results at Personnel Door During Re-Entry for
SQTS 04 and SQTS 05

Metals	Employee 1 SQTS 4 (ug/m3)	Employee 2 SQTS 4 (ug/m3)	Employee 1 SQTS 5 (ug/m3)	Employee 2 SQTS 5 (ug/m3)
Aluminum	33.8	78.6	57.1	34.3
Copper	2.03	3.54	2.03	1.69
Iron	7.07	14.4	11.1	10.2
Manganese		0.34		

5.0 Discussion

The intention of this report is to gain as much information as possible from the SQTS to develop some insight for potential Industrial Hygiene-related concerns for the use of beryllium within the CFF chamber. It must first be noted that the information discussed in this report is based on aluminum being the primary mass weight of metal within the CFF chamber. It is acknowledged that there is a considerable difficulty when comparing aluminum to beryllium. Beryllium is one third the weight of aluminum and has a melting point about twice as high. Finally divided aluminum dust is easily ignited which can add to the heat of reaction which may render difficulty in creating analogies to beryllium. This information is problematic when considering beryllium's ability to hold its shape over a wide temperature range, its brittle nature, its high heat-absorption capacity, and that it is about six times stiffer than steel. Beryllium is similar to aluminum within the CFF chamber environment in that its chemical properties are such that they are both metals resistant to attack by acid due to the formation of a thin oxide film. Comparison becomes even more problematic when it is considered that there is relatively little or no information on the particle characterization of beryllium within an explosive test environment, especially when performed indoors.

5.1 Bulk Dust

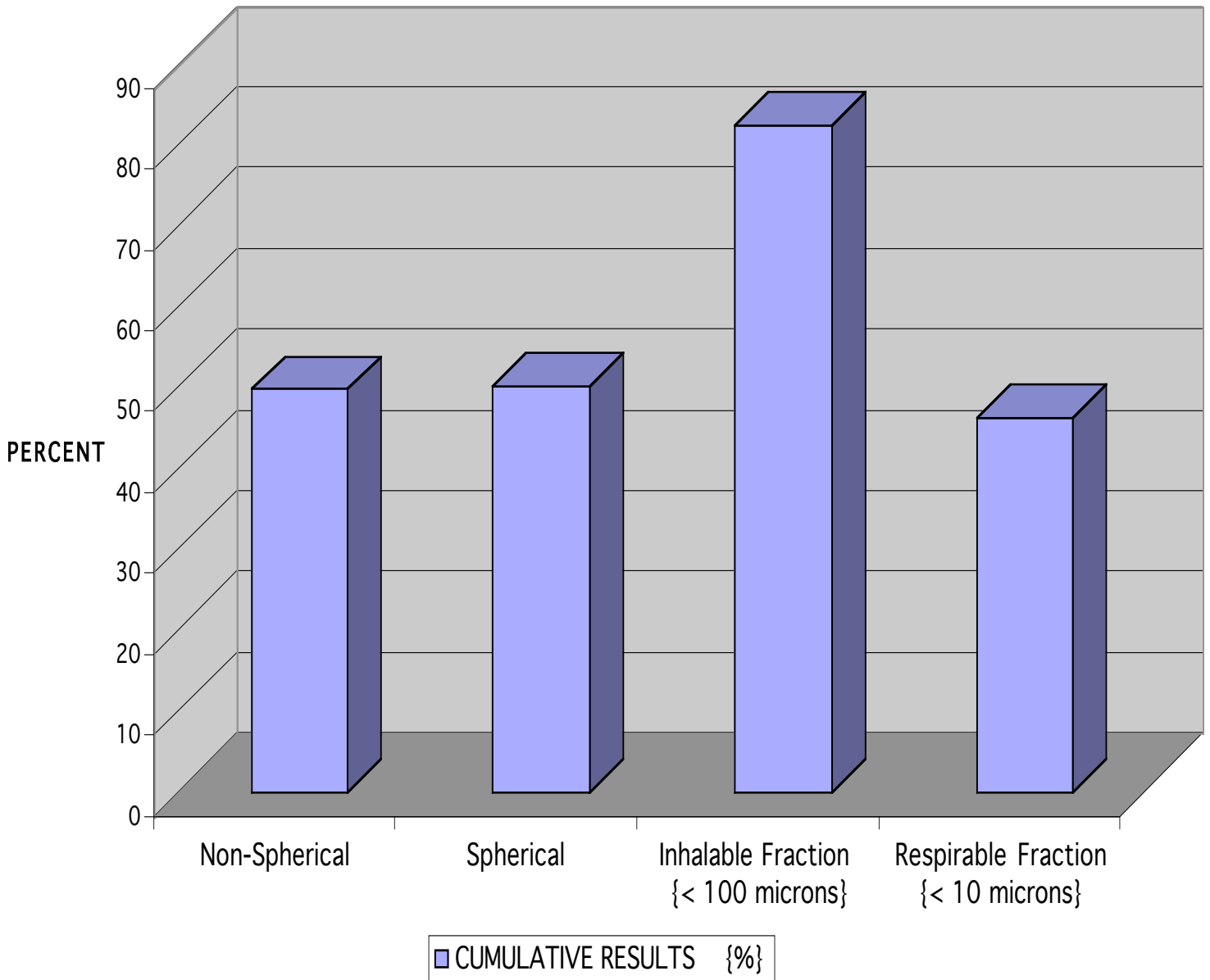
The bulk dust metals analysis indicates a nearly complete conversion of the aluminum casing to an oxide form and the creation of a variety of aluminum-containing compounds. The higher than expected heat of combustion due to aluminum dust's ignitability within the chamber may have accounted for this conversion being more complete than expected. The cumulative information is summarized in Table 12 below. Table 12 reflects the percentages across all the bulk dust samples for general morphology and size selective information. Morphological information is available for SQTS 02 - 05 and it indicates an even split in the creation of both spherical and non-spherical particulates. Size selective analysis for the entire test series shows 83% of the particulates are in the inhalable size range of less than 100 microns and 46% of the particulates are in the respirable range of less than 10 microns. The inhalable mass fraction is considered to be the size range within which airborne particulates can enter into the human respiratory system. The respirable mass fraction is the size range that allows airborne particulate to enter the alveolar region of the lungs where scarring is most likely to occur.

