Determination of Particle Size Distribution and Chemical Composition of Particulate Matter from Selected Sources in California

Volume III
(Appendices)

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Appendix B
Standard Operating Procedures
Parallel Impactor Sampling Device

STANDARD OPERATING PROCEDURE

OMNI ENVIRONMENTAL SERVICES, INC.

Parallel Impactor Sampling Device (PISD)

rev. 10/87

I. OVERVIEW

A. Physical Description

The PISD (Parallel Impactor Sampling Device) was developed to simultaneously collect particulate samples on 47-mm filter media in four different size fractions. These filters may then be analyzed for chemical composition and results used in receptor modelling (CMB). The PISD was primarily designed for use as a source characterization device, although it may be used for collecting ambient samples as well. The PISD achieves its size fractionation by use of impactors designed to have cut-points at 1.0 μ, 2.5 μ, and 10 μ. It also provides a TSP (total suspended particulate) sample with no impactor. These impactors are simple one-stage impactors which retain by impaction particles greater than the designed cut-point at a nominal 10 lpm; particles less than the design cut-point pass around the impactor stage and are collected on filters. When the sampler is used in situations where a large fraction of the particles are coarse ($>2.5\ \mu$), a preseparator cyclone with a cut-point of 5 μ is placed on the inlets to the 1 μ and 2.5 μ sampling tubes to prevent overloading the impaction plates. A 10-inch tube between the impactor stage and the filter provides for collimation of air flow and for uniform filter deposits.

Filter media used depends on the analysis desired. Teflon membrane filters are used when XRF (x-ray fluorescence spectroscopy) analysis or any analysis in which the carbon in the Teflon is not an interference is desired. Prefired quartz filters are used when carbon or organic analyses are desired. In actual use, two samplers may be operated simultaneously, one loaded with Teflon filters and one loaded with quartz filters, to provide the widest flexibility in analyses of a given airshed or source.

The sampler is shown in Figures 1, 2, and 3; the air flow through the sampler is depicted schematically in Figure 4. The sampler consists of two basic parts: the sampling module and the control module. The sampling module consists of a tripod-supported platform to which are attached four sample inlet tubes, vacuum gauges, a manifold containing four critical orifices for flow control, and a flexible hose connecting the manifold to the control module. The control module contains a rotary vane pump, a cooling fan, an on/off switch, a non-resettable elapsed time meter, and a master vacuum gauge.

Figure 1
PISD OVERVIEW

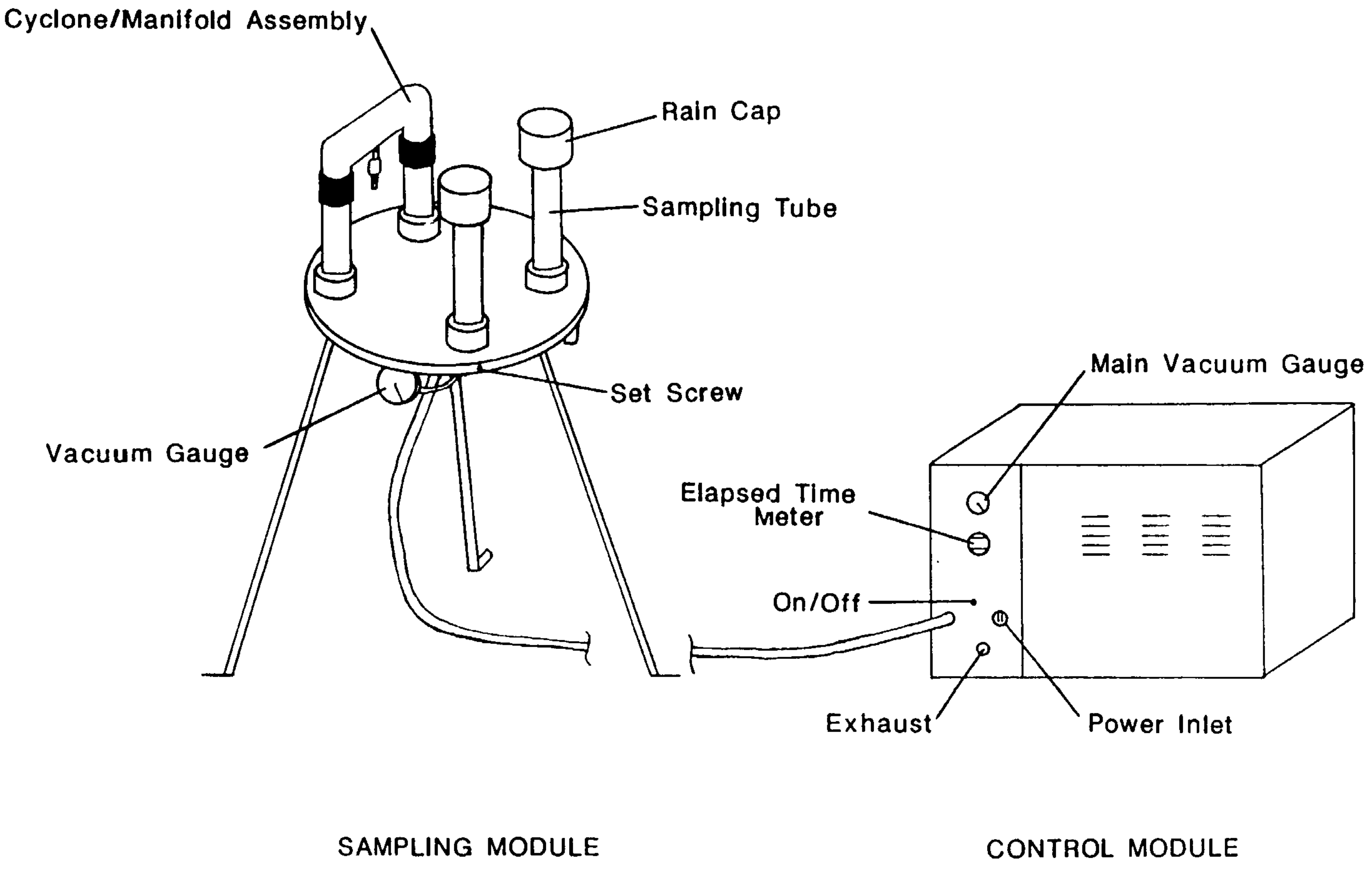


Figure 2

PISD SAMPLING TUBE DETAIL

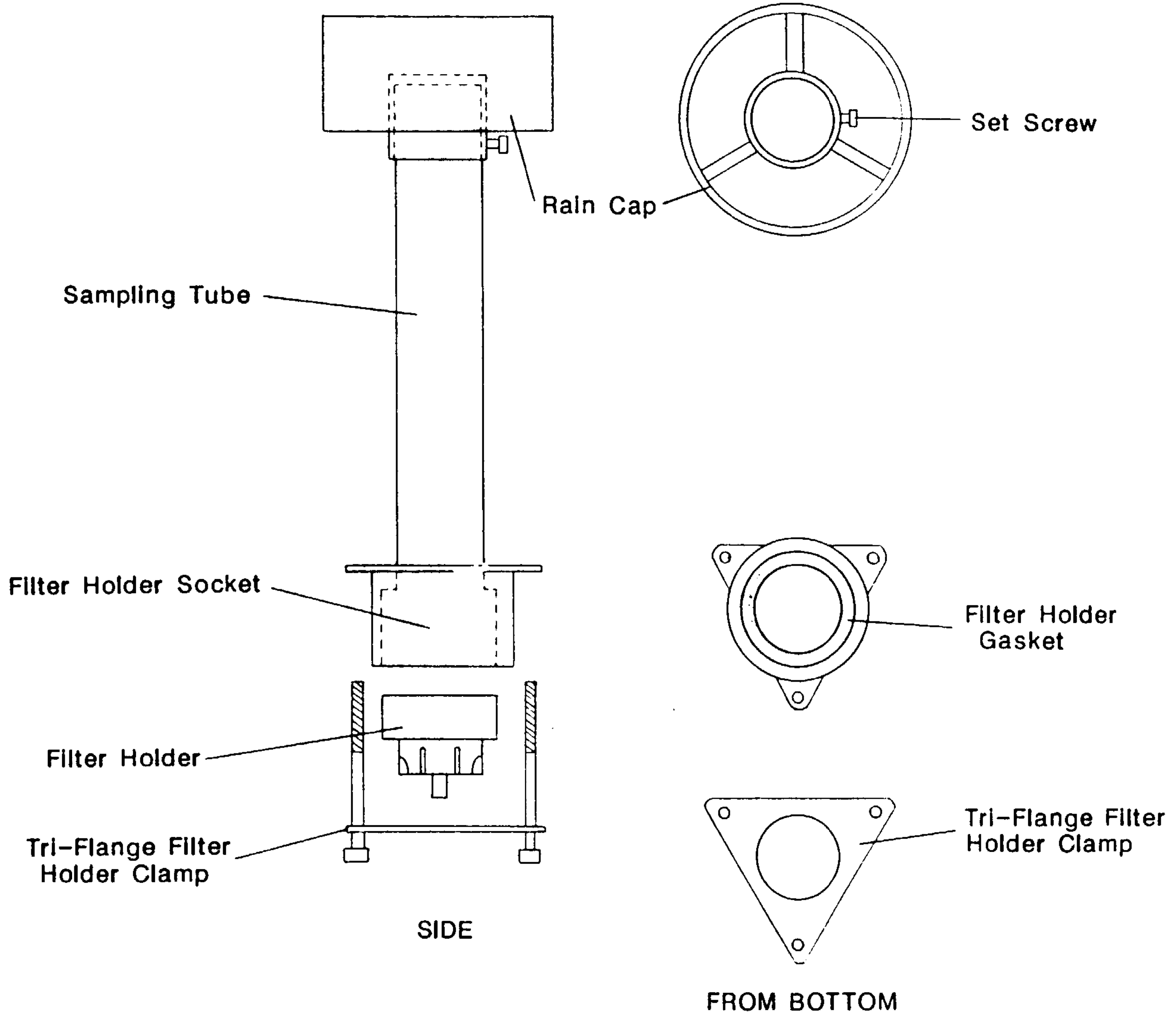


Figure 3

PISD IMPACTOR DETAIL

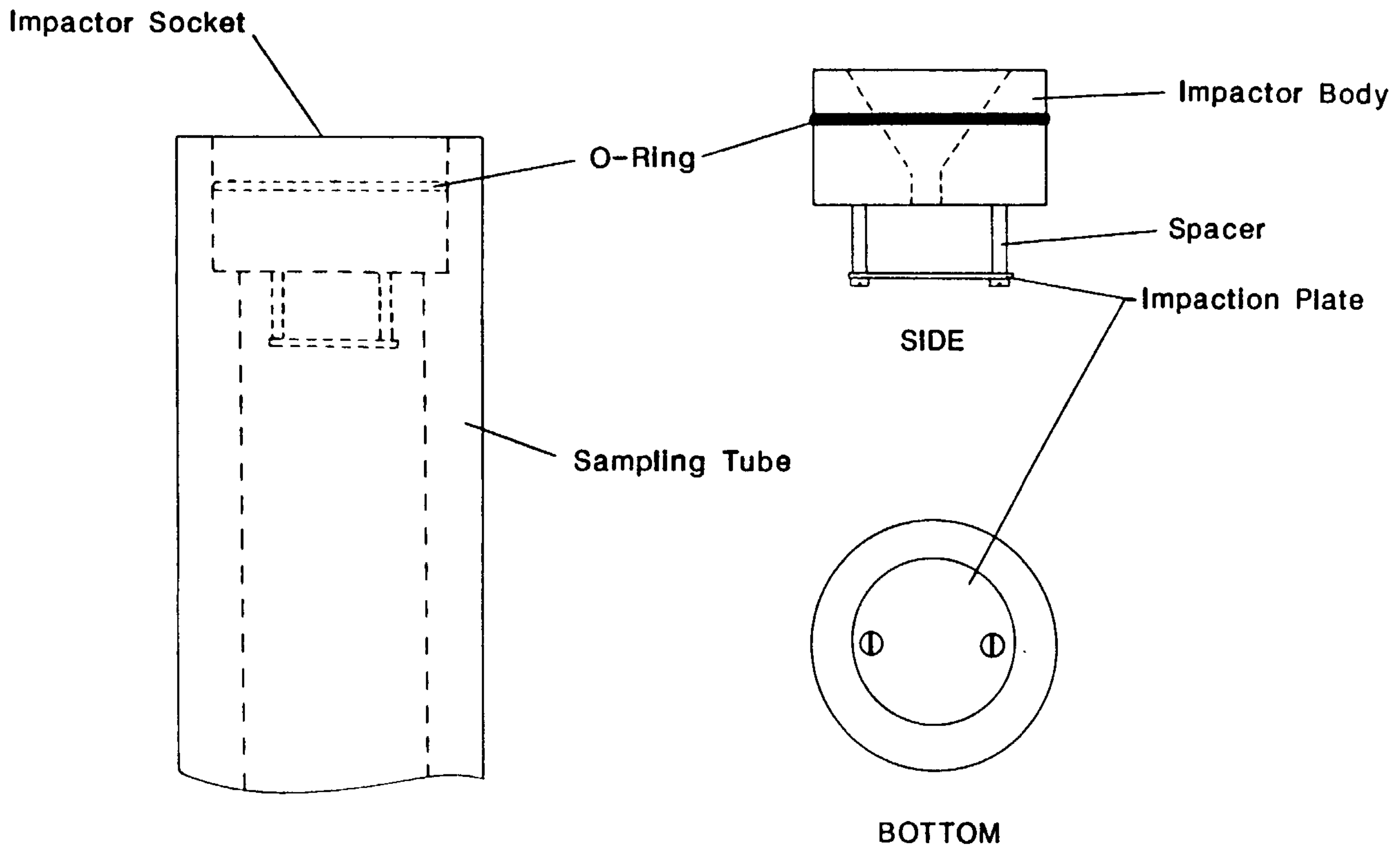
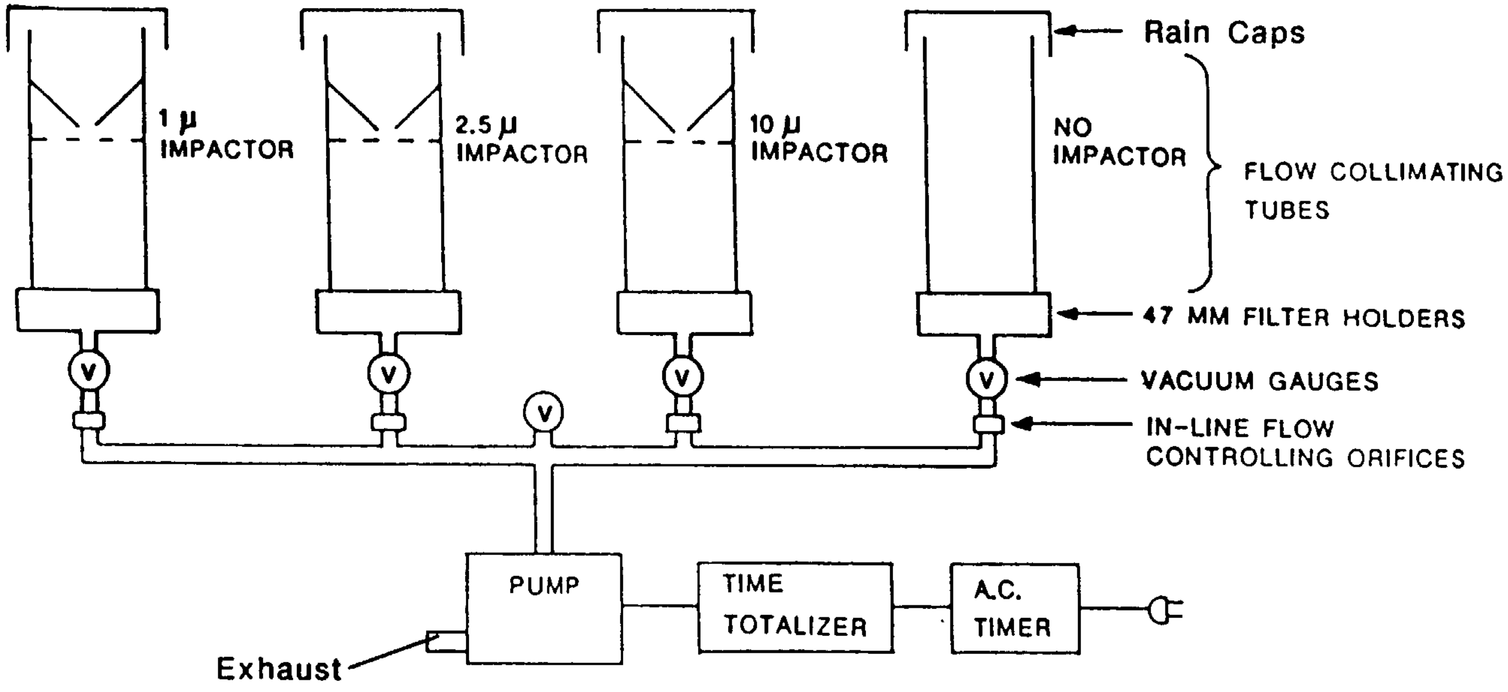


