Gravimetric Determination

Method no.: PV2121

Target concentration: Table 1 of Appendix A

Procedure: Samples are collected by drawing a known volume of air through tared 37-mm diameter low-ash polyvinyl chloride (PVC). Samples are dried and weighed in a special climate-controlled room.

Recommended sampling time and sampling rate: 480 min at 2.0 L/min (960 L)

Reliable quantitation limit: 66 µg

Status of Method: Partially evaluated method. This method has been subjected to established evaluation procedures of the Methods Development Team and is presented for information and trial use.

Date: March 2003   Team Effort

Methods Development Team
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Purpose: To define the procedure to prepare pre-weighed filters for use by OSHA IHs and to describe the procedure to analyze the filters after they have been returned to the Salt Lake Technical Center (SLTC) for analysis.

Scope: This standard operating procedure (SOP) applies to all analysts that are assigned to the Gravimetric work station or are involved in preparing the filters for use.

Procedure: The following describes the step-by-step SOP for weighing filters using the OSHA Automated Weighing System. The filter media is 37-mm diameter low-ash polyvinyl chloride (PVC). These preweighed filters are used by the field compliance officers for the collection of respirable and nonrespirable particulate material. These PVC filters in their "flying saucer" type cassette are exclusively recommended for respirable sampling. An abstract of a memorandum that describes their use in the field is attached. (Appendix A) The sealed cassettes with the preweighed and numbered filters are shipped from the SLTC.
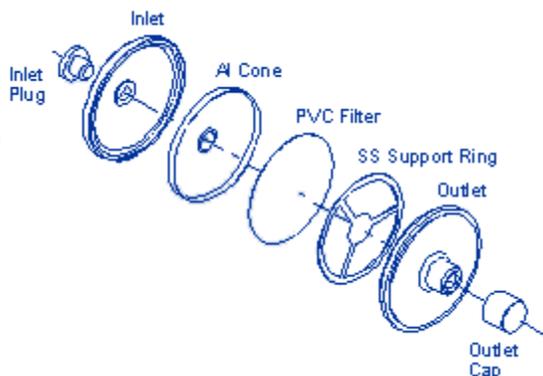
The automated weighing system comprises an environment-controlled chamber, drying ovens, analytical balance, two automated Zymark robotic systems, and associated computer and network connections. The Automated Weighing System SOP describes the proper calibration and use of this instrumentation. These procedures apply in both the preweighing for a tare weight of the filter and the final weighing to determine total particulate weight.

1. Only authorized laboratory personnel are allowed in the weighing area. Lock the weighing room after business hours and when not in use. Challenge any non-laboratory worker entering the weighing area without an escort.

2. The automatic weighing program which utilizes robotics is described in Appendix B. Appendix B outlines the sequence of events the robot performs to weigh the filters. Additionally, the software commands that control the robot are listed. Analysts should refer to Appendix B for details of the robot's operation.

3. The filters are weighed in a laboratory environmental controlled room with the temperature being controlled to 21 ± 1ºC (70 ± 2ºF) and relative humidity to 35 ± 5%. Deviations from these parameters must be addressed before the weighing of filters. Consult your supervisor or arrange for repair services to correct the problem.

4. Strictly comply with good laboratory practices with no smoking or eating in the weighing room or laboratory. Before handling filters or gravimetric samples, work areas should be cleaned with damp towel and adsorbent pads placed on the bench top. Handle all filters with tweezers or a gloved hand. Preweighed filters are not handled with bare hands.

5. The PVC filter assemblies are purchased from MSA, Inc. and are packaged in a plastic sleeve containing 40 assemblies. The filter assemblies are removed from the sleeves and a bar code is affixed to the aluminum cone. The assemblies are place on a 75-position weighing rack. The filters will remain on this rack for the entire weighing process. Desiccate all filters in a vacuum oven before pre-weighing. The initial desiccation is under a vacuum (-15 inHg) at 60ºC using a dry air bleed for a total of 36 hours. Subsequent

desiccations are under a vacuum (-15 inHg) at 40ºC using a dry air bleed for 15 hours. A minimum of two desiccations is required. All assemblies must have two consecutive weights that are ±10 μg of each other.

6. Change desiccant well before it is depleted (e.g., completely changes color - blue desiccant turns pink). Depending on the local relative humidity, this might be required several times a week or only weekly. A filter is used to clean air metered into the vacuum oven when releasing the vacuum. Change this filter as needed or annually. Release the vacuum to the oven in a controlled and gradual manner.