The size selective information across all bulk dust samples indicate that 46% of the particulates are less than 10 microns and 37% of the particulates are within the 10 to 100 micron size range. This is important to consider in light of the Industrial Hygiene personal sampling protocol. The current beryllium standard within the LLNL Chronic Beryllium Disease Prevention Program, calls for the use of a Total Dust sample cassette which is not size selective and can not be correlated with size specific information. The

current version of the ACGIH Threshold Limit Values (TLV) for Chemical Substances⁷ has beryllium on its Notice of Intended Changes with an 8-hour Time Weighted

Average of $0.1 \mu\text{g}/\text{m}^3$ taken as an inhalable sample. Other occupational exposure limit setting bodies are considering sampling for beryllium in the respirable fraction only.

TABLE 12; Cumulative Results; As Percentages Across All Bulk Dust Samples



When particulates in the inhalable size range are captured on a personal sampling cassette, there is an increase in weight of the sample collected which may be

problematic for low occupational exposure values for toxic chemicals, such as beryllium's current $0.2 \mu\text{g}/\text{m}^3$ Action Level. Alternatively, although particulates in the respirable size range will weigh less as collected, they will remain in the air much longer than larger particulate. This is especially true for low density elements like beryllium. To better understand this concept it is important to discuss the terminal settling velocity of particulates which is reflected in the following equation⁸:

$$V_{\text{TS}} = (\rho_p)(d^2)(g)/18\eta \quad \text{for } d > 1 \mu\text{m},$$

$$V_{\text{TS}} = \text{Terminal Settling Velocity (cm/s)}$$

$$\rho_p = \text{Density of the Particle (g/cm}^3\text{)}$$

$$d = \text{Diameter of Sphere (cm)}$$

$$g = \text{Acceleration of Gravity (cm/s}^2\text{)}$$

$$\eta = \text{Gas Viscosity (g/cm-s)}$$

Terminal settling velocity increases rapidly with particle size, being proportional to the square of particle diameter. A simpler version of this equation, valid only for unit-density spheres at standard conditions is:

$$V_{\text{TS}} \approx 0.003(d^2) \quad \text{for } 1 < d < 100 \mu\text{m}$$

Therefore, the relative size of the particulates available to become airborne becomes extremely important in determining the potential for it to be collected on a personal sampling cassette. The larger sizes may settle quicker, however more mass will be collected on a given cassette. The smaller sizes may not weigh as much on collection, however they have a much greater chance of staying airborne for a longer period of time. Since the density of beryllium is approximately three times less than aluminum, even larger beryllium-containing particulates will have a greater chance of remaining airborne longer than aluminum-containing particulates. This simplified approach breaks down somewhat with consideration for morphology and other environmental factors, however more research into this area is necessary before more specifics can be realistically added to the equation.

5.1.1 Possible Metal Sources (SQTS 01 - 05)

Virtually all of the elements within the CFF chamber can be accounted for. It is the source of these elements that may be put into question. It has become apparent that all of the test apparatus constituents be considered. Also to be considered is not only the residual layers of dirt and debris that accumulate on the chamber surfaces, but also materials used on the surfaces of the chamber. This includes the paint used to coat the

chamber floors and walls. Residual environmental surface levels of beryllium and cadmium, for example, seem to be flushed out of the chamber after SQTS 03. An attempt to account for these sources is presented below in Table 14 and include information from Table 1 and during calibration for these results as well, and

environmental digestion of a pre-filter Total Threshold Limit Concentrations (TTL) for metals presented in Table 13.

TABLE 13

PRE-FILTER METALS ENVIRONMENTAL ANALYSIS	TOTAL THRESHOLD LIMIT CONCENTRATION VALUES (mg/kg)
Antimony	3.3
Arsenic	< 1.6
Barium	65.7
Beryllium	2.2
Cadmium	2.3
Chromium	122
Cobalt	2.6
Copper	334
Lead	35.1
Manganese	126
Mercury	24.2
Molybdenum	3.9
Nickel	43.4
Potassium	575
Selenium	< 1.5
Silver	2.7
Strontium	454
Thallium	< 4.4
Vanadium	18.8
Zinc	729

TABLE 14

BULK DUST ANALYSES	POTENTIAL SOURCES
Aluminum	Casing and Shot Stand
Antimony ***	Below a High Limit of Detection (40 ug/g)
Arsenic	Below a High Limit of Detection (50 ug/g)
Barium	Experimental Assembly
Beryllium	Environmental Background, Bullnose Surface. Flushed Out After SQTS 03
Cadmium	Environmental Background. Flushed Out After SQTS 03
Calcium*, **	Environmental Background, Concrete
Chromium	Stainless Steel Wires Used for Seismic Restraint, Aluminum Alloy
Cobalt	Experimental Assembly
Copper	Stainless Steel Wires Used for Seismic Restraint, Aluminum Alloy
Iron	Nuts, bolts, screws, washers
Lead	Experimental Assembly, Lead/Tin Solder
Lithium**	Environmental Background, Possible Historical Bullnose Surface
Magnesium	Aluminum Alloy
Manganese	Stainless Steel and Aluminum Alloy
Mercury ***	Mildew Resistant Additive in Paint Coatings
Molybdenum	Experimental Assembly, Stainless Steel Alloy, Lubricant
Nickel	Stainless Steel Wires Used for Seismic Restraint
Niobium **	An Alloy of Depleted Uranium, Bullnose Surface
Potassium ***	Environmental Background, Possible Historical
Selenium	Below a High Limit of Detection (100 ug/g)
Silica *, **	Aluminum and Stainless Steel Alloy, Paint Coating Additive, RTF Sealant
Silver	Experimental Assembly, Solder
Sodium **	Environmental Background
Strontium ***	Possible Historical Background or Bullnose Surface
Thallium	Below a High Limit of Detection (30 ug/g)
Titanium*	Pigment in the Chamber Surface Paint Coating
Uranium	Below a High Limit of Detection (70 ug/g)
Vanadium	Experimental Assembly
Zinc	Experimental Assembly, Aluminum Alloy