Figure 4

PISD FLOW SCHEMATIC



B. Specifications

Technology	4-port low volume sampler, with single stage impactors for size segregation.
Particle Collection	Four size cuts: TSP, $<10\ \mu$, $<2.5\ \mu$, and $<1\ \mu$.
Flow rates	Maintained by a critical orifice on each port at a nominal 10 lpm.
Accuracy of flow	± 15% of nominal 10 lpm; actual calibrated flow data is in appendix.
Pump	Rotary vane vacuum pump, Gast 1022–V103–G272X, $^{3}/_{4}$ HP.
Elapsed time meter	6 digits, readable to $^{1}/_{10}$ minutes; non-resettable.
Filter media	47 mm; Teflon membrane or quartz recommended.
Connecting tubing	1" OD, $^{3}/_{4}$" ID, average 10 m length between sampling and control modules.
Power required	115 VAC, 60 Hz, 10 amps (6 amps after starting surge).
Dimensions	Sampling platform: circle 56 cm × 15 cm; Tripod legs: 92 cm; Sampling tubes: 31 cm × 5 cm; Control module: 61 cm × 31 cm × 48 cm.
Net weight	Sampling module: 25 kg; Control module: 33 kg.
Total shipping weight	83 kg.

C. Operational Description

When the sampler is operating, air is pulled through a rain cap (or a preseparator cyclone, if used) which sits on top of the sampling tube and which prevents large particle fallout and precipitation from entering the sampler. The air is then pulled through the jet of the impactor (except for the TSP fraction, which has no impactor stage), causing the velocity of the sample stream to increase. As the gas stream exits the jet and flows around the impactor plate, larger particles, by virtue of their large momentum with respect to the viscous drag of the changing direction of air flow, impact on the plate and are effectively removed from the sample flow. The impaction plate is coated with a thin layer of grease to prevent particle bounce ànd re-entrainment into the sample flow. Smaller particles are carried along with the air flow and continue into the sampler. The cut-point of the impactor assemblies is dependent upon the rate of flow of sample gas, upon the physical dimensions of the impactor jet, and the distance between the jet and the impactor flow. In reality, both the aerodynamic size and density of a given particle determines whether it will impact or not, but it is assumed that impactors are primarily size fractionating devices.

The sample gas then flows through a length of aluminum tube to allow the flow to become collimated and uniform and through the filter media on which final particle collection takes place. The filtered gases pass by a vacuum gauge, through a critical orifice for flow control, and into a manifold which is connected by hose to the control module. Each of the four ports on the PISD has a critical orifice allowing a nominal flow rate of 10 lpm. The critical orifice consists of a small opening across which a large vacuum difference is created. With a vacuum difference of greater than 15 inches of mercury the air flow through the orifice reaches sonic velocity and becomes constant. Each of these ports is individually calibrated with a mass flow meter periodically to provide accurate calculations of relative particle sizing to be performed. The vacuum gauges allow the operator to check that sufficient vacuum drop exists across the orifice to allow it to work properly. The vacuum gauges may also be used as rough indicators of filter loading. The gauges on the PISD are oil-filled to allow more steady readings to be obtained and to extend the life of the gauges.

Air flow through the control module initiates at the quick disconnect fitting to which a flexible hose is attached, connecting the pump to the sampling module. Air flows through a filter jar, through the carbon vane vacuum pump, through a muffler jar, and is exhausted through a bulkhead fitting on the front of the control module. An optional exhaust tube may be connected to this bulkhead fitting to direct exhausted gases away from the sampler if sample contamination is a serious concern. The operator should be aware that the vanes in the pump are carbon-based and that small particles of carbon may exist in the exhausted gases. The control unit should be located downwind if possible of the sampling module and as far away as the connected tubing will allow to minimize sample contamination.

In sampling situations where significant quantities of large particles are likely (e.g., sampling resuspended dust), the rain caps on the 2.5 μ and 1 μ sampling tubes are replaced with a single manifold to a cyclone inlet designed to have a cut-point at about 5 μ at a nominal 20 lpm (the combined flow of the two inlets). This cyclone imparts a twist to the air flowing through it, causing larger particles to move to the outer wall within the cyclone and fall to the bottom. The smaller particles (less than 5 μ) are collected from the center of the cyclone and continue into the 1 μ and 2.5 μ sampling tubes. The cyclone, because of its larger reservoir for particles removed from the air stream, serves to prevent overloading of the impactor plates in the 2.5 μ and 1 μ impactor assemblies.

The operator(s) of this equipment should be familiar with the PISD quality assurance document, which addresses the validity of this sampling method and proper techniques to ensure the integrity and validity of the samples collected.

II. MATERIALS AND SUPPLIES

The following items are needed to operate the PISD and to collect filter samples:

- 47 mm filter holders loaded with fresh filters (refer to the Filter Handling and Loading SOP)
- Field data sheets (refer to Figure 5)
- Ballpoint pen with dark blue or black ink
- Apiezon grease, types M and T
- Dow Corning high-vacuum silicone grease
- Small metal spatula
- Kimwipes or other lint-free paper towels
- 95% ethanol in wash bottle
- Impactor removal tool or other large diameter dowel
- Pipe cleaners
- Small screwdriver
- Ziplock or other plastic bags
- Large rubber bands
- Two allen wrenches to fit set screws in rain cap and sampling platform

III. PRE-SAMPLING PREPARATION

A. Disassembly, Cleaning, and Assembly

The PISD sampling tubes must be removed from the sampling platform and cleaned before use. Disassembly proceeds as follows:

- Remove rain cap by loosening set screw and pulling straight up.
- Remove the impactor assembly (except for the TSP tube) by inserting a large wooden dowel through the sampling tube from the bottom and gently pushing upwards. The impactor includes an O-ring seal located midway on the impactor body. Once this is clear of the impactor socket the impactor may be picked off the top of the tube. Take care that the impactor is not pushed out so forcibly that the impactor pops out and falls to the ground.
- Remove the two screws on the bottom of the impactor assembly holding the impaction plate in place. Note that each screw should only be loosened a couple of rotations at a time to avoid cocking the plate and jamming the screw. The plastic spacers, which maintain the correct pre-determined distance between the impactor jet and the impaction plate, will hold the screws to the impaction plate and should not be removed.

Figure 5
PISD Field Data Sheet

Sample Description ____________________
Site/City ____________________
Sampler ID ____________________
Sampler Configuration ____________________
Impactor Grease Type ____________________
Sampling Sequence ____________________
Technician(s) ____________________

TSP	: Filter ID ____________	Filter Type ____________	
10 μ	: Filter ID ____________	Filter Type ____________	
2.5 μ	: Filter ID ____________	Filter Type ____________	
1 μ	: Filter ID ____________	Filter Type ____________	

Resting Vacuum Readings		Leak Check Vacuum Readings	
Pump ________	inches Hg	Pump ________	inches Hg
TSP ________	inches Hg	TSP ________	inches Hg
10 μ ________	inches Hg	10 μ ________	inches Hg
2.5 μ ________	inches Hg	2.5 μ ________	inches Hg
1 μ ________	inches Hg	1 μ ________	inches Hg

Sample Start:

Date/Time ____________________
Elapsed Time Meter ________ minutes

Pump ________ inches Hg
TSP ________ inches Hg
10 μ ________ inches Hg
2.5 μ ________ inches Hg
1 μ ________ inches Hg

Sample Stop:

Date/Time ____________________
Elapsed Time Meter ________ minutes

Pump ________ inches Hg
TSP ________ inches Hg
10 μ ________ inches Hg
2.5 μ ________ inches Hg
1 μ ________ inches Hg

Filter Removal:
Date ____________________
Technician ____________________

Appearance: (color, uniformity, defects)
TSP ____________________
10 μ ____________________
2.5 μ ____________________
1 μ ____________________

Comments (weather, unusual conditions or problems) ____________________
____
____
____
____

- Loosen the set screw in the base of the sampling module and remove the sampling tube.

- Remove the filter holder gasket by pulling out with the fingers; this is simply a friction fit.

The PISD should be cleaned thoroughly with ethanol and Kimwipes, with a particular emphasis on removing all loose particles and deposits from all surfaces in contact with the sample flow. Specifically, the rain caps, impactor assemblies, collimating tubes, filter holder socket, and filter holder gasket should be well-cleaned (refer to Figures 1 and 2). This operation is best done indoors in a relatively dust-free area. Note that ethanol is both toxic and flammable: provide adequate ventilation (a copy of the Material Safety Data Sheet for ethanol is attached to this SOP as an appendix). Care should be taken with the impactors that the jet throats and both sides of the impactor plates are well cleaned. Pipe cleaners will aid in cleaning the jet throats as long as any residue fuzz is rinsed away afterwards. The sampling tube may be cleaned by rinsing with ethanol and dried by pushing wads of lint-free paper towels through the tube with a long dowel or similar device. Do not soak the filter holder gaskets in ethanol for prolonged periods of time.

After all parts have been cleaned and allowed to dry, reassemble the sampling module as follows:

- Place the sampling tube in the socket of the sampling module platform closest to the appropriately labelled vacuum gauge. Each tube is etched with identification as to sampler number and size fraction. The sampler number is etched on the manifold on the bottom of the sampling platform. It is important when several samplers are disassembled and cleaned at once that they are reassembled with their own parts. Lightly tighten the set screw in the platform to hold the sampling tube in place.

- Carefully apply a small dab of Apiezon grease to the wells in the impactor plates using a small metal spatula. Note that two types of Apiezon grease are recommended for use. The type M grease may be used when sampling temperatures are expected to be between 10 to 30 degrees C (50 to 86 degrees F). However, if colder or warmer temperatures are expected, especially during outdoor sampling, use the type T grease, which has a greater working range of 0 to 120 degrees C (32 to 250 degrees F). The danger in using the type M grease in hotter applications is that the it liquifies and will splatter when the sample air is blown into it. The problem with colder applications is that the grease will become so viscous that trapping of large particles is diminished. The type M grease is less expensive than the wider range type T and should be used preferentially when the working temperature is within its range. Apply only enough grease to fill the well.

- Smooth the grease applied by either spreading it even with a razor blade or by heating the impaction plate with a hair dryer or heat gun. Note that if heating is used to even the surface the spatula should be used to overcome the surface tension of the melted grease and completely fill the well. Do not leave excessive grease directly under the impactor jet to avoid grease splatter when the sampler is operated.

- Screw the appropriate impactor plates to the impactor bodies. The sampler ID and size fraction of each plate is etched on the bottom side and should be matched to its appropriate body. Again, tighten the screws in turn only one or two rotations at a time to avoid cocking the impactor plate sideways and jamming the screws. Take care to avoid scraping the metal with the screwdriver and removing particles of metal. The heads of the screws should be wiped with a Kimwipe after assembly to ensure that all loose metal filings have been removed.

- Lightly grease the O-ring with high-vacuum silicone grease on the impactor body to allow easier insertion into the sampling tube.

- Press the appropriate impactor assembly into the impactor socket at the top of the sampling tube. When seated the top of the impactor will be even with the top of the sampling tube.

- Replace the filter holder gasket by pressing it into place in the filter holder socket.

- Slip the threaded half of the tri-flanged filter holder clamp over the sampling tube.

- Replace the rain cap on the top of the sampling tube. Secure by lightly tightening the set screw.

In sampling conditions where large quantities of coarse particles are expected, the cyclone/manifold assembly should be used. This is disassembled by loosening the compression nut holding the cyclone to the manifold, removing the cyclone, removing the reservoir tube from the end of the cyclone, and rinsing all parts thoroughly with ethanol. The black rubber connector sleeves should be rinsed as well. After all parts have dried completely, assemble in the reverse order as disassembly. The compression nut should be tightened only enough to provide firm resistance when the cyclone is twisted. Do not over-tighten this nut as the plastic ferrule will be deformed. The inside of the metal tube at the bottom of the cyclone to which the collection reservoir is attached may be lightly greased with Apiezon grease to enhance removal of large particles.

Very lightly grease the inside of the rubber sleeves using silicon high-vacuum grease to provide some lubrication during assembly. Note that a high-vacuum grease is necessary in that it has a low vapor pressure and will not contaminate samples collected for carbon analysis. Avoid over-greasing these sleeves or a bead of grease will form at the top of the sample tubes as the sleeves are pushed on; this excess grease can cause problems by trapping desired sample. Press the rubber sleeves carefully over the 2.5 μ and 1 μ sampling tubes about half way down. Press the cyclone/manifold assembly into the remaining half of the two sleeves. Again, the cyclones and manifolds are etched with sampler ID numbers, and parts between samplers should not be mixed. Hose clamps on the rubber sleeves are not necessary as there is little vacuum drop at this point in the sampler and the sleeves grip the tubes firmly. The manifold and the sampling tubes should be pushed together as close as possible to minimize exposure of the sample gas to the rubber sleeves and to minimize dead air space.