7. The pre-weighed assemblies are returned to the plastic sleeves with blue filter spacers separating each assembly and shipped to MSA, Inc. MSA places each PVC filter assembly in a polystyrene cassette. Each cassette is sealed with shrink-wrap tape and flow tested. The cassettes are then returned to the SLTC. Six cassettes are selected at random from the lot of returned cassettes to be analyzed. After going through the desiccation procedure, the difference in pre-weights and post-weights is checked for outliers.

8. Use the following protocol when receiving and logging in gravimetric filter cassette samples. Use the exclusive bar code number affixed to the filter cassette as the sample submission number. Replace another sample submission number with the bar code number by lining it out and initialing it. Record the changes on the OSHA 91A and include a note on the OSHA 91B analysis report form. Use the following statement in the note. "*When requesting gravimetric analysis, please use the affixed filter bar code number as the sample submission number*." If the bar code number does not appear on the OSHA 91A, include in a note that "xxx" submission number corresponds to "yyy" bar code. The set of laboratory numbers is attached to the sample cassette for gravimetric analysis. Stamp the numbers on the OSHA 91A as the samples are originally logged in. After the gravimetric analysis has been completed, return the filters in the labeled petri dishes to the proper drawer in the sample room for the additional analysis.

9. After verifying and recording the condition of seals and number agreement, remove filters from their cassettes and place on a 75-position weighing rack. Desiccate all filters (except oil mist, CrO3 or samples with the potential to lose volatile organic compounds) in a vacuum oven before pre- and post- weighing. The initial desiccation is under a vacuum (-15 inHg) at 40ºC using a dry air bleed for a total of 36 hours. Subsequent desiccations are under a vacuum (-15 inHg) at 40ºC using a dry air bleed for 15 hours. A minimum of two desiccations is required. Change desiccant well before it is depleted (e.g., completely changes color - blue desiccant turns pink). Depending on the local relative humidity, this might be required several times a week or only weekly. A filter is used to clean air metered into the vacuum oven when releasing the vacuum. Change this filter as needed or annually. Release the vacuum to the oven in a controlled and gradual manner.

10. Use an electronic balance capable of weighing to the nearest 0.001 mg. Record the test weights, temperature and humidity in the appropriate notebook daily before using the balance to weigh filters. After weighing the test weights, press **SELECT 1** twice on the robot's balance. The balance is automatically zeroed prior to each weighing. The balance should be checked by a qualified service technician **every twelve months** or when performance is not acceptable.

11. Check the calibration of the balance annually with National Institute of Standard and Technology Class S-1.1, ASTM Class 1 weights or equivalent. Use one of the NIST-traceable weights stationed at the balance. Record its weight into a logbook. One or more other weights - 1- to 1000-mg weights, which are also NIST-traceable - are used to check the balance and the measured values are documented in the annual balance audit.

12. Weigh the filters using the protocols described in Appendix C. Equilibrate filters in the environmental controlled room for six hours prior to **each** weighing. Use extreme care to avoid spilling loose dust from the cassette assembly.

13. Weigh all filters *at least twice*.

a. Differences greater than ±10 µg in the two weights require a third desiccation. If the filter has gained significant weight (e.g., >0.2 mg), it must be reweighed until the weights agree within five percent of the total weight.

b. A weight of <66 µg can be reported as "*None Detected, Reporting Limit = 66 micrograms*."

14. Analyze (weigh) a set of quality control samples with each analytical set. Quality control samples should be treated in the exact same manner as the compliance samples. Report and obtain quality control results. Acceptable QC results must be within the control limits established before the compliance samples can be reported. Complete a QC Corrective Action Sheet for any outliers and take appropriate steps to remedy the problem.

15. A detailed description of how to perform the calculations using the **GRAV** (Excel) software is given in Appendix C. Calculations are based on taking the difference between a pre- and post- weights in milligrams. Subtract a positive weight for the blank from the net weight. Then divide the results by an air volume in cubic meters to obtain a particulate concentration in air. **DO NOT** add negative weights of the blank to the gravimetric weight. These calculation conventions are included in the **GRAV** program.