* From SEM Analysis

** From ICP Calibration Scan

*** From TTLC Environmental Analysis

5.2 Surface Swipes

Surface swipe samples were taken after SQTS 05 on both the equipment door and personnel door frames to indicate the characteristics of airborne metals due to leakage past the gasket seals. These surface samples were taken of a white, powdery residue that formed across the inside frame of the door and across the gasket seal. The surface swipes were taken, over an area of approximately 100 square centimeters. These surface swipe samples were taken to indicate the characteristics of airborne metals due to leakage past the gasket seals. A mean of 88.4% of the particulates on the door frames were of a non-spherical morphology which indicates leakage quickly after the initial shot time. The personnel door frame also had 6 times more particulates larger than 100 microns in width when compared to the equipment door. This may reflect positively on efforts to seal in larger particles within the chamber at the equipment door. This information is similarly supported by the airborne metals information and carbon monoxide leak detection methods discussed below.

5.3 Airborne Metals

Area, non-personal, measurements for airborne metals, reflected in Table 10, were all taken at the Hinge Side during SQTS 04 and 05. Measurements for metals at the door were taken against the hinge side of the equipment door and also at 15 feet away from that point, both at a height of 4 feet above the floor¹. The levels for aluminum taken during SQTS 04 were 226 $\mu\text{g}/\text{m}^3$ at the door and 74.7 $\mu\text{g}/\text{m}^3$ at a point 15 feet away. The reductions in exposure levels for aluminum, as well as copper and manganese, were reduced to a limit below the limit of detection for SQTS 05. Only iron was present at levels of 10.2 $\mu\text{g}/\text{m}^3$ at the door and 2.86 $\mu\text{g}/\text{m}^3$ at a point 15 feet away. Here the area air sampling for metals was yet another indicator of the success in improving the integrity of the Equipment Door's seal. A possibly unrelated, yet still interesting, question is brought up with the information in Table 13 as to why only iron was seen when aluminum concentrations within the chamber dust were at a concentration of at least three times higher than iron for SQTS 05. It has been theorized that this may be indicative of hot, high pressured air being forced across the mild steel deflection barriers and not necessarily reflecting a leak of the dust from within the chamber.

Personal measurements for airborne metals, reflected in Table 11, were taken as part of the re-entry procedures into the personnel door and vestibule during SQTS 04 and 05. It should be noted that the information presented in Table 10 reflects area, non-personal, sampling and Table 11 presents personal sampling results of employees performing the re-entry procedures for SQTS 04 (57 and 55 minute sampling period) and 05 (57 and 56 minute sampling period).

Taken from the information presented in Table 11, Employee 1 performed the entry into the chamber area while Employee 2 remained in the vestibule area during the entire sampling period, never entering the chamber. The mean personal exposure level for aluminum was $50.9 \mu\text{g}/\text{m}^3$ during the sampling periods for SQTS 04 and 05. This exposure level is 255 times the beryllium Action Level and covers initial entry procedures only. Of greater interest is Employee 2 personal sampling results during SQTS 04. This individual obtained a personal sample result for manganese at $0.34 \mu\text{g}/\text{m}^3$ within the vestibule. Although manganese is test constituent at only trace levels, this is the only personal metals exposure within the range of the beryllium Action Level.

5.4 Combining Metals Analysis With Leakage Potential

Combining metals analysis and leakage results¹ indicates the potential for a problematic personal exposure to metals external to the chamber unless modifications are made. Previous carbon monoxide monitoring indicates a leak potential at five locations: Camera Room, Short Chase, Long Chase, Equipment Door, and Personnel Door.. Metals analysis results show that virtually all metals within the chamber, even trace levels as alloys or on the chamber surfaces, can be accounted for in the dust analyses and therefore may be present wherever leaks may occur as both airborne and surface particulate. This consideration expands the potential for exposures to include the pre-filter staging area during the removal and replacement of used pre-filters. Leaks from either the Equipment Door or the Personnel Door may also complicate re-entry and retrieval protocol in the attempt to control personal exposure levels and surface contamination control within the High Bay area as well.