If the sampler will be moved under dusty conditions or if the sampler will not be used for some time, the impactors may be pressed into the ends of the sampling tubes and both ends of the sampling tube covered with clean plastic bags held in place with rubber bands. Alternatively, the entire sampling tube may be placed in a large plastic bag. The rain caps should be placed in large ziplock bags and sealed. The cyclone/manifold assembly may be sealed with plastic bags at each opening or the entire unit placed in a large plastic bag.

The PISD should be completely disassembled and cleaned before sampling. The sampler need not be completely cleaned if the sampler is in the field and replicate samples from the same source are being obtained. However, in this case the impactors must be removed, the impactor plates cleaned, and fresh Apiezon grease applied to the grease wells. Care must be taken that precipitation or other contamination does not either get in the sampling tubes or on the impactors when unit is partially disassembled.

B. Field Installation

After the appropriate sampling site has been selected, attach the three legs to the sampling platform and secure with set screws. Place the sampling tubes in the sampling platform sockets nearest the appropriately labelled vacuum gauge and secure with set screws. Attach the 1" hose between the sampling module and the control module. As with the other components of the sampler, the hose is labelled with a sampler ID number; this number and the length of the hose is etched on the metal QD (quick-disconnect) fittings at both ends. Attach the QD fittings by pulling back on the silver sleeve, pressing the fitting firmly over the corresponding nipple, and allowing the silver sleeve to pop back into position to lock the fitting. The silver sleeve must return fully to its resting position or the connection is not complete.

Making sure the power switch is off, attach a grounded 110 VAC power cord to the sampling module. Note: the control module is grounded internally; to prevent possibly serious shock hazard, especially when working outdoors, DO NOT use a two wire power source and DO NOT use a three-prong-to-two-prong electrical adapter in the power line. Proper grounding must be provided for safety.

Begin filling in a field data sheet for the sample to be collected (refer to Figures 5 and 6), providing a detailed description of the sample, site, type of sample collected, sampler ID number, impactor grease type (M or T), and technician initials. The space for "Sampler Configuration" should be used to designate either that rain caps are used on the 2.5 μ and 1 μ inlets or that the cyclone/manifold assembly is used. "Sampling Sequence" should be completed as either "continuous" or as "timed" with on/off cycles described.

Remove a previously loaded filter holder (see Filter Loading and Handling SOP) from the field sample case by operating the QD fitting. Record the filter ID and filter type (Teflon or quartz) on the field data sheet; the filter ID number should be found on a sticky label on the bottom of the filter holder. Remove the red cap from the top of the filter holder, place in a clean ziplock bag, and return to the field sample case. Take care that the filter surface is not exposed to precipitation and particle fallout. Make sure that the filter holder ring is snug. Place the filter holder into the filter holder socket at the bottom of the sampling tube and secure with the bottom half of the tri-flange filter holder clamp. Tighten the filter holder clamp evenly and firmly. Repeat this procedure for the three remaining filters.

Leaving the tube between the vacuum gauges and the filter holders disconnected, turn the pump on and allow the sampler to run for two minutes. After two minutes, record the vacuum readings on each of the four sampling module gauges and on the main pump gauge. If all gauges are within 5 inches of mercury of each other, proceed with the next step. If one or more gauges read lower than the others, attempt to find and correct the leak; refer to the troubleshooting section for more information.

Connect the clear plastic tubing from the appropriate vacuum gauge to the filter holder by operating the QD fitting. The sampler is now ready to begin sampling.

IV. SAMPLE COLLECTION

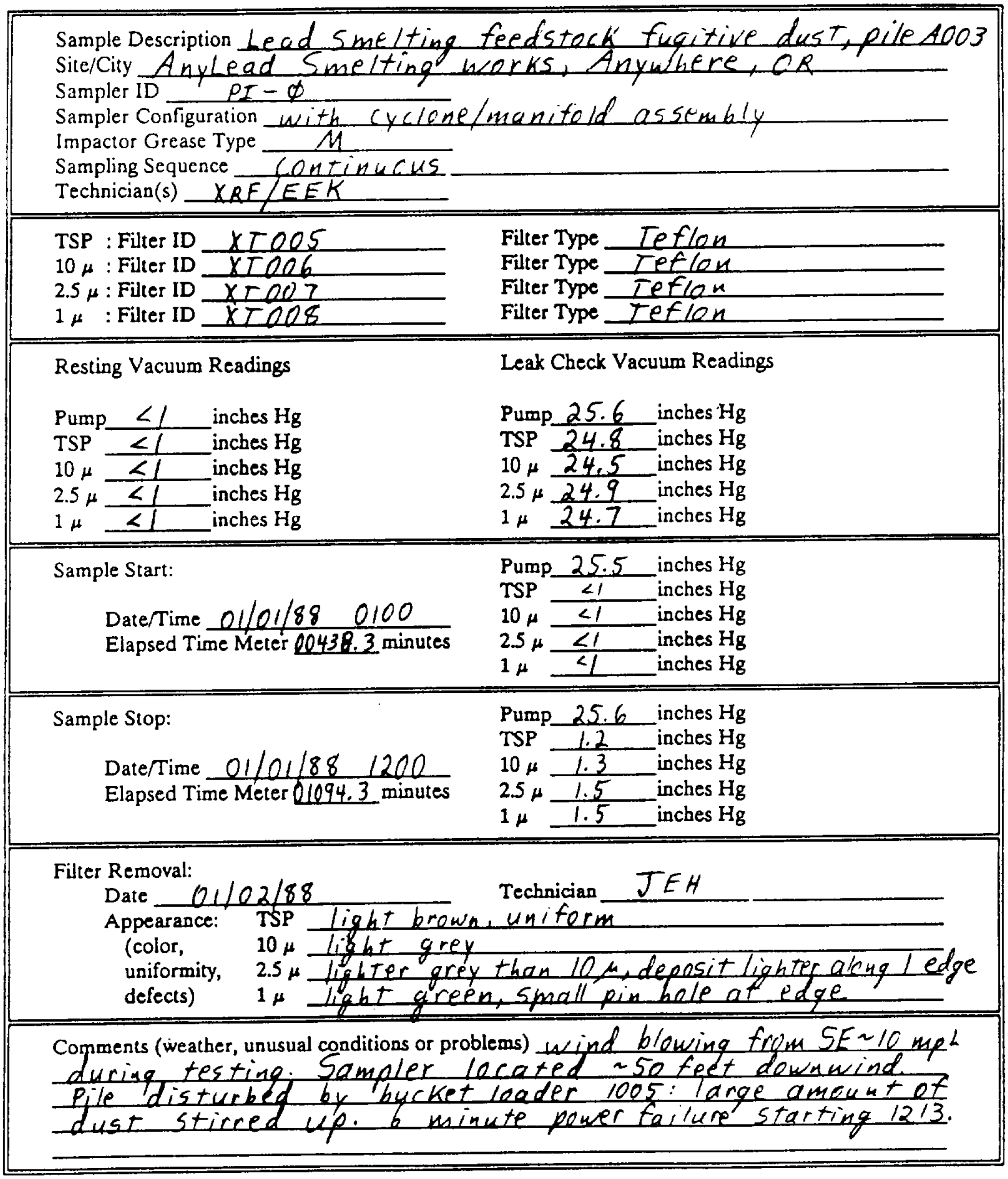
Before sampling begins, record the elapsed time meter reading. These meters are read to the tenths of minutes.

Sample collection is begun by simply turning the pump on or by activating the cycle timer providing power to the pump. If a cycle timer is used, ensure that the control module on/off switch is left in the ON position. Record the sampling start date and time. Also, record the vacuum gauge readings from all five gauges immediately after sampling begins. When the vacuum gauge needles are in the broad bar near zero, record as "< 1". Note that the first calibrated marks begin with 1.0 inches of mercury.

Any unusual problems or events during sampling which might impact the quality or composition of the deposits on the filters should be noted in the comments section at the bottom of the Field Data Sheet.

Optimum loading on the filters for XRF analysis ranges between 0.5 and 3 mg of deposit. Rough estimates as to sampling times to achieve this loading level may be made based on periodic visual inspection of the filters, although the nature of the deposit, the color of the deposit, and the

Figure 6
PISD Field Data Sheet



Sample Description Lead Smelting feedstock fugitive dust, pile A003
Site/City AnyLead Smelting works, Anywhere, OR
Sampler ID PI-0
Sampler Configuration with cyclone/manifold assembly
Impactor Grease Type M
Sampling Sequence Continuous
Technician(s) XRF/EEK

TSP	Filter ID XT005	Filter Type Teflon
10 μ	Filter ID XT006	Filter Type Teflon
2.5 μ	Filter ID XT007	Filter Type Teflon
1 μ	Filter ID XT008	Filter Type Teflon

Resting Vacuum Readings			Leak Check Vacuum Readings		
Pump	<1	inches Hg	Pump	25.6	inches Hg
TSP	<1	inches Hg	TSP	24.8	inches Hg
10 μ	<1	inches Hg	10 μ	24.5	inches Hg
2.5 μ	<1	inches Hg	2.5 μ	24.9	inches Hg
1 μ	<1	inches Hg	1 μ	24.7	inches Hg

Sample Start:
Date/Time 01/01/88 0100
Elapsed Time Meter 00438.3 minutes

Pump	25.5	inches Hg
TSP	<1	inches Hg
10 μ	<1	inches Hg
2.5 μ	<1	inches Hg
1 μ	<1	inches Hg

Sample Stop:
Date/Time 01/01/88 1200
Elapsed Time Meter 01094.3 minutes

Pump	25.6	inches Hg
TSP	1.2	inches Hg
10 μ	1.3	inches Hg
2.5 μ	1.5	inches Hg
1 μ	1.5	inches Hg

Filter Removal:
Date 01/02/88 Technician JEH
Appearance: (color, uniformity, defects)
TSP light brown, uniform
10 μ light grey
2.5 μ lighter grey than 10 μ, deposit lighter along 1 edge
1 μ light green, small pin hole at edge

Comments (weather, unusual conditions or problems) wind blowing from SE ~10 mph during testing. Sampler located ~50 feet downwind. Pile disturbed by bucket loader 1005: large amount of dust stirred up. 6 minute power failure starting 1213.

particular size fraction inspected will affect these visual estimates. Whenever the sampler is turned off to allow interim inspections, the time and duration of the non-sampling period should be noted in the comments section of the Field Data Sheet. Periodic checks of the vacuum gauges during sampling will also provide some rough estimation of filter loading, although it should be kept in mind that the different size fractions will not load at the same rate or produce equal effects on the vacuum readings. If any of the four sample gauges registers more than five inches of mercury or if the difference between any of these four gauges and the main pump vacuum gauge drops below 15 inches of mercury, sampling should be stopped. It is suggested, time and filters permitting, that at least three sets of samples be collected, with two sets bracketing the field-determined optimum sampling time. This increases the chances that optimum loading will occur on at least one set of filters.

V. POST-SAMPLING PROCEDURES

When sampling is completed, record the five vacuum gauge readings and turn off the sampler. Record the stop date and time and the final elapsed time meter reading. If the field technician is not the same person that began sampling, his initials should be recorded at the top of the Field Data Sheet. Remove the tubing from the filter holders by operating the QD fittings, loosen the tri-flange filter holder clamps, and remove the filter holders. Taking particular care that the filter deposit surfaces are not disturbed or contaminated, replace the red caps from the field sample case and return the filter holders to the case. Immediately place small pieces of masking tape or similar marking on the red shipping caps over exposed filters to help keep the field operator from accidentally reusing those filters later in the same day.

If replicate samples of the same source are desired, the sampler need not be completely cleaned between samples. However, the impactors need to be removed and the impactor plates cleaned and re-greased. Refer to Section III.A. for details. New Field Data Sheets must be completed for each set of samples collected.

At the end of all sampling, it is recommended that the sampler be cleaned as soon as possible.

Exposed filters should be removed from the filter holders and placed in Petri slides, preferably in a laboratory in a laminar flow hood. If filter holders must be reused without access to a properly equipped laboratory, filters may be removed in a clean, draft-free area following the Filter Handling and Shipping SOP. In either case, the appearance of the deposits should be noted by the technician on the Field Data Sheet.

Copies of the field data sheets should accompany the filters when they are shipped for analysis.

VI. ABBREVIATED SAMPLING CHECKLIST

- Collect necessary cleaning and assembly supplies and tools.
- Disassemble sampling tubes, impactors, and cyclone/manifold assembly; clean all parts and rain cap thoroughly with ethanol.
- After parts are dry, apply appropriate type of Apiezon grease to impactor plate and smooth either by heating or by trowelling with a razor blade.
- Reassemble impactors; wipe heads of impaction plate screws to remove any loose metal filings which may have resulted from the assembly process.
- Grease O-ring and place impactor into proper sampling tube.

- Place threaded portion tri-flange filter holder clamp over sampling tube
- Replace rain cap or cyclone/manifold assembly, as appropriate
- Install sampler at appropriate sampling location and load filters.
- Complete first two sections of Field Data Sheet, including vacuum leak test.
- Begin sampling; complete "Sampling Start" section of Field Data Sheet.
- After sufficient amount of deposit has been collected on the filters, determined either by visual inspection or by unacceptable vacuum gauge readings, complete "Sampling Stop" section of Field Data Sheet and turn off sampler. Recap exposed filters and place in field sample case.
- If replicate samples of the same source are desired, remove impactors and clean and re-grease impactor plates.
- When filters are unloaded from filter holders, record appearance of filters on Field Data Sheet.
- Ship copies of Field Data Sheets with filters to analyzing laboratory.

VII. DATA REDUCTION

Total volume sampled by each of the four ports on the sampler may be calculated by:

- Determine the calibrated flow rate for each critical orifice by consulting the calibration sheet attached to the back of this SOP.
- Determine the elapsed sampling time in minutes by subtracting the end elapsed time meter reading from the initial meter reading. This value should be compared to the start and stop dates and times, taking into account possible use of a cycle timer, to ensure that the sampler operated as expected.
- Multiply the flow rate by the total sampling time to yield total volume sampled for each of the four inlets.
- Any analytical results must take into account the ratios of the flows between the four ports before direct comparison of the different size fractions can take place. This is especially true if size fraction subtraction is performed to determine chemical compositions of particles between two sampled fractions (e.g., $<10\ \mu$ but $>2.5\ \mu$).

VIII. MAINTENANCE

Regular maintenance of the PISD primarily includes the cleaning of all parts which normally occurs as preparation for sampling. The conditions of impactor O-rings, filter holder gaskets, and all tubing should be checked on a regular basis, e.g., during each cleanup and installation.

The vacuum gauges should be checked on a monthly basis for zeroing. If the needle is not in the bar around zero on the gauge, the gauge may be zeroed or equilibrated to ambient pressures by removing the two screws from the gauge bracket to allow access to the rubber plug on the back of the gauge; the gauge need not be completely removed from the bracket, which would require loosening the brass fitting from the stem of the gauge. Push one side of the rubber plug toward the opposite side enough to allow air to enter or escape and bring the gauge back to zero. The plug need not be completely removed. If the plug is accidentally removed, take care that the dampening oil in the gauge is not lost.