16. Report the results of the gravimetric analysis using the following IMIS (analyte) codes. Also report any detected blank values. Additional codes can be found in Appendix A. Report gravimetric results using the conventions below and with these two IMIS codes:

G302 Sample Weight and blanks reported in mg (Y)

G301 Gravimetric Analysis reported in mg/m^3 (M)

Report additional results based on the gravimetric determination using the appropriate IMIS codes listed and report in mg/m^3 (M):

IMIS	Analyte		IMIS	Analyte
			P104	Portland Cement
9130	Particles Not Otherwise Regulated (Respirable Fraction <1% Silica)		W103	Wood Dust (General)
			9210	Hardwood Dust
9135	Particles Not Otherwise Regulated (Total Dust)		9211	Softwood Dust
			C120	C120 Coal Dust (>5% Silica)
5010	Oil Mist		9040	9040 Coal Dust (<5% Silica)
1300	Glass Fibrous or Dust		A100	Aluminum Dust

Report additional results based on the determination using other analytical techniques (e.g., XRD, UV-Vis spectrophotometer, AAS). The IMIS codes listed are for the more frequently requested additional analysis:

IMIS	Analyte	IMIS	Analyte
9010	Respirable Quartz	S103	Nonrespirable Quartz
		Z102	Zinc Oxide

Care should be taken to report appropriate IMIS codes based on whether the sample is respirable or nonrespirable. Size determines respirable vs. nonrespirable particles. Respirable dust particles are those that are small enough to enter the gas exchange area of the human lung. For OSHA compliance purposes, these respirable particles are collected using a 10-mm nylon cyclone where the mean size collected is approximately 3.5 µm. For practical purposes in reporting gravimetric results, when the sample has been collected using a cyclone at a sampling rate of 1.7 L/min, the sample is respirable. Respirable PEL standards often include a limitation that the dust does not contain silica in certain percentage. Consult with your team leader as whether to report this respirable PEL without a silica analysis. Below are current conventions for reporting gravimetric samples using the LISA database. These instructions were drafted to

help acting team leader's understand the appropriate reporting conventions. Our new Oracle database (LISA) should fine-tuned in the future to eliminate many of the current shortcomings.

ALL GRAVIMETRIC SAMPLES REQUIRING SUBSEQUENT CRYSTALLINE SILICA ANALYSIS

a. All samples should be Ok'd, Dated and Released when the gravimetric analysis is reviewed. The samples should be released in the LISA database. The samples and paperwork should be transferred for crystalline silica analysis immediately after the completion of the supervisor review.

b. The gravimetric samples associated with crystalline silica samples should be faxed when the crystalline silica samples are released.

ALL OTHER GRAVIMETRIC SAMPLES

c. All other samples should be Ok'd, Dated and Released when the gravimetric analysis is reviewed.

d. If additional analyses are requested (i.e., ICP, AAS), please make sure that a note is added stating that "*The samples have been resubmitted for 'X' analysis. Results will be reported separately.*"

e. If the gravimetric results yield a result less than the PEL for the additional analysis requested, report the additional analysis at the same time the gravimetric samples are released.

f. These reports should be completed by the analyst who performed the gravimetric analysis. Each IMIS analyte must be reported using the gravimetric results and a "<" symbol. A note should be included on the FM91B stating that: "*The gravimetric analysis yielded an air concentration that was less than the PEL. The samples will not be analyzed unless special circumstances exist and the CSHO calls the SLTC.*"

g. If the additional analyte was for mist and a bulk was submitted, it should be also reported at this time with the following note: "*The gravimetric analysis for the air samples yielded an air concentration that was less than the PEL. The bulk sample will not be analyzed.*"

h. If it appears that the CSHO only used the preweighed filters because they were convenient, and a weight was not required, please include a note with the gravimetric analysis that "*Preweighed filter should only be used when a gravimetric analysis is required.*"

18. Confidence limits for the gravimetric analysis are defined by the reporting of a sampling and analytical error (SAE). The sampling or pump error is assumed to be ±5% and the analytical error is derived by analyzing single blind Quality Control samples. A group of 100 data points is necessary to establish the analytical error. The current SAE for gravimetric analysis is 0.083 (as of 3/24/03) and covers the range of 1.0 – 5.0 mg. This gravimetric SAE will be reported for respirable and nonrespirable PNOR (IMIS 9130 and 9135) and other results based solely on a gravimetric determination. The quartz results (IMIS 9010) have a separate SAE, and this number should be referenced for that analysis.

Appendix A
A Description of the Automatic Weighing Field Program

The SLTC is providing preweighed filters for gravimetric analysis. These filters should reduce sample preparation time by CSHOs in the field because the filters are weighed at SLTC and shipped to the field assembled and ready for use in inspections. **The filters will then be returned to SLTC for gravimetric analysis.** Because these filter cassettes are somewhat different from those currently used, the SLTC has prepared these instructions.