Preliminary estimates have been made that attempt to link personal sampling information to metal concentrations within the bulk dust samples for an individual test. There are no protection factors used, only the use of minimum metal concentrations from Table 1 relative to the highest personal sampling results from Table 11. Results for personal exposure to copper from SQTS 04 were compared to the lowest bulk dust concentration during the same test with the intention of achieving a personal exposure level at the Be Action Level without the consideration an 8-hour Time Weighted Average. This rough estimate also presumes that 50% of the mass within the CFF chamber is not completely consumed. Within these guidelines, approximately 10 milligrams of beryllium per each Kg of mass within the chamber may lead to exposures during personnel door re-entry procedures above the $0.2 \mu\text{g}/\text{m}^3$ Action Level. Similar considerations should also be made for areas external to the chamber where previous carbon monoxide monitoring data indicates a leak potential: Camera Room, Short Chase, Long Chase, Equipment Door, and Personnel Door.

6.0 Recommendations

- 2) **Particle Size Selective Data Collection.** There is a need to continue this metals analysis protocol and extend it into future tests at the CFF. Metals analysis should continue for all tests, however most important is the need to begin developing a data set for various levels of beryllium-related activities. This is best performed in conjunction with standard Industrial Hygiene sampling protocol. This will afford the ability to correlate personal exposure potential to the dynamics of a particular testing protocol and the inclusion of highly toxic materials as part of the experimental assembly.
- 3) **Equipment Door and Personnel Door Considerations.** Reduction in the leak rates at the Equipment Door were seen, and corroborated by an area metals sampling protocol, after efforts had been made to improve the Equipment Door's integrity and seal. The combined metals and carbon monoxide monitoring results support the recommendation that efforts to improve the seals at the Equipment Door should be continued to reduce the potential for leaks at these entry portals. Efforts should also be made for the Personnel Door to control the personal exposure potential in the vestibule. Further, it is recommended to install or connect a HEPA-filtered exhaust ventilation system for use within the vestibule area. This will also assist in maintaining a negative pressure for the 3-stage decontamination unit, sealed in place at the entry into the vestibule, that will be necessary for maintaining appropriate housekeeping during beryllium-related activities within the chamber.
- 4) **Potential for Airborne and Surface Contamination.** Previous carbon monoxide monitoring indicates a leak potential at five locations. The metals analysis results that show the potential for exposure outside of the chamber should also include the pre-filter staging area during the removal and replacement of used pre-filters. Metals analysis results show that virtually all metals within the chamber, even trace levels as alloys or on the chamber surfaces, can be accounted for in the dust analyses and therefore may be present wherever leaks may occur as both airborne particulate and surface contamination. Preliminary estimates based on existing SQTS data indicate that approximately 10 milligrams of beryllium per each kilogram of mass within the chamber may lead to personal exposures, during personnel door re-entry procedures, above the $0.2 \mu\text{g}/\text{m}^3$ Action Level for beryllium. The current re-entry procedures are only a preliminary indication of the potential for exposure during normal re-entry protocol that will include the disturbance of the chamber's dusty environment to retrieve experimental equipment before the water wash-down occurs. Of great interest is the personal sample result for manganese at $0.34 \mu\text{g}/\text{m}^3$ within the vestibule. Although manganese is a test constituent at only trace levels, this is the only personal sampling exposure for metals within the range of the beryllium Action Level of $0.2 \mu\text{g}/\text{m}^3$.

7.0 References

- 1) Zalk, D., Non-Occupational Industrial Hygiene Monitoring Results for the Structural Qualification Test Series 01 - 05. May 30, 2001.
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- 3) Zalk, D., Air monitoring during re-entry procedures for Structural Qualification Test Series 02. May 8, 2001
- 4) Zalk, D., Air monitoring during re-entry procedures for Structural Qualification Test Series 03. May 14, 2001.
- 5) Zalk, D., Air monitoring during re-entry procedures for Structural Qualification Test Series 04. May 17, 2001.
- 6) Zalk, D., Air monitoring during re-entry procedures for Structural Qualification Test Series 05. May 20, 2001
- 7) ACGIH Threshold Limit Values for Chemical Substances, 2001
- 8) Hinds, W.C., Aerosol Technology: Properties, Behavior, and Measurement of Airborne Particles. John Wiley and Sons, New York, 1982.