The pump includes two filter elements inside jars connected to the inlet and exhaust sides of the pump. Every six months these filter elements should be checked for excessive build-up of filtered material, particularly the exhaust filter which will slowly collect carbon particles exhausted by the pump. The filters may be checked by removing the service panel from the front of the control module. NOTE: disconnect AC power from the control module before removing the panel. If the filters need to be removed, unscrew the filter jar and the bottom metal plate at the bottom of the filter element. The filter elements may be either blown clean with compressed air or replaced. If oil or moisture appears in the jar, replace the filter elements. Ensure that the filter jars are firmly tightened when replaced.

IX. TROUBLESHOOTING

Problem: vacuum gauge needles do not rest in bar near zero when sampler is not running.

Solution: zero gauges following procedure in Section VIII.

Problem: poor leak check during pre-sampling vacuum check on one inlet.

Solution: check tubing and fittings between manifold under sampling platform and QD fitting for loose fittings and split or damaged tubing. Replace or tighten as appropriate.

Problem: poor leak check on main vacuum gauge.

Solution: any main vacuum readings less than 20 inches of mercury should be cause for concern, in that the pump may not be able to maintain the proper vacuum drop across the critical orifices as the filters begin to load. Check all plumbing and fittings between the sampling module QD fittings and the control module QD fitting. In particular, look for loose fittings and split or damaged tubing. If nothing can be found, remove the service cover from the control module after disconnecting AC power and check that all internal fittings and tubing are in order. Check that filter jars are intact and tight. If no plumbing problems can be found, check for obstructions or kinks in the tubing between the main vacuum gauge and the pump. Finally, check that the voltage being supplied to the pump is 115 VAC; low voltage will affect the operation of the pump.

Problem: pump makes strange noises.

Solution: verify that AC power is 115 volts. Screeching sounds or uneven grinding sounds probably indicates a bearing problem. Refer to the pump service information for further details.

Problem: pump will not run.

Solution: verify that AC power is being supplied at 115 volts. Verify that wiring inside control module is intact and that switch is working properly with an ohm meter. NOTE: unplug AC power source before opening service panel; terminal strips and solder connections are open and may be touched accidentally when working inside the unit. If the pump is humming but will not pump air and AC power is correct, pump will need to be checked for either a seized bearing or a broken vane which has jammed the rotor on the pump; refer to the pump service information for further details or refer the pump to a qualified service technician.

Problem: elapsed timer will not work or runs slowly.

Solution: verify that supply voltage is correct at 115 VAC and 60 Hz. After removing AC power, open the service panel on the control module and check for the integrity of electrical connections to the meter. If no obvious problems can be found, replace the meter.

Problem: cooling fan in control module will not work or runs slowly.

Solution: verify that input voltage is correct. After removing AC power, open the service panel on the control module and check for loose electrical connections or broken wires. If no problems can be found, replace the fan. NOTE: the pump should not be run if the cooling fan is not working properly. The pump is cooled both by imparting some heat to air exhausted from the pump and by the cooling fan forcing air over the pump and through the louvers in the sides of the control module. The pump may overheat without the assistance of the cooling fan, especially if the sampler is operated outdoors in direct sunlight.

Problem: audited flow through critical orifice not within specifications.

Solution: ensure that the vacuum difference between the orifice in question and the main vacuum gauge on the control module is at least 15 inches of mercury. If the vacuum difference is sufficient, check the orifice. The critical orifice is located in the fitting on manifold under the sampling platform. Remove this fitting and inspect the orifice for foreign matter plugging the hole. Using compressed air, thoroughly clean the orifice and the manifold opening. NOTE: take care during removal and reinstallation of this fitting that loose particles of Teflon tape are removed and cannot later plug the orifice. If no problems can be found with the orifice, check for leaks around fittings or the press-fit seams in the sampling tube and for kinks or leaks in connecting tubing.

X. AUDITS

Total system leak checks and flow audits should be performed at the beginning and end of a sampling project or every six months, whichever is sooner. Refer to the PISD quality assurance for more information.

XI. MORE INFORMATION

For information about the sampler or its use or for problems not covered in section IX, contact:

OMNI Environmental Services, Inc.
10950 SW 5th Street, Suite 160
Beaverton, OR 97005
(503) 643-3755

Direct questions to Dr. James Houck or Mr. Lyle Pritchett.

EM
EM SCIENCE
111 Woodcrest Road, P.O. Box 5018, Cherry Hill, N.J. 08034-0395, Phone (609) 354-9200

MATERIAL SAFETY DATA SHEET

Essentially Similar to U.S. Department of Labor Form OSHA-20

SECTION 1 NAME & PRODUCT

Chemical Name: Ethyl Alcohol, Anhydrous, Denatured	Catalog Number: EX0278, EX0285
Trade Name & Synonyms: Ethanol	Chemical Family: Alcohol
Formula: CH_3CH_2OH	Formula Weight: 46.07

SECTION 2 PHYSICAL DATA

Boiling Point, 760 mm Hg (°C)	77°C	Specific Gravity (H_2O = 1) @ 20°C	0.79
Melting Point (°C)		Solubility in H_2O, % by wt. at 20°C	Soluble
Vapor Pressure at 20°C	48 mm Hg	Appearance and Odor clear, colorless	
Vapor Density (air = 1)	1.6	liquid with characteristic odor	
Percent Volatiles by Volume	100	Evaporation Rate (Butyl Acetate = 1)	~ 3.3

SECTION 3 FIRE AND EXPLOSION HAZARD DATA

Flash Point (test method) 51°F (cc)	Flammable Limits	Lel 3.3 (Pure Ethanol)	Uel 19.0 (Pure Ethanol)

Extinguishing Media: Dry chemical, CO_2, "alcohol" foam

Special Hazards and Procedures: Wear self-contained breathing apparatus, full face shield, positive pressure.

Unusual Fire and Explosion Hazards: Vapors are heavier than air and may travel and be ignited by pilot lights, other flames, sparks, heaters, smoking, etc.*

SECTION 4 REACTIVITY DATA

Stable X	Conditions to Avoid heat, sparks, open flame
Unstable	* at locations distant from material handling point

Materials to Avoid

( ) Water ( ) Acids ( ) Bases ( ) Corrosives (✓) Oxidizers

( ) Other (specify)

Hazardous Decomposition Products: CO_x and various hydrocarbons

SECTION 5 SPILL OR LEAK PROCEDURES AND DISPOSAL

Steps to be Taken in Case Material is Released or Spilled: Eliminate ignition sources. Evacuate non-essential personnel. Absorb w/ vermiculite

Waste Disposal Method: To be performed in compliance with all current local, state and federal regulations

EX0278, EX0285

SECTION 6 HEALTH HAZARD DATA

Threshold Limit Value

TLV-air: 1000 ppm

TXDS:
orl-hmn LDLo: 500 mg/kg
orl-rat LD50: 13.7 g/kg
orl-man TDLo: 50 mg/kg Toxic effects:
G.I.: diarrhea, constipation, ulceration

Effects of Overexposure

Irritation to eyes, skin, G.I. tract (nausea, vomiting, diarrhea); headache, dizziness, fatigue, unconsciousness. Blindness and death possible by ingestion. Lab animals: damage to liver & kidneys, eyes, spleen, lungs, brain, central nervous system

First Aid Procedures

Skin: wash with soap/water; remove contaminated clothing
Eyes: flush with water lifting lids occasionally; get medical assistance
Inhalation: remove to fresh air; get medical assistance
Ingestion: give 2 glasses water & induce vomiting at once if conscious; get medical help

SECTION 7 SPECIAL PROTECTION INFORMATION

Ventilation, Respiratory Protection, Protective Clothing, Eye Protection

Provide adequate general mechanical and local exhaust ventilation
Protect eyes and skin with safety goggles and gloves
Wear impervious clothing, boots & air-supplied mask in confined areas

DOT - Flammable Liquid

SECTION 8 SPECIAL HANDLING AND STORING PRECAUTIONS

Keep container tightly closed. Store away from heat, sparks, open flame.

SECTION 9 HAZARDOUS INGREDIENTS

(refer to section 3 through 8)

	Approx %	TLV
Ethanol	93	1000 ppm
Methanol	4	200 ppm
Ethyl Acetate	1	400 ppm
Methyl iso-Butyl Ketone	1	100 ppm
Hydrocarbon Solvent	1	

SECTION 10 OTHER INFORMATION

Containers are hazardous when empty; observe all stated precautions.

EMERGENCY PHONE NUMBER (609) 423-6300

AUTHORIZED SIGNATURE

DATE ISSUED: 7/81

DATE REVISED: 8/83

70-270
G370PL
(3-87)

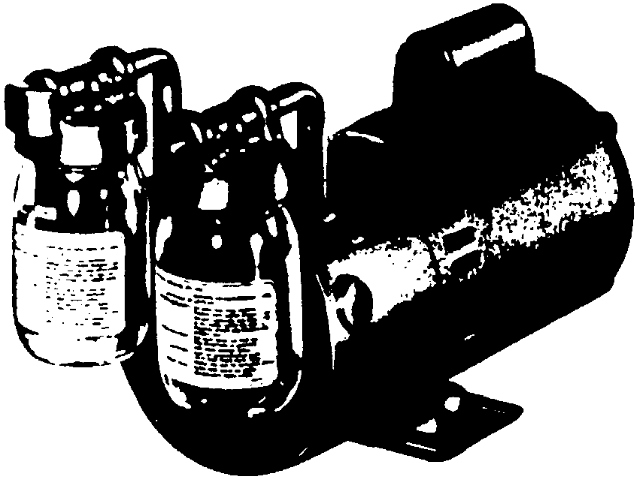
GAST MANUFACTURING CORPORATION
P O BOX 97 BENTON HARBOR MICHIGAN 49022
PHONE 616 926 6171

PARTS LIST and OPERATING and MAINTENANCE INSTRUCTIONS

0322
0522
0822
1022

OIL-LESS VACUUM PUMPS and COMPRESSORS

• Special Motors Available



Model 1022 Series

Oil-less, Motor Mounted

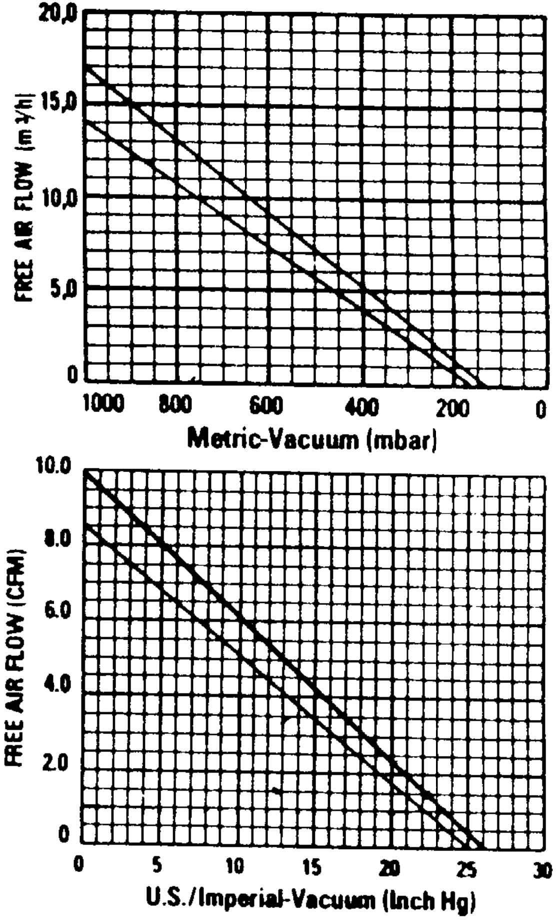
INCLUDES (ALL MODELS)

- Muffler and Filter
- Thermotector (Single Phase Motors Only)

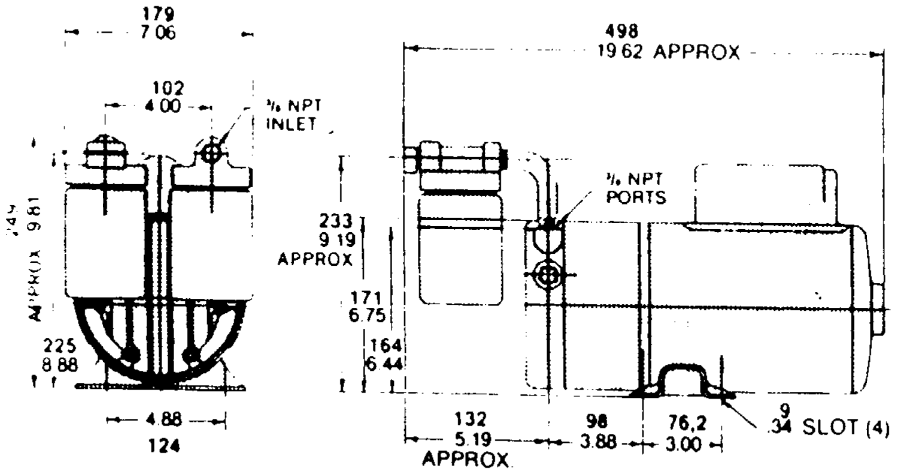
RECOMMENDED

- Vacuum Relief Valve AA840A
- Vacuum Gauge AA640
- Repair Kit K223

Black line on curve is for 60 cycle performance.
Blue line on curve is for 50 cycle performance.