The filter media is a 37-mm diameter, low-ash polyvinyl chloride. These filters should be used for silica (quartz) analysis, aluminum and any other appropriate substance having a high PEL or requiring gravimetric analysis. The filters may be used without the cyclone attached for total dust analyses. **Please indicate on the 91A form all analytes of interest.** If gravimetric analysis gives a result less than the PEL for the requested substance(s), no further analysis will be provided unless specifically requested. (*The unassembled filters cost more than five dollars, so* ***they should only be used for samples requiring crystalline silica gravimetric analysis or gravimetric analysis for substances having high PELs*** *which may require no further analysis based on total weight.*) Attached is a partial listing of substances that can be submitted to the SLTC on the pre weighed cassettes.

These filters are shipped pre weighed and assembled in the cassettes. They are available directly from the SLTC. They may also be ordered through the Cincinnati Technical Center (CTC) "Expendables Supplies" menu in the "OSHA Property Management Inventory System (OPMIS)." (Listed as Cassette, Aerosol, 37MM, Preassembled, **Preweighed at SLTC**, #FES0000161). The SLTC initially planned to distribute a supply of filters to each area office. The demand for pre weighed filters has overwhelmed our capability to send the initial supply of preweighed filters every office and replenish them automatically. Currently the SLTC is distributing preweighed filters to the area offices by request only. Any area office that has not received an initial supply of the filters should contact the SLTC or CTC. Preweighed filter supplies will be replenished upon request.

Be sure to follow all appropriate protocols for calibration, sampling and submission of samples. A blank should be included with every set of samples. Due to the smaller size of the sampler, check frequently to prevent filter overloading. This can be accomplished by looking into the inlet sampling port of the cassette or by visual observation of the airborne dust concentration around the worker.

The SLTC is now shipping a modified version of the pre-weighed cassettes. Externally these cassettes are identical to those previously shipped. The only difference is that new cassettes have a stainless steel filter support instead of the Tyvek backup pad on the older style. The stainless steel support seems to be less affected by humidity (providing a more stable blank weight) than the old pad. Earlier versions of the cassette may have a numbered sticker on the outlet side of the cassette, it can be ignored.

It is not necessary to discard the older style filters. Both styles may even be used in a single inspection. However, **it is very important that the blanks submitted with a set of samples have the same backup pad as the samples.** Therefore if both styles of filters are used in one inspection, submit blanks of each style. This will enable the SLTC to perform the correct blank subtraction for the sampled filters.

The inlet side of the cassette is marked on the polystyrene cassette. This is the side of the filter cassette with the metallic antistatic shield. The Tyvek backup pad or the stainless steel support is visible from the outlet side of the assembly. Each of the filter assemblies is bar coded for weighing purposes. To aid in the tracking of the filters, **please use the bar code number for the sample submission number when filling out OSHA Form 91A.**

Inlet   Outlet   New Stainless support (drawing)

These filters were created for use in Mine Safety and Health Administration inspections by MSA, Inc. They are therefore more readily adaptable for use in the MSA style cyclone assembly. However, the standard MSA coupler (used with a standard 2- or 3-piece cassette) will not fit these cassettes. They need a coupler available from MSA (part #457391, price $7.60). This new coupler is plastic instead of stainless steel. These couplers are also available through the CTC OPMIS.
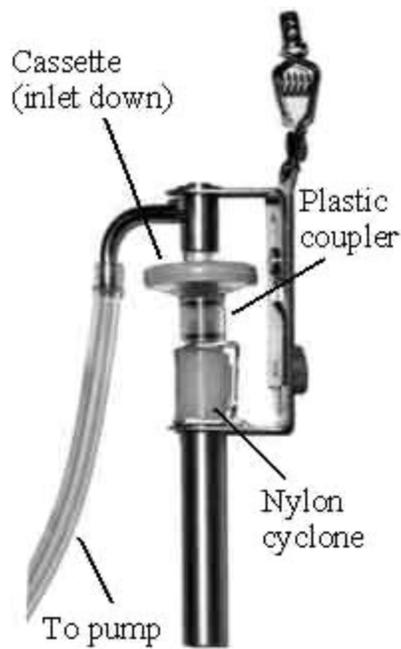
MSA cyclone holder with cassette   Bendix holder with cassette

If you have any further questions about the filters or the autoweighing program, please contact Fred Cox (801-524-7932) or Steven Edwards (801-524-7952).