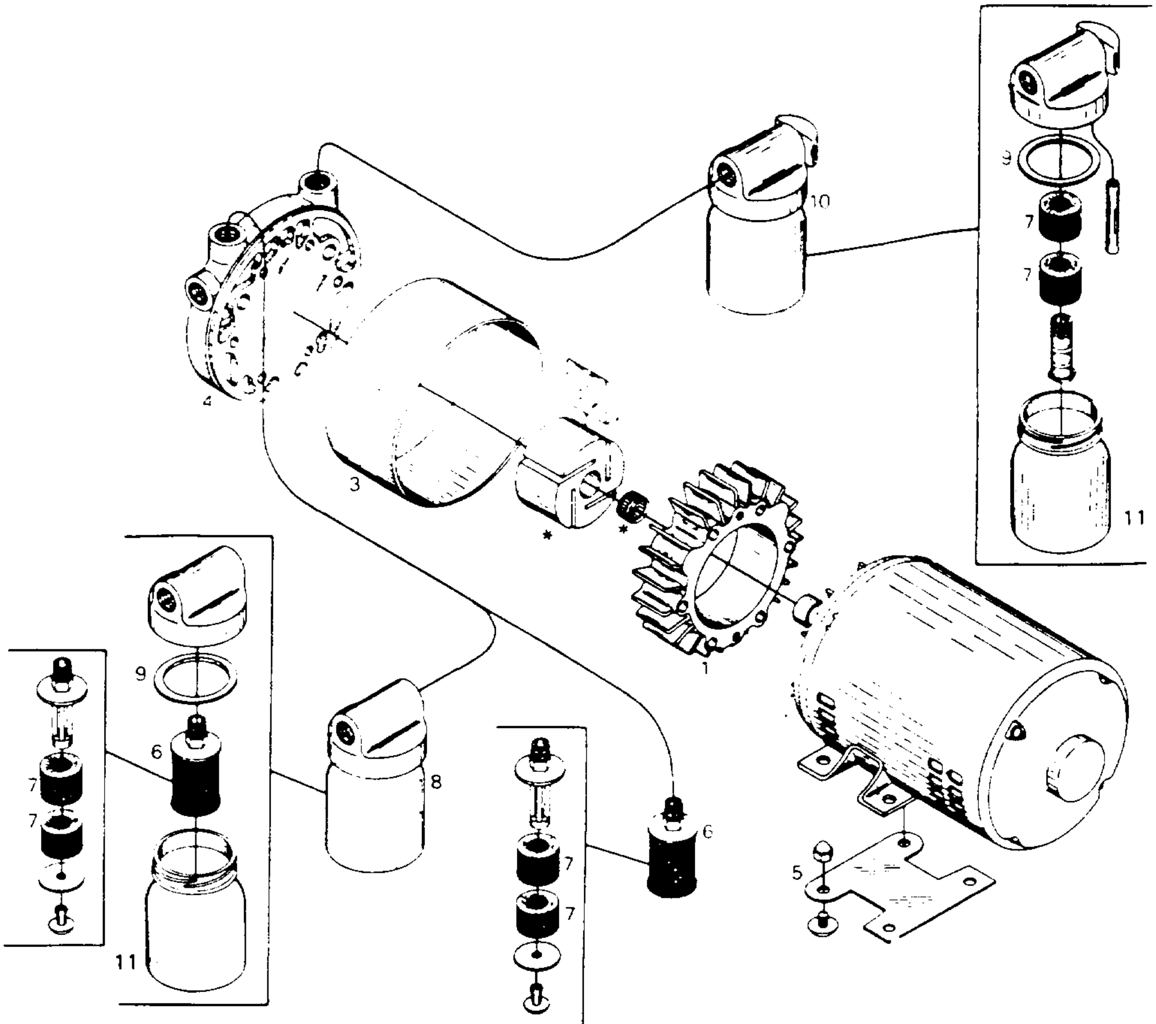


Note: On all drawings
Metric
U.S./Imperial



Model No. Chart	Motor	RPM 60 Cycle	RPM 50 Cycle	HP	kW	Net Wt lbs.	Net Wt kg
1022-V103-G272X	115/230-60-1	1725		¾	0,56	50	22,7
1022-V103-G274X	110/220-50-1		1425	½	0,37	50	22,7
*1022-V703-R50X	220/240-50-1		1425	½	0,37	50	22,7
*1022-V703-R76	220/250, 380/440- 50/60-3		1425	¾	0,56	50	22,7

*Available only in Europe



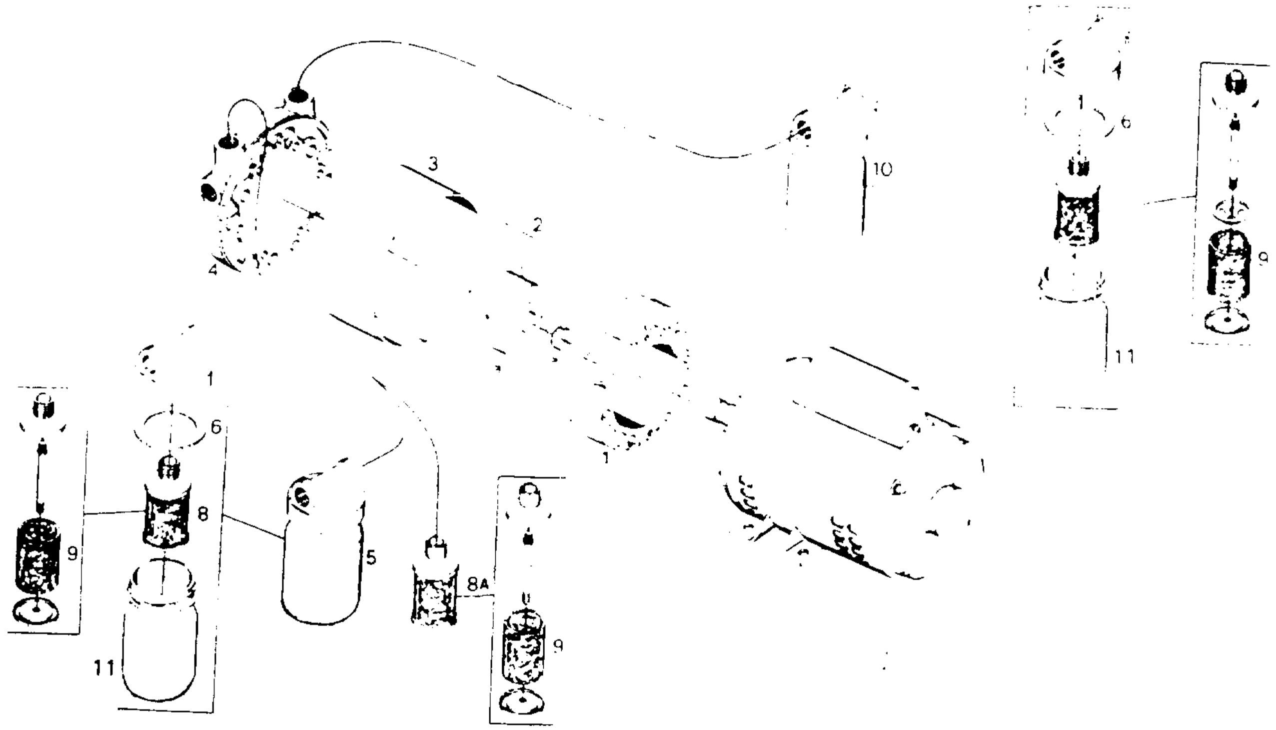
Vacuum Pump
Intake Filter Assembly

Compressor
Intake Filter Element Assembly

Ref No	Description	Part Qnty	Vac Pump 0322-V103	Vac Pump 0522-V103	Compressor 0322-P102	Compressor 0522-P102
1	Body	1	AF107	AF108	AF107	AF108
• 2	Vane	4	AF109C	AF109C	AF109C	AF109C
3	Shroud	1	AF111	AF111	AF111	AF111
4	End Plate	1	AF112E	AF112E	AF112E	AF112E
5	Foot Support Assembly	1	AC136	AC136	AC136	AC136
6	Intake Filter Element Assembly	1	B343B	B343B	B343B	B343B
• 7	Filter Felt	4	B344A	B344A	B344A	B344A
8	Intake Filter Assembly	1	V400G	V400G		
• 9	Cover Gasket	2	B62A	B62A		
10	Muffler Assembly	1	V425L	V425L		
11	Jar	2	AA125A	AA125A		
	Service Kit	1	K247	K247	K247P	K247P

• Denotes parts included in Service Kit. Extra filter felts and cover gaskets are included when kit is used for compressor.

* Under most circumstances rotor and tolerance ring should not be replaced in the field



Vacuum Pump
Intake Filter Assembly

Compressor
Intake Filter Element Assembly

Ref. No.	Description	Part Qnty.	Vac Pump 0822-V103	Compressor 0822-P102	Vac Pump 1022-V103	Compressor 1022-P102
1	Body	1	AB978	AB978	AB887	AB887
• 2	Vane	4	AB992B	AB992B	AB992B	AB992B
3	Shroud	1	AB981	AB981	AB981	AB981
4	End Plate	1	AB982C	AB982C	AB982C	AB982C
5	Intake Filter Assembly	1	AB599		AB599	
• 6	Cover Gasket	2	AA405		AA405	
7	Muffler Element Assembly	1	AC434		AC434	
8	Filter Element Assembly	1	AC433		AC433	
8A	Filter Element Assembly	1		AC432		AC432
• 9	Cartridge	2	AC393	AC393	AC393	AC393
10	Exhaust Muffler Assembly	1	AB599B		AB599B	
11	Jar	2	AA805		AA805	
	Service Kit	1	K223	K223P	K223	K223P

• Denotes parts included in Service Kit. Extra filter cartridge and cover gaskets are included in kit when used for compressor.
* Under most circumstances rotor and tolerance ring should not be replaced in the field.

OPERATING and MAINTENANCE INSTRUCTIONS

CAUTION: NEVER LUBRICATE THIS DRY "OIL LESS" AIR PUMP. The carbon vanes and grease packed motor bearings require no oil.

CONSTRUCTION: The outer end plate, body, rotor and mounting bracket are all cast iron. Consequently any moisture that accumulates in the pump will tend to corrode the surface when pump stands idle. The vanes are made of hard carbon and are precision ground. They should last 5,000 to 10,000 hours depending upon the degree of vacuum or pressure at which the pump is run.

STARTING: If the motor fails to start or slows down when under load, shut the unit off and unplug. Check that the supply voltage agrees with the motor post termination and the motor data nameplate. If the pump is extremely cold, bring to room temperature before starting. If anything appears to be wrong with the motor, return the complete pump to an authorized Gast service facility.

NOTE: ALL DUAL VOLTAGE MOTORS ARE SHIPPED WIRED FOR THE HIGH VOLTAGE. REFER TO STARTING INSTRUCTIONS.

WARNING - THE MOTOR MAY BE THERMALLY PROTECTED AND WILL AUTOMATICALLY RESTART WHEN THE PROTECTOR RESETS. *ALWAYS* DISCONNECT POWER SOURCE BEFORE SERVICING. PERSONAL INJURY AND/OR PROPERTY DAMAGE COULD RESULT.

FILTERS: Dirty filters restrict air flow and if not corrected could lead to possible motor overloading and early pump failure. Check filters periodically and clean when necessary by removing felts from the filter and washing in a solvent*. Dry with compressed air and replace.

FLUSHING: Should excessive dirt, foreign particles, moisture or oil be permitted to enter the pump, the vanes will act sluggish or even break. Flushing of the pump should take care of these situations. In order to flush a pump, remove the filter and muffler assemblies and introduce several teaspoons full of solvent* into the pump through the intake WHILE THE PUMP IS RUNNING. Flush unit in a well ventilated area. Eye protection is recommended. Keep face away from exhaust port and do not flush unit with flammable solvent. Repeat the flushing procedure and if it does not remedy the situation, remove the end plate for further examination.

*Recommended Solvent: Gast Flushing Solvent part AH255.

WARNING - DO NOT USE KEROSENE OR OTHER COMBUSTIBLE SOLVENTS. PERSONAL INJURY AND/OR PROPERTY DAMAGE WILL RESULT.

DISASSEMBLY: If flushing does not eliminate the problem, remove the six bolts holding the end plate to the body. Now remove the end plate and the four vanes (DO NOT REMOVE THE ROTOR OR LOOSEN ANY ELECTRIC MOTOR "THRU-BOLTS"). If the pump fails to produce the proper vacuum or pressure, the vanes could be worn. A metallic clanging could mean the rotor and body are touching. The top clearance may be adjusted by "LIGHTLY" tapping on the pump body (either top or bottom depending upon whether clearance is too large or small). The rotor should be turned while setting clearance to assure that all points on the rotor clear the body. End clearance for the drive end is .0015 and .003 for the dead end.

DANGER: To prevent explosive hazard, do not pump combustible liquids or vapors with these units. Personal injury and/or property damage will result.

It is usually quickest and least expensive to send the unit in for repair. All returns are F.O.B. Benton Harbor, Michigan.
†Authorized service facilities are located at:

Gast Manufacturing Co., Ltd.
Halifax Road, Cressex Estate
High Wycombe, Bucks HP12 3SN
England
High Wycombe 23571

Brenner-Fiedler & Assoc.
13824 Bentley Place
Cerritos, Ca. 90701
(213) 404-2721

Gast Manufacturing Corp.
2550 Meadowbrook Road
Benton Harbor MI 49022
616/926-6171

Wainbee, Ltd.
121 City View Drive
Rexdale, Ontario, Canada M9W 5A9
416/243-1900

Gast Manufacturing Corp.
505 Washington Ave.
Carlstadt NJ 07072
201/933-8484

Wainbee, LTD.
215 Brunswick Blvd.
Pointe Claire, Montreal,
Canada H9R 4R7
514/697-8810

Appendix C
Standard Operating Procedures
Dilution Sampling System

Standard Operating Procedures
Dilution Sampling Systems

Table of Contents

LIST OF FIGURES

1.0 INTRODUCTION

The OMNI Dilution Sampling System (DSS) is a unique stack sampler which withdraws an aerosol stream from an industrial stack or flue, mixes and cools the source aerosol with excess filtered ambient air, and collects size-fractionated particles from the diluted aerosol on filters suitable for detailed chemical analyses. The natural diluting and cooling of stack aerosols as they emerge from the stack and mix with ambient air result in numerous suspected or confirmed chemical and physical changes, including condensation, agglomeration, vaporization, and secondary chemical reactions. These changes may have significant impacts on the size distributions and chemical compositions of stack particles. Traditional stack sampling methods which do not attempt to duplicate the dilution/cooling process may yield samples which are not truly representative of particles originating from high temperature point sources.

Two interfaced Parallel Impactor Sampling Devices (PISD) allow size characterization of emission particles. Any set of size fractions can be collected with the interfaced PISD's depending on the requirements of the study. Standard impactor design theory allows for the design of impactors for any reasonable cut-point. Generally 1μ, 2.5μ, and 10μ cut-points have been used with the PISD. Each PISD has four inlets; hence four different size fractions or replicates can be collected siumltaneously with each PISD. This size fractionation is important not only because differently sized particles may be different chemically, but also because different size fractions of particles have different health and environmental (e.g., visibility) impacts associated with them. For source impact assessment, size fractionation which parallels that of the ambient sampling equipment makes quantification of source impacts by receptor modeling more accurate.

Filter media used in the DSS are typically stretched Teflon membrane filters or quartz filters. Teflon is analytically clean and is the medium of choice for multi-element analysis by X-ray fluorescence (XRF). Teflon also lends itself well to examination by scanning electron microscopy (SEM). Quartz filters are principally used for carbon analyses. In addition, both quartz and Teflon filters can be used for wet chemical analysis techniques such as ion chromatography (IC); atomic absorption spectrophotometry (AAS); inductively coupled argon spectroscopy (ICAP); and ammonium analysis. Generally samples are collected simultaneously on both Teflon and quartz filters, since two PISD's are interfaced with the DSS. The use of two filter media, together with the dilution provided by the DSS, provide the ability to produce much improved source chemical fingerprints over traditional stack characterization techniques.

In addition to particle fractionation and collection, gaseous analyzers may be connected to the DSS at existing extra ports either to take advantage of the lower concentrations or such gases as sulfur dioxide as a result of the dilution or to study the depletion or enrichment of such gases as a result of any secondary chemical reactions or adsorption of gases onto the particles.