Table 1
PARTIAL LIST OF SUBSTANCES FOR AUTOWEIGHING SUBMISSION

Analyte	IMIS	mg/m³
α-Alumina		
Total dust	0160	15
Aluminum Metal (as Al)		
Total Dust	A100	15
Respirable fraction	A110	5
Ammonium Sulfamate		
Total dust	0185	15
Respirable fraction	A111	5
Barium Sulfate	B101	15
Total dust	B104	5
Bismuth Telluride, undoped		
Total dust	0370	15
Respirable Fraction	B110	5
Boron Oxide		
Total dust	0380	15
Calcium Carbonate		

Total dust	0505	5
Respirable fraction	C130	5
Calcium Hydroxide		
Total dust		15
Respirable fraction		5
Calcium Oxide	0520	5
Calcium Silicate		
Total dust	C112	15
Respirable fraction	C122	5
Calcium Sulfate		
Total dust	C104	15
Respirable fraction	C123	5
Carbon Black	0527	3.5
Cellulose		
Total dust	0575	15
Respirable fraction	C124	5
Coal dust (<5% Sio$_2$)		
Respirable fraction	9040	
Coal dust (>5% SiO$_2$)		
Respirable fraction	C120	
Cotton dust (The SLTC will supply special preweighed cotton dust filters and cassettes on request.)		
Dicyclopentadienyl Iron		
Total dust	0904	15
Respirable fraction	D100	5
Emery		
Total dust	1016	15
Respirable fraction	E102	5
Grain dust		
(oat, wheat, barley)	G109	10
Glycerin (mist)		
Total dust	1363	15
Respirable fraction	G115	5
Graphite, synthetic		
Total dust	1366	15
Respirable fraction	G100	5
Gypsum		
Total dust	1367	15
Respirable fraction	G101	5
Kaolin		
Total dust	1568	15
Respirable fraction	K100	5
Limestone		
Total dust	1593	15

Respirable fraction	L100	5
Magnesite		
Total dust	M113	15
Respirable fraction	1615	5
Magnesium Oxide fume		
Total particulate	1610	15
Marble		
Total dust	1626	15
Respirable fraction	M114	5
Oil Mist(Mineral)	5010	5
Particulates not otherwise regulated (PNOR)		
Total dust	9135	15
Respirable fraction	9130	5
Pentaerythritol		
Total dust	1987	15
Respirable fraction	P157	5
Perlite		
Total dust	2035	15
Respirable fraction	P101	5
Plaster of Paris		
Total dust	2127	15
Respirable fraction	P102	5
Portland cement		
Total dust	0557	15
Respirable fraction	P104	5
Rouge		
Total dust	2229	15
Respirable fraction	R102	5
Silica, amorphous, precipitated and gel	9050	
diacotomaceous earth, containing <1% crystalline silica	S112	
Silica, crystalline		
Cristobalite,		
Respirable dust	9015	
Quartz		
Respirable dust	9010	
Tripoli, (as quartz)		
Respirable dust	S114	
Tridymite,		
Respirable dust	9017	
Silica, fused		
Respirable dust	9013	
Silicates (<1% crystalline silica)		
Mica,		

Respirable dust	9075	
Soapstone,		
Total dust	9025	
Respirable dust	S121	
Silicon		
Total dust	2235	15
Talc (containing no asbestos)		
Respirable dust	9030	

Appendix B

A description of the robot and software routines for Automated Gravimetric Analysis of samples.

The robotic systems used for the automated weighing of filters was obtained from Zymark Corporation, Hopkinton, MA. It consists of eight major components:

1) Zymark Zymate XP robot to manipulate the filters

2) turntable for storing up to 600 filters

3) bar code reader and spinner

4) Mettler MX-5 balance or equivalent, capable of weighing to 1 µg

5) Zymark System V controller

6) printer

7) drying ovens

8) Microsoft Windows-based computer for data viewing, storage and networking

The Zymark System V controller maintains all the software for the robot operation and gives commands to the other components of the robotic system as needed for the efficient gravimetric analysis of the filters. It also tells the robot where the various components are aligned and allows for periodic adjustment of the components as needed. The PC is used for input and output from the System V controller. The controller appears as a window in the Windows operating screens titled "System V Terminal." This window may be opened by double clicking the icon "System V Terminal" in the "Zymate Utilities" program group.

Old Robot Instructions

The commands for the gravimetric analysis are stored in a *dictionary* in the System V controller and also on the hard drive of the computer. The dictionary used by this system is USD-1.

The two programs most often used from the dictionary are *LOAD.TRAYS* and *WEIGH.* To run a program, enter the name of the program at the **Z>** prompt in the System V Terminal window.