2.0 PRINCIPLES OF OPERATION

A schematic of the DSS is presented in Figure 1, and a sketch is presented in Figure 2. When the DSS is operating, a sample of the gases is collected from the stack via a standard stainless steel buttonhook nozzle and pulled through stainless steel stack probe into the dilution chamber. Upon entry into the dilution chamber the sample is mixed with filtered ambient air. Complete mixing is produced by turbulence which is caused by ninety degree bends in the system. An aliquot of the diluted, mixed, sample stream is withdrawn at the exhaust end of the dilution chamber. This aliquot is sampled for particles by two specially adapted Parallel Impactor Sampling Devices (PISD) to provide size fractionation. The fractionated particles are collected onto 47 millimeter filters downstream of the impactors.

Withdrawal of the particles and gases from the stack is accomplished by the interaction of two high-volume blowers with adjustable flows, one blowing into the system to provide dilution air, and the other pulling the diluted aerosol stream from the system. The difference in flows between the inlet and exhaust blowers provides the negative pressure required to pull a sample from the stack. Calibration curves are developed in the laboratory which relate the difference in pressure (ΔP) between the dilution chamber and the inside of the stack to the velocity of the gas pulled through the inlet nozzle (see Figure 3). By monitoring the ΔP and the DSS chamber gas velocity, the nozzle flow (and subsequently, the nozzle velocity) may be determined from the calibration curves, and the dilution ratio may be determined.

The principle behind using the ΔP as a method for determining the nozzle flow is Bernoulli's equiation of continuity, which demonstrates that the linear velocity of the gas entering the buttonhook nozzle is dependent only on the pressure drop and the density of the gas. Bernoulli's equation is only strictly applicable to idealized fluids, which is the reason laboratory calibration curves are required. The density of the stack gases will also change with differeing temperatures, pressures, and varying gaseous compositions and cannot be assumed to be static. In most applications the uncertainties produced by these parameters are not problematic, and small corrections can be applied. In addition, the dilution ratio need not be precisely known, and the deviations from isokinetic sampling (if desired) are small as compared to the temporal variability of stack gas velocities.

3.0 PHYSICAL DESCRIPTION/SPECIFICATIONS

The DSS requires 110 VAC, 60Hz electical power. Current drawn by the sampler is dependant on the operating speed of the blowers and the load of the pumps. Circuits rated to at least a total of 35 amps should be used to prevent power failure.

The DSS is constructed of the following components:

Inlet:

Standard stainless steel buttonhook nozzles in $^1/_4$, $^3/_8$, and $^1/_2$ inch diameters.

A 4' and 2' stainless steel probe.

A 4" diameter stainless steel manifold with a sheath for the probe and a dilution air port. A water trap may be attached to the bottom of the manifold to collect condensation.

Dilution Chamber:

Stainless steel 4" pipe in varing lengths and 90° bends connects the manifold to the dilution chamber.

One 4' section has three ports for monitoring chamber gas velocity, temperature and pressure.

The chamber sits on a 2' × 3' table with adjustable legs. The chamber, made of aluminum, has four $^3/_4$" threaded ports for supplementary gas monitoring or sample collection.

Sampling Tubes:

Eight aluminum sampling tubes sit vertically in the bottom of the dilution chamber. The top of these tubes hold impactors with cut points of <1 μm, <2.5 μm, and <10 μm. Filter holders containing 47 mm filters are held in place with brackets at the bottom of the sampling tubes. Two vacuum pumps and eight rotometers control the vacuum to the filters.

Blowers:

Both dilution air and exhaust functions are provided by two Cadillac blowers. Adjustments in flow rates are provided by two Powerstat variable transformers. Filtering of the dilution air is accomplished by an 8" × 10" glass fiber filter held in aluminum in-line filter holder.

Miscellaneous:

A Kurz model 441M linear velocity meter provides measurement of the flow within the dilution chamber during sampling.

Three Magnehelic differential pressure gauges, with ranges of 0-2, 0-5, and 0-10 inches of water, allow accurate monitoring of ΔP while sampling.

A flexible stainless steel fiber thermocouple is used to read stack temperature. A short stainless steel probe is used to monitor dilution chamber and ambient air temperature. These temperatures are read on a digital themometer.

4.0 OPERATING INSTRUCTIONS

4.1 Sampler Preparation

All components of the DSS which come in contact with sample gas should be completely cleaned with water, wiped down with ethanol, and dried completely before a different source is sampled. DO NOT use soap or detergent or any other chemical that may leave a residue.

The impactors are cleaned by unscrewing the impactor plate, cleaning all parts with ethanol, and regreasing the impactor plates. The screw heads should be wiped with ethanol after the impactor is reassembled to remove any metal filings.

4.2 Presampling Preparation

For each sampling location a field data sheet "Dilution Sampler Field Data Sheet: Stack Description and Parameters" (Figure 4) must be completed.

Measurements:

Measure the stack velocity pressure with the S type Pitot tube inserted into the port a distance coresponding to the sampling point. Take care that the port is sealed around the tube when this is done. Record the reading on the Field Data Sheet.

Measure the static pressure with the Pitot tube perpendicular to the gas flow and with one of the tubes disconnected. Note whether the static pressure is positive or negative.

Measure and record the stack temperature at the point of sampling using the fexible stainless steel probe wired to the Pitot tube.

Measure and record the ambient temperature with a short TC probe in the shade.

Calculations:

Calculate the stack velocity (mps) using the formula on the Field Data Sheet.

Correct stack velocity to the calibration temperature.

Considering the estimated stack humidity, stack temperature, and stack velocity converted to ambient temperature, select a nozzle size and dilution ratio by referring to the calibration charts. A sample calibration plot is presented in Figure 3. These plots show both the ΔP and the chamber velocity plotted verses nozzle velocity. The ΔP is indicated by open circles on a curved line, whereas the chamber velocity line is straight and without marked points. Several guidelines should be followed when picking a nozzle size and dilution ratio:

> A hot stack will require a higher dilution ratio to cool the stack gases to as close to ambient as possible. This must be balanced against the fact that higher dilution ratios also mean longer sampling times will be required to collect sufficient amounts of deposit on the filters.
>
> High stack or flue humidity also require more dilution air to prevent excessive condensation of water inside the sampler and loss of particles into that water.
>
> A calibration curve should be selected so that the nozzle velocity is relatively close to the middle of the curve. Points that fall near the top of the curve require the blowers to work harder and leave less room for adjustments in the blowers' flow rates as filters begin to load. Points that fall near the bottom of the curve generally require that the blowers run relatively slowly, which makes accurate regulation difficult.

Obtain from the selected calibration curve the dilution chamber velocity (in mps) and ΔP (in inches of water) based on the corrected nozzle velocity.

4.3 Assembly of DSS

Attach selected probe and pressure tube, depending on the size of the duct, to the manifold.

Attach selected nozzle to the end of the probe positioned into the flow when the manifold is positioned such that the water trap port is at its lowest point.

Run the stainless steel thermocouple through the $^1/_4$" tube at the end of the manifold. Attach it to the probe such that the end is near the nozzle but does not restrict flow.

Bolt the manifold either to a plate that is in turn clamped to the port or to a female 4" theaded plate that may be screwed into some sampling ports.

Attach a 90° bend followed by the 4' pipe with the three ports on the downstream side.

Attach the adjustable legs to the table. Note that there is easier access to the filters if the system is set up such that the table is as high as posible.

Set dilution chamber on table.

With the remaining pipe and bends and by moving and adjusting the height of the table, connect the manifold to the dilution chamber. Note some adjustment may be obtained from the caps on the table legs and by moving the manifold up or down within the sampling port.

Attach a blower to a flexible blue hose, to the hi-vol filter holder, and then to the dilution air port of the manifold. The hi-vol filter holder should be positioned such that the screen suports the filter against the flow.

Use the shortest blue flexible hose to connect the exhaust port of the dilution chamber to the exhaust blower. Attach the remaining hose to this blower to discharge the exhaust away from the location where the dilution air inlet is positioned.

Plug each blower into its respective variac.

Zero the Magnehelic gauge in the position it is to be used.

Attach the Tygon tubing to the appropriate Magnehelic gauge with the high side attached to the probe pressure sensor, and the low side to the chamber port.

Insert the temperature probe into the chamber port.

With the impactors inserted into their respective sampling tubes, bolt the chamber door on.

Bolt the rotometer rack to the table and connect the tubing into its respective vaccum manifold.

Connect the vacuum hoses to the manifolds and to their respective pumps.

Plug one variac and one blower into each of two seperate 110V circuits to prevent overloading.

Load filters into the holders in a clean environment.

Insert $^1/_4$" black gaskets into the bottom of the sampling tubes.

Load the filter holders into the bottom of their respective sampling tubes.

4.4 Flush Run

A flush run is sugested to both purge the system of contaminants and to get an idea of filter loading time. A scrap filter may be used to estimate the loading rate. The other other tubes may either be covered with plastic bags in which case the other filters may be in place or red caps may be inserted into the bottom of the sampling tubes. Note the flush run on the data sheet. A run of at least 15 minutes is sugested.

4.5 Sample Collection

Begin by filling the required data on the field data sheet entitled "Dilution Sampler Field Data Sheet: Start" (Figure 5). Enter the plant, stack, run number, operator, start date, and the ΔP and chamber velocity previously selected.

Record the filter ID numbers and the pump timer readings on the respective PISD data sheets.

Note the test start time and turn on the vacuum pumps. Immediately adjust the rotometers to 10.0lpm. Since 10 liters per minute actual flow is required for correct impactor performance, required rotometer settings will change with elevation. Temperature as well as pressure (elevation) also has an effect on rotometer readings, albeit smaller.

The equation to correct rotometer readings for pressure and temperature is:

$$V_2 = V_2 \sqrt{\frac{P_1}{P_2} \times \frac{T_2}{T_1}},$$

where, V_1 is the velocity at STP (1 atmosphere, 293° K),
V_2 is the velocity at the actual ambient temperature and pressure,
P_1 is 1 atmosphere pressure,
P_2 is the actual ambient pressure in atmospheres,
T_1 is 293° K, and
T_2 is the actual temperature in degrees Kelvin.

Simultaneously adjust the ΔP and the chamber velocity to the values previously determined by adjusting the variacs connected to the inlet and outlet blowers. Hint on adjusting the blowers: increasing the the power to the exhaust blower increases the ΔP and the chamber velocity; increasing the power to the inlet blower decreases the ΔP and increases the chamber velocity. Note: normal stack turbulence and variability in plant processes will often make the ΔP readings rather variable; try to set the blowers to achieve an average ΔP at the value desired.

Periodically recheck the sampler settings, the ΔP and the chamber velocity and make corrections as necessary. Record the data requested on the Intermittent Inspections Field Data Sheet (Figure 6). Between sampler checks, remove the velocity sensor from the chamber port to prolong its lifespan, then recap the port.

At the end of the sampling period, record the required information on the "Stop" Field Data Sheet (Figure 7). Shut off the flow through the rotometers, then turn off the pumps. Slowly turn down the blower variacs. If bulk samples of raw materials, products, baghouse dust, etc. are obtained from the plant, note these on the data sheet. Unload the filters. Visually estimate the amount of loading to determine if additional samples are necessary and what optimum sampling time would be.

4.6 Repeat Sampling

If replicate samples are desired follow the above steps including replacement of the hivol filter. (Save the hivol filter for possible analysis). The impactors should be regreased between runs. Use a wooden dowel to remove them from the sampling tubes. With a laboratory spatula wrapped in a Kimwipe, wipe off the used grease. Use the spatula to regrease the impactor by evenly spreading grease in the depression of the plate.

The entire system need not be cleaned between runs of the same source.

4.7 Disassembly of DSS

Follow the reverse precedure as outlined in section 4.3. Watch for signs of any problems which may affect the quality of the samples such as condensation or a plugged nozzle.

5.0 Maintenance

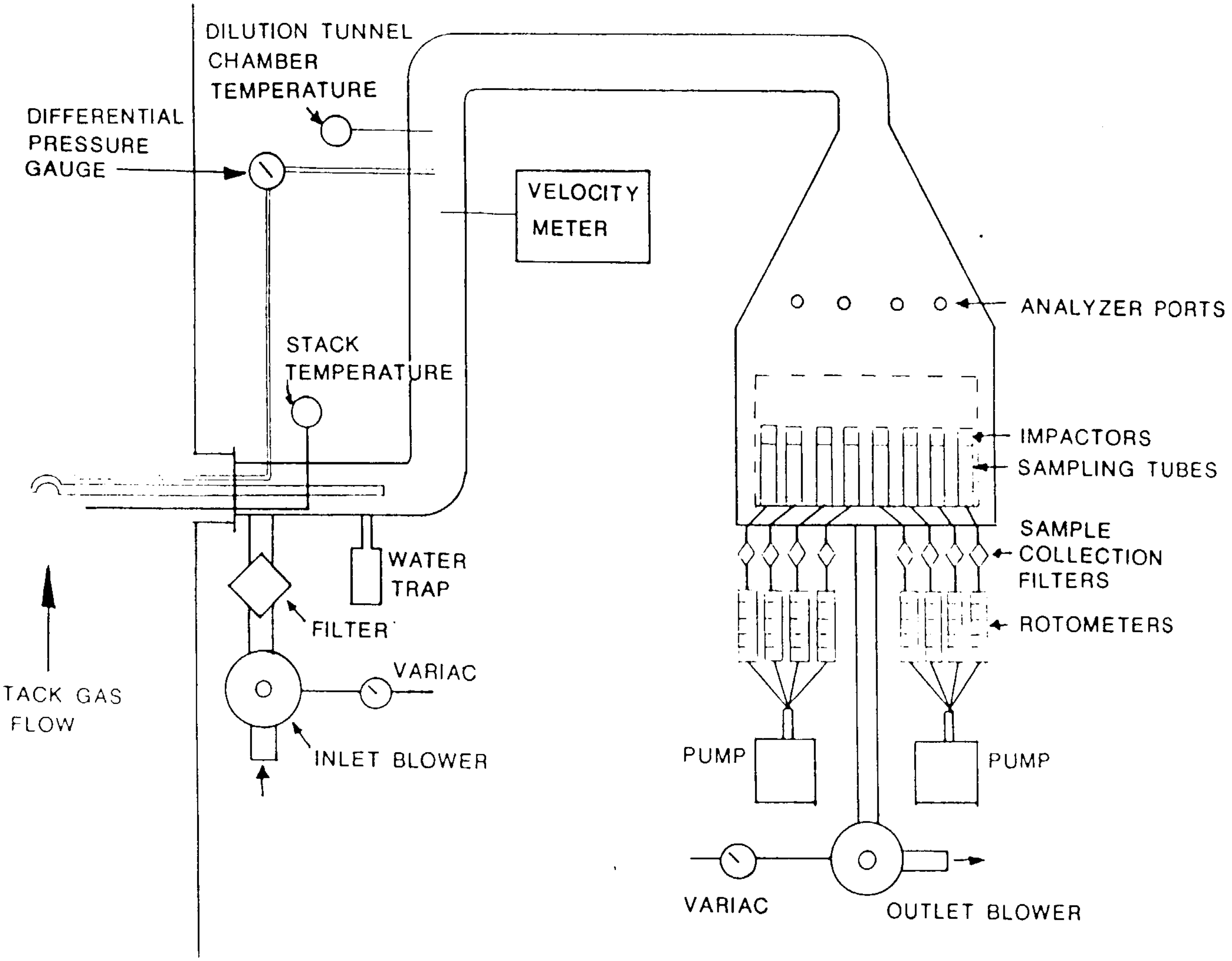
Maintenance for most of the components in the DSS consists of periodically checking for worn or broken parts. Inspections of tubing, fittings, buttonhook nozzles, and the like should be incorporated into the cleaning procedure. The brushes in the high-volume blowers should be checked periodically for wear. Access to the brushes is through two caps at the motor end of the blower.