The *LOAD.TRAYS* (also accessible as *LOAD.RACKS*) positions the turntable for easy placement of a filter rack on the turntable. When it is run, it prompts for the desired turntable rack position. Upon entry of a rack position, the turntable moves until the requested position is closest to the edge of the table. CAUTION: Make sure the space around the turntable is clear before entering a position number to avoid possible harm or damage to equipment.

When loading a rack on the turntable, make sure the bracket on the rack fits securely over the brace and the rack is sitting all the way down on the turntable. The program will then prompt for additional positions to be loaded. Enter *N* to exit the program and return to the *Z>* prompt.

The *WEIGH* (or *OSHA.FILTER.WEIGHING*) program is used to analyze a batch of filters. When *WEIGH* is entered at the *Z>* prompt, the program initially asks if the previous data was saved. Enter *Y*. The program will then initialize the robotic system. This procedure takes a few minutes. After the initialization is complete, the program will prompt: *What is the starting TURNTABLE rack number of this batch of filters? (1–8)*. Enter the turntable rack position. **NOTE: this is position of the rack on the turntable, not the number printed on top of the rack!** The program will then prompt: *What is the starting column number of this batch of filters (1–5)?* There are five columns on each rack (numbered 1–5, from left to right.) A batch may start in the topmost position of any column. Enter the starting column number. The program will then prompt: *How many samples are in this batch?* Enter the number of samples to be weighed in this batch. NOTE: A batch may continue into another rack, but may not continue from the rack in turntable position 8 to turntable position 1. The program will then ask for verification of the information about the batch. At this time a filename is created for data storage. The filename consists of the current date and time. The format for the filename is *MMMDDHHM.#* where the month (*M.M.*) is an abbreviation of the current month and the extension (#) is the number of the batch entered. (Example: May12112.2 is the second batch input on May 12 between 11:20 and 11:30 A.M.)

The program will then ask if there are additional batches to be weighed. When all the batches have been entered, the program will verify that there is a disk in the System V controller. The program will then prompt: *Enter the start delay in hours (0, for an immediate start)*. Enter the start delay (the time before the robot starts weighing the filters). Decimal hours are permitted (e.g., 1.25). The program will then prompt: *Enter robot exercise time in minutes (0 for none, 10 max)*. Enter the time for the robot to exercise or warm up (1–3 minutes are recommended). The screen will then show the time remaining until the weighing will begin.

When the robot begins the weighing, it first exercises, then it collects the proper "hand" to manipulate the filters. Before weighing any filter, the controller queries the balance calibration status. The Mettler MX-5 balance monitors its calibration and will recalibrate as necessary. If the query shows the balance needs recalibration, which will take place before the next filter is processed. Once the balance calibration is stable, the robot takes the next filter and checks to make sure it has a filter by placing the filter above a sensor. If it does not sense a filter, it will try again to grab the filter. The robot will try to grab a filter four times before writing to the data file the rack position and the message *No_Filter*. Each time it tries to grab the filter, it reaches a little farther into the rack.

Once the robot grabs the filter, it places it in the bar code reader station to determine the bar code. While in the bar code station, the filter is spun and the reader makes multiple attempts to read the bar code. In order to exit this station, the bar code must agree on two successive readings. If the bar code doesn't agree after three attempts (meaning six readings), the controller will store the bar code as *NOT_READ* and continue.

After reading the bar code, the robot retrieves the filter and moves it to the balance station. In the balance station the filter is held over a Staticmaster to remove the static. The balance is tared and the filter placed on the balance. The controller then asks the balance for a stable weight. Once the stable weight is returned, the filter is retrieved from the balance. (The controller verifies that the filter has been retrieved by querying the balance.) The filter is then placed back in the turntable and the rack position, bar code and weight are written into the data file. The robot then continues until all filters are weighed. Once all filters and all batches have been weighed, the "hand" is parked, the robot shuts down and the controller returns a *Z>* prompt.

New Robot Instructions

The commands for the gravimetric analysis are stored in a ***dictionary*** in the System V controller and also on the hard drive of the computer. The dictionary used by this system is USD-4.

The two programs most often used from the dictionary are **LOAD.TRAYS** and **WEIGH.** To run a program, enter the name of the program at the Z> prompt in the System V Terminal window. If the **Z>** is not visible, flip the toggle switch to the **UP** position or toward the turntable. The toggle switch is on the black box located to the left of the balance controller. If the robot is active, wait until it is finished before flipping the switch. It is also possible to run the **LOAD.TRAYS** program by first clicking on the ABORT button. After adding or removing the filter racks, type

WEIGH and press ENTER to continue.

The **LOAD.TRAYS** (also accessible as **LOAD.RACKS**) positions the turntable for easy placement of a filter rack on the turntable. When it is run, it prompts for the desired turntable rack position. Upon entry of a rack position, the turntable moves until the requested position is closest to the edge of the table. CAUTION: Make sure the space around the turntable is clear before entering a position number to avoid possible harm or damage to equipment. When loading a rack on the turntable, make sure the bracket on the rack fits securely over the brace and the rack is sitting all the way down on the turntable. The program will then prompt for additional positions to be loaded. Enter **N** to exit the program and return to the **Z>** prompt.

The **WEIGH** (or **OSHA.FILTER.WEIGHING**) program is used to analyze a batch of filters. When **WEIGH** is entered at the **Z>** prompt, the program initially asks if the previous data was saved. Enter **Y**. The program will then initialize the robotic system. This procedure takes a few minutes. After the initialization is complete, the program will prompt: **What is the starting TURNTABLE rack number of this batch of filters? (1–8)**. Enter the turntable rack position. **NOTE: this is position of the rack on the turntable, *not* the number printed on top of the rack!** The program will then prompt: **How many samples are in this batch?** Enter the number of samples to be weighed in this batch. NOTE: A batch may continue into another rack, but may not continue from the rack in turntable position 8 to turntable position 1. The program will then ask for verification of the information about the batch. At this time a filename is created for data storage. The filename consists of the current date and time. The format for the filename is **MMMDDHHM.#** where the month (**M.M.**) is an abbreviation of the current month and the extension (#) is the number of the batch entered. (Example: May12112.2 is the second batch input on May 12 between 11:20 and 11:30 A.M.)

The program will then ask if there are additional batches to be weighed. When all the batches have been entered, flip the toggle switch **DOWN** or toward yourself. The robot will move the filters to an available oven. The filters will be desiccated and returned to their original positions in the racks of the turntable. An equilibration time of 6 h is pre-programmed into the robot.

Before weighing any filter, the controller queries the balance calibration status. The Mettler MX-5 balance monitors its calibration and will recalibrate as necessary. If the query shows the balance needs recalibration, which will take place before the next filter is processed. Once the balance calibration is stable, the robot takes the next filter and checks to make sure it has a filter by placing the filter above a sensor. If it does not sense a filter, it will try again to grab the filter. The robot will try to grab a filter four times before writing to the data file the rack position and the message **No_Filter**. Each time it tries to grab the filter, it reaches a little farther into the rack.

Once the robot grabs the filter, it places it in the bar code reader station to determine the bar code. While in the bar code station, the filter is spun and the reader makes multiple attempts to read the bar code. In order to exit this station, the bar code must agree on two successive readings. If the bar code doesn't agree after three attempts (meaning six readings), the controller will store the bar code as **NOT_READ** and continue.

After reading the bar code, the robot retrieves the filter and moves it to the balance station. In the balance station the filter is held over a Staticmaster to remove the static. The balance is tared and the filter placed on the balance. The controller then asks the balance for a stable weight. Once the stable weight is returned, the filter is retrieved from the balance. (The controller verifies that the filter has been retrieved by querying the balance.) The filter is then placed back in the turntable and the rack position, bar code and weight are written into the data file. The robot then continues until all filters are weighed. After the first weighing, all of the filters are returned to the ovens for another desiccation period. All of the filters are reweighed after another equilibration period. After the second weighing, any filter that have a difference of >10 µg between its two weights is returned to the ovens for a third desiccation. The

filters are weighed after a third equilibration time. Once all filters and all batches have been weighed, the "arm" is parked, the robot shuts down and the controller returns a *Z>* prompt.

If you need to open an oven door for some reason, click the ABORT button. Type ***OPEN.FILTER.OVEN.#***, where # is the number of the oven that needs opening. ***CLOSE.FILTER.OVEN.#*** will close the oven when you are done.

Troubleshooting:

The robot may be stopped at any time by clicking on the ***Stop*** button in the System V Terminal window or by pressing ***F9*** on the keyboard. Note: *pressing **Shift F9** should stop the robot immediately. Pressing **F9** may allow the robot to finish the program line that is being processed*. Once the robot has halted, you may be able to continue by clicking the ***Step*** button or the ***Cont*** button on the screen or by pressing ***F9*** to step forward or ***F10*** to continue the program. Once the program has stopped, clicking on the ***Abort*** button or pressing ***Shift F10*** will end the program.

Sometimes the robot needs to have its data arrays reset to default values. This is done from the ***Z>*** by typing ***INIT.SYSTEM***. This is only done after all scheduled weighings are finished and the samples are reported.