6.0 Trouble Shooting

Difficulty in obtaining a desired ΔP-chamber velocity combination may be due to leaks or poor connections at both ends of the blower hoses, improperly closed filter holders, or overloading of the dilution air filter.

Abnormally low chamber velocity readings may be caused either by fouling of the sensor tip or low battery voltage in the meter. The sensor tip may be GENTLY cleaned by dabbing with an ethanol moistened paper towel. The battery in the meter may be recharged by connecting the electrical adapter to the meter. Note: if possible, use the adapter at all times during sampling.

Figure 1 Dilution Sampling System Schematic



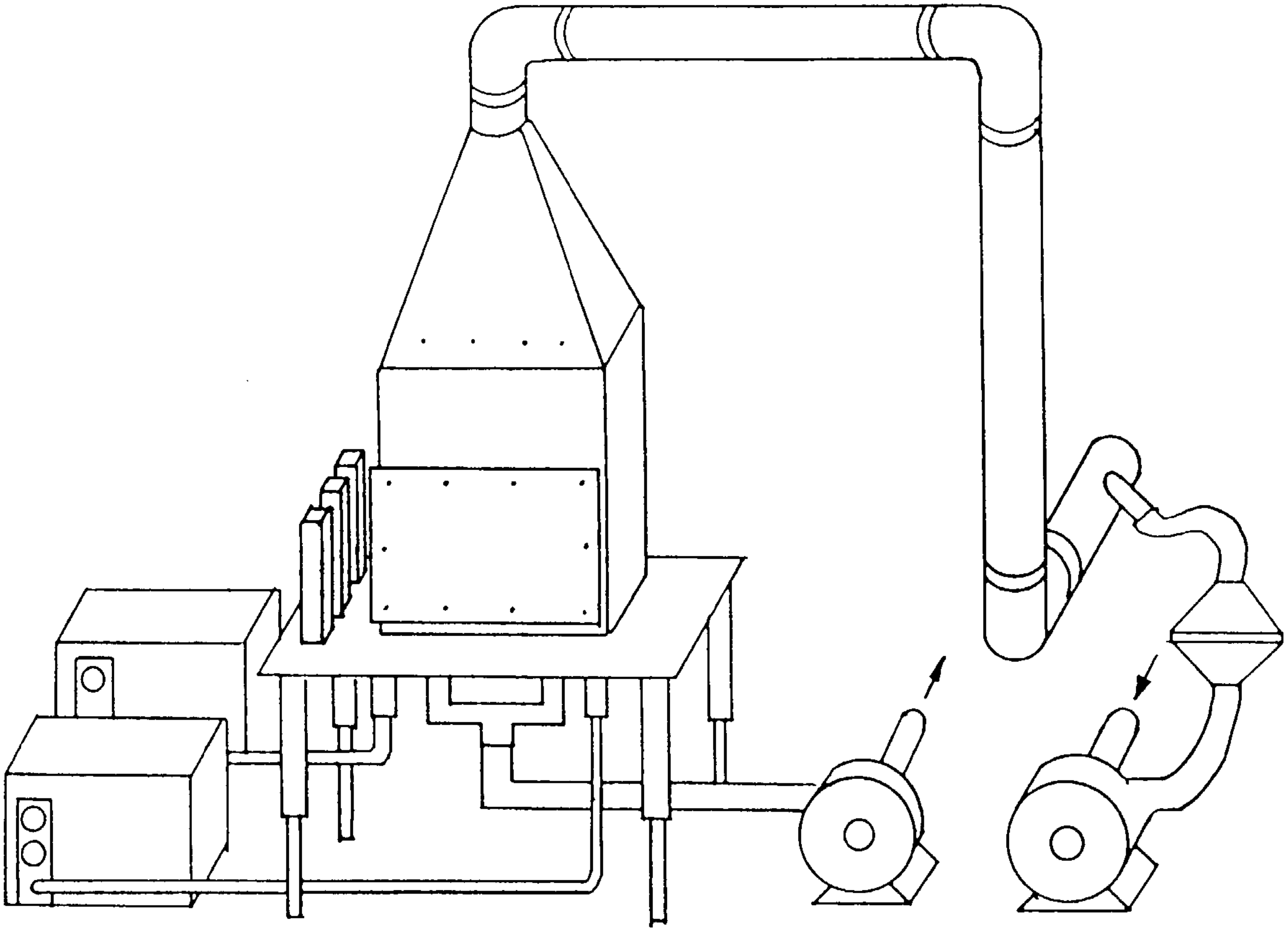


Figure 2 Dilution Sampling System Illustration

Figure 3

CALIBRATION PLOT

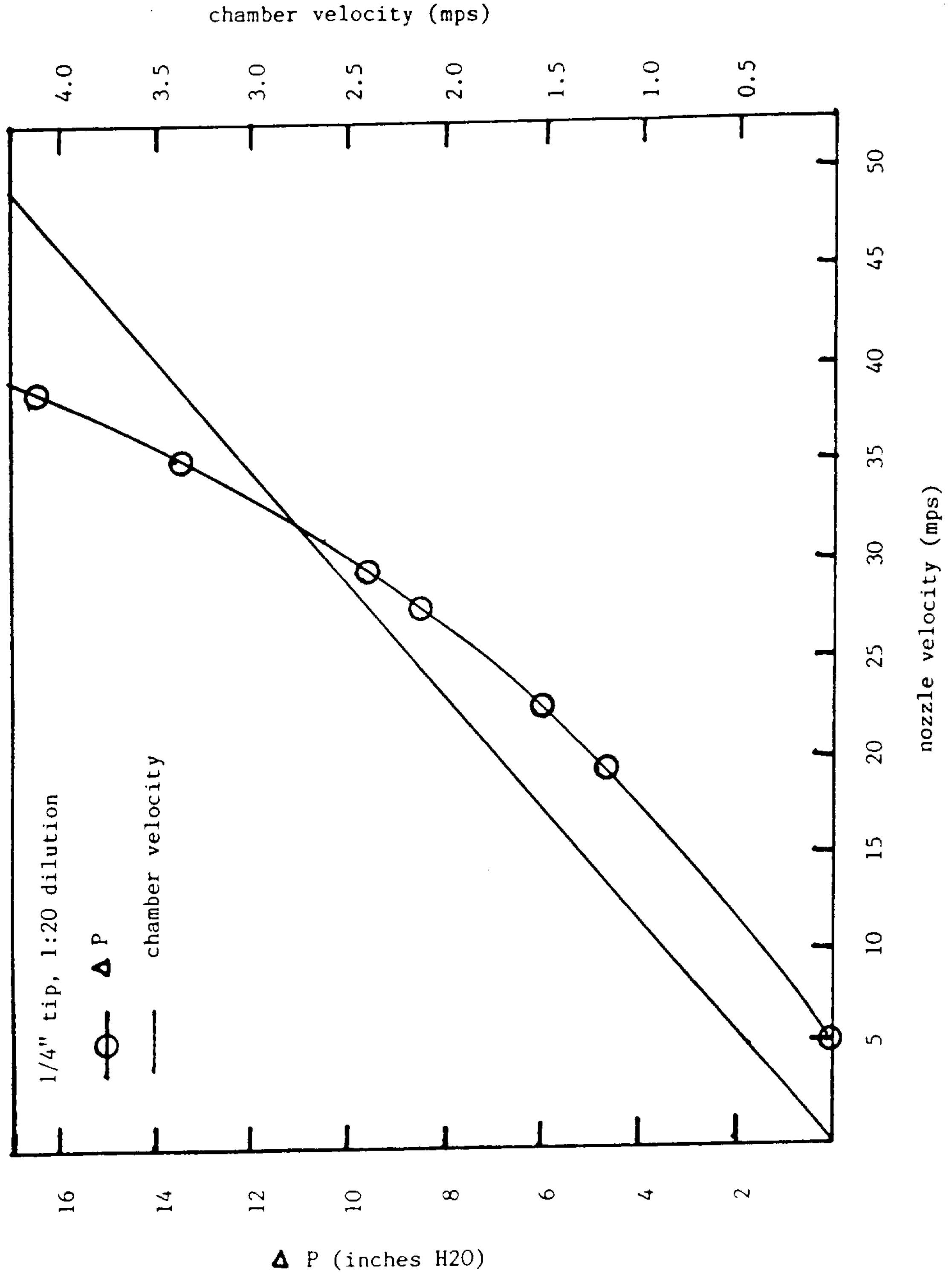


Figure 4

Dilution Sampler Field Data Sheet:
Stack Description and Parameters

Plant ____

Stack ____

City ____

Sample Port Description ____________________________

____

Unusual Conditions ____________________________

____

Date ____________________ Time ____________________

Operator ____________________

* *

$$°C = (°F - 32) / 1.8$$

Ambient Temperature _____ °F _____ °C   Stack Temperature _____ °F _____ °C

Δ Pressure _________ " H_2O Stack Static Pressure ________

$$v(mps) = 0.987 \sqrt{(\Delta P \text{ in. } H_2O)(\text{stack temp } °C + 273°)}$$

Calculated Stack Velocity _______ mps

$$v \text{ (corrected)} = \frac{295°}{\text{stack temp } °C + 273°} (v \text{ stack})$$

Effective Stack Velocity, corrected to ambient temp ____________ mps

* *

Selected Nozzle Diameter _________   Dilution Ratio _______________

[from calibration chart:]

Dilution Chamber Velocity ________ mps Δ P _______________

Distance of inlet from stack wall ____________________

Comments ____

____

____

____

Figure 5

Dilution Sampler Field Data Sheet: Start

Plant ____

Stack ____

Run Number ______________________ Δ P ____________________________" H2O

Operator ________________________ Chamber Velocity ______________ mps

Start Date ______________________ Start Time ________________________

Stack Temperature _____°F _____°C

Ambient Temperature _____°F _____°C

Inlet 8 X 10 Filter ID _____________ Type ___________________________

Comments ____

____

____

____

____

____

Figure 6

Dilution Sampler Field Data Sheet
PISD

Sample Description ____
Site/City ____
Sampler ID ________________________
Sampler Configuration ____
Impactor Grease Type _______________
Sampling Sequence ____
Technician(s) _____________________

10 µ : Filter ID ___________________ Filter Type _____________
10 µ : Filter ID ___________________ Filter Type _____________
2.5 µ: Filter ID ___________________ Filter Type _____________
2.5 µ: Filter ID ___________________ Filter Type _____________

****

Resting Vacuum Reading

Pump _______ inches Hg

****

Sample Start:
Date/Time _______________________ Pump _______ inches Hg
Elapsed Time Meter _________ minutes

****

Sample Stop: Pump _______ inches Hg

Flow:
10 µ _______ lpm
10 µ _______ lpm
2.5 µ _______ lpm
2.5 µ _______ lpm

Date/Time _______________________
Elapsed Time Meter _________ minutes

****

Filter Removal:
Date ____________________________ Technician _____________
Appearance: 10 µ ____
(color, 10 µ ____
uniformity, 2.5 µ ____
defects) 2.5 µ ____

****

Comments (weather, unusual conditions or problems) ________________
____
____
____
____

Figure 7

Dilution Sampler Field Data Sheet: 15 Minute and Intermittent Inspection

Plant ____

Stack ____

Run Number ____________________

Operator ______________________

Date __________________________ Time _____________________________

ΔP Checked and Corrected __________________

Chamber Velocity Checked and Corrected ______________

Stack Temperature _____°F _____°C

Ambient Temperature _____°F _____°C

Dilution Chamber Temperature _____°F _____°C

PISD Flows Checked and Corrected ___________

Comments ____

____

____

____

____

____

Figure 8

Dilution Sampler Field Data Sheet: Stop

Plant ____

Stack ____

Run Number ____________________ ΔP _________________________ "H2O

Operator ______________________ Chamber Velocity _____________ mps

Stop Date _____________________ Stop Time __________________________

Stack Temperature _____°F _____°C

Ambient Temperature _____°F _____°C

Dilution Chamber Temperature _____°F _____°C

Comments ____

____

____

____

____

____

Bulk Samples Collected ____

____

____

____

____

Appendix D
Dust Sampling and Laboratory Preparatory Procedures

Contents of Appendix D

Soil, Dust, and Bulk Material Sampling
Standard Operating Procedure

General Discussion

The objective of soil and bulk material sampling is to obtain representative samples of the dust likely to be entrained from various area sources. Criteria used to determine sampling locations include proximity to ambient sampling sites, accessibility, potential contribution to the ambient aerosol, and a desire to obtain a representation of a variety of soil and bulk material dust types.

Materials and Equipment

- Ziploc bags
- Large spatula
- Kaydry towels

Sample Collection

1. Clean the spatula with a Kaydry towel. Loosen the top layer of soil or bulky material with the spatula.

2. Using the spatula, transfer the loosened material to a new Ziploc bag, filling it about half-full. Seal the bag.

3. Record your initials, the date, sample ID number, location, description, and other comments on the data sheet.

4. Place the bagged sample and data sheet inside another Ziploc bag.

5. Hand-carry the samples back to the laboratory, log them in, and place in sample storage cabinet.

Example Data Sheet

Soil and Bulk Material Source Sample

Date ________________ Initials ______________________

Sample ID # _________________________

Location: ____

Comments: ____

Paved Road Dust Sampling

Standard Operating Procedures

General Discussion

The objective of paved road dust sampling is to obtain representative samples of the road dust likely to be entrained in the ambient aerosol. Criteria for sample location selection include proximity to ambient sampling sites, accessibility, potential contribution to the ambient aerosol, past studies, and a desire to obtain a representation of a variety of road dust types.

Materials and Equipment

- Road dust sampler, consisting of an enclosed high-volume motor and filter holder fitted with an adaptor cone, hose, brush, and shoulder strap.
- Glass fiber filters, 8 × 10 inches.
- Portable generator or other 115 VAC, 8A, 50/60 hz power source.
- Extension cord.
- Envelopes, about 7 × 10 inches.
- Small hand broom and dust pan.

Sample Collection With Road Dust Sampler

1. Clean the hose, brush, adapter cone, and filter-hold-down frame with the stream of exhaust air from the high-volume motor. Disassemble the hose sections and shake the flexible portion of the hose to help dislodge any dust in the system.