If the robot is frozen and not processing samples, power down the entire system. This may be done by shutting off the UPS under the table for a few minutes and then turning it on again. When the controller is rebooted, the dictionary (USD–4) will need to be reloaded from the disk in the System V controller. To do this, click on ***Options*** and ***Load Dictionary*** in the System V Terminal window. Enter the name of the dictionary (USD–4) and click ***OK***. Loading the dictionary will take a few minutes then the process can be restarted.

<div align="center">

Appendix C

Gravimetric Calculations using GRAV (Excel) Software

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1. Double click on the **GRAV** icon. The **GRAV** icon is usually located on your desktop on computers using WindowsNT with the Excel client program installed. Also, the **GRAV** program can be reached with the icon entitled with one of these terms "AA_Weighing" on some computers.

2. Filter Data. *Purpose: Transfers filter weighing data files from the ALPHA to the NTSERVER to enable calculations*.

    a. Select **Weighing Samples** button. From the Main Menu of the **GRAV** program select **Filter Data from Rack**.

    b. A dialog box will appear titled "Form 1". The default drive that should appear is entitled **\\ROBOT\WEIGHING or \\USD4\WEIGHING**. If not, reselect it by clicking on it from the list of choices provided and shown by clicking on the down arrow.

    c. Select the **BIN** subdirectory on the \\ROBOT\WEIGHING or \\USD4\WEIGHING drive, then click on one of your raw data files (e.g., Apr27135.1). Click **OK**.

    d. Select **Used** from the dialog box. **New** is for filters that have been preweighed but not used in the field. Once the data transfer is complete, the Main Menu will reappear.

    e. If you have more than one data file, repeat steps a-d.

3. From the Main Menu select **Sample Range** and then **New Sample Range** in the Project dialog box, up to 100 samples. Type in your sample ranges using common conventions (e.g., R74001–23, R74102, R74111–2, etc.). Enter QC sample ranges with the prefix of **Q** (e.g., Q45600–3). After completing the range enters click **OK**. *Purpose: Identification of sample sets for weighing calculations.*

4. Weighing Calculations. *Purpose: Calculates the weight of material on the filter.*

    a. Select (double click) **Weighing Calculations** from the Main Menu, then click on **Select**.

    b. The dialog box shown lists the sample number, new weights, and used weights for one sample in the sets identified. Select the new and used weights that you want to use in the sample weight calculations. The program defaults to the lowest weights unless you change them. Click **OK** to accept your choices. Select or confirm a choice on the subsequent samples.

    c. When you get to the QC's a second dialog box will appear titled "**Bar**." Enter the bar code (e.g., I490) for the QC sample identified in the Select dialog box, then click **OK**.

5. Generate Report. *Purpose: To print a copy of the new and used weights for each sample and QC with the designation of the chosen weights.*

    a. After the last sample is displayed in 4c above, click on the **Report** button. A window opens with a text file that lists all weights with your selection for each filter chosen in step 4b.

    b. Edit the document file using hard returns, when necessary, to make it easier to be read when printed. Change the printer settings in Notepad to have the left and right margins to be 0.25 inches. Print the file to a networked printer (e.g., \\NTSERVER\XRAYHP4). Close Notepad and return to the Select dialog box.

6. Sample calculations. *Purpose: Calculates the air concentration for all samples using the sample weight and sample air volume.*

    a. Click the **Excel** button in the Select dialog box.

    b. Two workbooks will open up in the Excel program. Select the one titled "MAINMENU.XLS." **Click Here to Run the Weighing Program** begins the procedure to perform the calculation. A third workbook will open up titled "WEIGHT.XLS" and seen as a tab at the bottom of the screen.

    c. Make sure air volumes are correct and fill in "AIR" for QC samples and "ABLNK" for QC blanks. **Click Here to Start** will open a workbook that contains all the sample information along with the individual chosen weights listed and a net weight calculation.

d. Highlight (click and drag the mouse pointer) an individual sample set including the blank from this workbook's column A. **Select Range, then Click Here for Blank Corrections!** to perform the air concentration calculation on that set. Repeat for each set of samples.

e. **To Print and Exit, Click Here!** button gives a printout of the air concentration in milligrams per cubic meter along with the net weight found on each filter in milligrams.

f. Close Excel.

7. After exiting, the Main Menu will reappear. Select **Load Resources to Lisa**. *Purpose: Transfers the data (both weight and air concentration) to Lisa for inclusion in the sample results record.*