2. Place a clean filter on the wire mesh filter holder. Install the filter-hold-down frame, being careful to center the filter. Mount the adapter cone and tighten wing nuts moderately tight. Assemble the hose sections and brush.

3. Carry the sampler with shoulder strap, keeping the filter horizontal during sampling and until after the sample is unloaded.

4. Start sampler and vacuum the road from the center line to the edge of the normal traffic flow. Vacuum a section of road large enough to collect a layer of material about one-eighth to one-half inch thick. If not sampling a paved road, vacuum a representative portion of the source area.

5. Shut the sampler off and position for filter unloading. If the wind is blowing, it may be necessary to find a relatively calm area.

6. Unscrew the wing nuts and remove the adapter cone and filter hold down frame. Carefully fold filter into fourths, first lengthwise, then widthwise, so that none of the sample falls off the filter. Place folded filter into a Ziploc bag and seal.

Sample Collection With Hand Broom

1. In some cases, enough material is on the pavement to permit sample collection with a hand broom and dust pan. Simply sweep material from a representative portion of the source area into the dust pan and transfer the material collected into a Ziploc bag.

Sample Handling

1. Record project, date, sample ID number, initials, sampling location, and other comments on the data sheet. Record project, date, sample ID number, and initials on an envelope, place bagged sample and data sheet into the envelope, and seal.

2. Hand-carry the samples back to the laboratory, log them in, and place in the sample storage cabinet.

Example Data Sheet

Road Dust Source Sample

Project ______________________ Date ______________________

Sample ID ______________________ Initial ______________________

Location ____

Comments ____

Soil, Bulk Material and Road Dust Pre-Processing Procedures

1. **Completely mix samples before processing.** Numerous samples are collected in most cases and placed in sample bags to make a composite. No mixing is generally conducted in the field.

2. **Crush soil "clumps".** Some agricultural fields from which samples can be collected have been recently watered, hence the soil can be in agglomerates. These agglomerates break down and become wind-transportable with time.

3. **Grind road cinder material with mortar and pestle or ceramic ball mill.** Road cinder is crushed by tire wear. Grind before processing to simulate tire wear.

Soil, Bulk Material, and Road Dust Sample Drying and Sieving
Standard Operating Procedure

General Discussion

Bulk, soil, and road dust samples must be dried and size-segregated before resuspension. This procedure describes the drying and isolation of the $<38\mu$ fraction. Special care must be taken to prevent the spread of dust to any other sample handling or analysis area of the lab, and to prevent cross-contamination of the samples. Clean the work area and spatulas with methanol-dampened Chemwipe towels between each sample handling.

Materials and Equipment

- Set of Tyler-equivalent sieves, as indicated, with top and bottom pan.

USA No.	Opening (μ)
28	600
48	300
100	150
200	75
400	38

- Sieve shaker.
- Glass vials.
- Chemwipe disposable towels.
- Compressed air.
- Spatulas.
- Methanol.
- Wire brush.
- Drying surface, e.g., metal sheet.

Drying

1. Sample material should be spread on clean metal sheets for drying at ambient temperature in a clean and low-humidity environment. The drying period should be at least twenty-four hours. Once the material is dry, transfer it to a labeled Ziploc bag. The metal sheet should be cleaned with methanol and Chemwipe towels between samples.

Sieve Cleaning

1. This procedure should be done in a room dedicated to sieve cleaning, or outside, to prevent the spread of dust to sample-handling areas of the lab.

2. Use compressed air to blow away all loose dust. Pay particular attention to the screen, the screen-to-frame joint, and overlapping surfaces on the frame.

3. Carefully brush all loose dust from the wire mesh screen with a wire brush.

4. Wipe all loose dirt from the frame with a brush or Chemwipe towel. Clean both inside and outside, and below as well as above the screen.

5. Repeat steps 2 through 4 until the sieve is clean.

Sieving

1. Clean the bottom pan using a methanol-dampened Chemwipe towel and assemble the cleaned Tyler-equivalent sieves in the following order, from the bottom to the top: bottom pan, 400 mesh screen, 200 mesh screen, 100 mesh screen, 48 mesh screen, 28 mesh screen.

2. Select a sample from the sample storage cabinet for processing.

3. Remove the inner bag and sample data card from the outer bag. Set them aside for later use.

4. Transfer one-half of the contents of the sample bag to the sieve assembly; minimize the loss of fine dust by dumping the sample into the sieve slowly and carefully. Set the sample bag aside for later use.

5. Clean the top cover using a methanol-dampened Chemwipe towel and compressed air, and put the top cover into position on the sieve assembly.

6. Place the sieve assembly into position on the shaker. Press the locking tab down firmly onto the sieve assembly with one hand and tighten the locking tab screw with the other hand. Repeat for the other locking tab.

7. Start the shaker. If dust escapes from the sieve assembly, either the assembly is not mounted tightly enough, or there is foreign material between the mating surfaces of the sieves. Correct and restart if necessary. Using the built-in timer, run the shaker for one half-hour.

8. Remove the sieve assembly from the shaker and set on a clean Chemwipe towel. Remove the sieves and top cover as a unit from the bottom pan and set on a clean Chemwipe towel.

9. Using a clean spatula, transfer dust from the bottom pan to a new glass vial. Label the glass vial. If an inadequate amount of material was collected, run the sieve an additional half-hour. If an inadequate amount of material is still passed through the sieve, add the other one-half of the sample to the top sieve and repeat steps 4 through 9.

10. Transfer the material remaining in the 28, 48, 100, 200, and 400 mesh sieves carefully into the bottom pan; tapping lightly on the sieve frame will remove most of the material.

11. Transfer the material from the bottom pan to the original sample bag.

Soil, Bulk Material, and Road Dust Resuspension
Standard Operating Procedure

Note: Refer to the PISD Standard Operating Procedure for a discussion of the theory of operation behind the PISD sampler, which is identical with the resuspension chamber.

1. Weigh and load pre-weighed 47 mm Teflon filters into Millipore filter holders. Load pre-fired quartz filters and assign ID numbers corresponding to those of the Teflon filters (e.g.,for Teflon filter AT070334 is a quartz filter AQ070334). Standard 47 mm filter holders may be used, but multiple adapters are used to allow the filter holders to interface with resupension table, and the standard lock rings (**not** machined for thick filter packs) must be used to ensure that the filter holder assemblies don't leak. Drain disks are used behind both the Teflon and the quartz filters.

2. Clean and assemble the PISD sampler as outlined in the attached cleaning procedures. Two sets of procedures are followed. Between different samples, a more complete cleaning of the system is undertaken. Between replicates of the same samples, only those areas in the sampler where excessive accumulation of material would cause a problem are cleaned and the impactor plates are regreased.

3. Prepare the Resuspension Data Sheets (entitled "DRI Source Sample Resuspension Data Sheet A") for the sample to be resuspended. Fill in the date, sample ID and description, sampler configuration (1, 2.5, 10, TSP), impactor grease type ("M"), and filter description ("F + DD" for "filter plus drain disk"). Refer to the attached example Data Sheet.

4. Tap each vacuum gauge and record the resting vacuum readings on the Data Sheet.

5. A leak check is performed only every 10–12 samples. Perform the leak check by leaving the filter holder tubing disconnected and turning on the pump. After allowing the pumps to run several minutes, tap each vacuum gauge and record the values obtained on the Data Sheet. Values should be within two inches of mercury of each other to indicate no leak.

6. A flow check is performed whenever a leak check is performed. Leave the pumps running, install an empty filter holder in a sample tube, and connect the tubing to the quick-disconnect fitting on the bottom of the filter holder. Using one of the rubber sleeves normally used on the cyclone/manifold assembly, connect the calibrated rotameter assembly to the top of the tube. Hold the rotameter in a vertical position and record the reading obtained (to the nearest 0.5 unit) on the Data Sheet in the Leak Check area. Repeat for each of the eight sample tubes. Compare the readings obtained to the readings from the previous flow check. If the readings differ by more than 0.5, identify and correct the problem before using the sampler. **Note:** Keep fingers clear of the opening at the bottom of the

rotameter. **DO NOT** adjust the rotameter valve; the rotameter was calibrated with the valve at this setting. Keep the rotameter in a sealed Ziploc bag when not in use to keep it clean and accurate. Turn the pumps off when the checks are completed.

7. Select four Teflon filters, remove the top caps, and load them into the bottom of the sampling tubes by threading the multiple adapters into the appropriate fittings. The four size fractions appear on labels on the corresponding vacuum gauges. Record the filter ID numbers on the Data Sheet. **Note:** It is extremely important that the filter IDs, size fractions, and filter positions are correctly recorded on the data sheet. Repeat for the quartz filters.

8. Attach the quick-disconnect fittings to the fittings on the filter holders. **Note:** It is extremely important that the correct hoses are attached to the appropriate filter holders. This is because the flows through each of these hoses are regulated by a separate critical orifice which has been individually calibrated. Because the flows through each of the tubes are slightly different, the deposit mass on each of the fractions must be normalized to a common total sample volume in order for the size splits to be meaningful.

9. Record the elapsed time meter readings from the two pumps. Note that the readings are in minutes and contain five digits plus one digit to the right of the decimal point.

10. Turn each of the pumps on. Try to stagger turning the pumps on slightly to avoid overloading a circuit breaker, but avoid long delays between turning on the two pumps. Check that each of the vacuum gauges reads 19 or more inches of mercury.

11. Tap each of the eight sample vacuum gauges and check that the difference between them and the pump vacuum readings is at least 15 inches of mercury. This generally means that the sample vacuum readings should be no more than 4 inches of mercury. Indicate that each of the sample ports have sufficient vacuum drop by checking the column labeled "Init. Vacuum."

12. Load 1/3 to 1/2 of a large spatula scoop of the sample (dried and sieved to $<38\mu$) into the dust suspension flask. Avoid loading too much material at once or filters may be overloaded and deposits will be unstable. Too little material will take longer to collect a sufficient deposit on the filters for XRF analysis.

13. Place the rubber stopper snugly in the top of the suspension flask. The high pressure of the suspension pump will blow the stopper out if it is not tight. Remove the bolt in the top of the chamber and place the side arm of the flask into the tubing. **Note:** Avoid excessive twisting or pressure on the tubing. **Note:** Avoid pressing the side arm too far into the tubing or removal will be difficult. Turn on the

suspension pump for five to ten seconds. The idea is to suspend and blow out the material in the flask, but not to blow more dilution air into the chamber than necessary. Remove the flask, replace the bolt in the inlet tubing, and record the time that resuspension took place in the comments column of the Data Sheet.

14. Allow the PISD pumps to continue to run for four minutes. This allows the samplers to collect as much sample as possible before the dust settles out; this period of sampling also pulls enough air across the deposit on the filters to encourage the deposit to stick. Multiple resuspensions may be done between weighings provided the operator is reasonably sure the filters will not be overloaded.

15. After checking the eight vacuum gauges for proper pressure drop, turn the PISD pumps off, remove the TSP and 10μ Teflon filters, and visually and gravimetrically estimate the amount of the deposit. Optimum loading is 1-3mg of material in an even deposit. The TSP and 10μm size fractions will usually reach optimum deposits first when resuspending geological materials. The 1μ and 2.5μ size fractions will generally load much more slowly. Continue steps 10 and 12 through 15 until the TSP and 10μ filters are optimally loaded. **Note:** Filters should be covered and kept in a horizontal position when transported to the weighing area. Record the elapsed time reading for these filters when they are removed for the last time and check the "final vacuum" column on the data sheet if vacuum readings were within normal limits.

16. Record the final gross weights of the TSP and 10μ Teflon filters. Place those filters back in the tackle box. Remove and store the TSP and 10μ quartz filters as well. **Note:** Keep the filter holders and tackle boxes for all resuspended samples in a horizontal position at all times and minimize vibrating or jarring the filters. Because these filters are not oil-coated, particle loss is a real possibility, especially with larger particles.

17. Replace the TSP and 10μ Teflon filters with new filters. **Note:** Use pre-weighed TX series filters (Pallflex Teflon-coated quartz) and pre-fired quartz filters as replacements; do not use dummy filters. Record the ID numbers of these replacement filters on the Data Sheet.

18. Continue steps 8 through 17 until the 2.5μ and 1μ filters are optimally loaded. Take care that the TSP and 10μ filters are not so heavily loaded that the deposit is in danger of flaking or falling off or that the vacuum readings across these filters are greater than 4 inches Hg. The replacement TX filters will not be analyzed by XRF, but they will be weighed; deposits as much as 6 mg are acceptable provided the deposit is not in danger of falling off. As necessary, replace these filters after recording the final vacuum readings and elapsed time readings.

19. When the fine fraction filters have been optimally loaded, record the final vacuum readings, elapsed time readings, and filter weights.

20. Repeat the entire procedure for additional samples. Ensure that the PISD and resuspension parts are thoroughly cleaned between different samples. Replicates of the same soils require only that the PISD cyclones and impactors be cleaned.

Source Sample Resuspension Data Sheet

Resuspension Date/Time: ____________________
Source Sample ID: ____________________
Sample Type: ____________________
Sampler Configuration: ____________________
Impactor Grease Type: ____________________
Filter T Series: ____________________
Description: Q Series: ____________________

	Vacuum Readings			
Particle Size	At Rest (in. Hg)		Leak Check (in. Hg)	
	T Series	Q Series	T Series	Q Series
TSP				
PM_{10}				
$PM_{2.5}$				
$PM_{1.0}$				

Sampling Sequence	Sequence Code	PM Size (μ)	Filter Type	Filter ID	Start Time (minutes)	End Time (minutes)	Init. Vacuum (in. Hg)	Final Vacuum (in. Hg)	Comments
Initial	1	30	Teflon						
			Quartz						
		10	Teflon						
			Quartz						
		2.5	Teflon						
			Quartz						
		1.0	Teflon						
			Quartz						
First Replacement	2	30	Teflon						
			Quartz						
		10	Teflon						
			Quartz						
		2.5	Teflon						
			Quartz						
		1.0	Teflon						
			Quartz						
Second Replacement	3	30	Teflon						
			Quartz						
		10	Teflon						
			Quartz						
		2.5	Teflon						
			Quartz						
		1.0	Teflon						
			Quartz						
Third Replacement	4	30	Teflon						
			Quartz						
		10	Teflon						
			Quartz						
		2.5	Teflon						
			Quartz						
		1.0	Teflon						
			Quartz						

Cleaning The Resuspension Chamber
Between Replicates
Standard Operating Procedure

Note: A less-than-complete cleaning of the resuspension system is acceptable between replicates of the same sample. The following is a checklist for this level of cleaning:

Remove the masking tape sealing the resuspension chamber to the aluminum table.

Using a methanol-moistened Kimwipe, clean the inlet tubing at the top of the chamber. This is necessary because all the samples pass through this small area and the potential for cross-contamination is high.

Likewise, use a methanol-moistened Kimwipe to wipe clean the rubber stopper and glass tube that fit into the suspension flask.

Remove the chamber from the top of the table.

Using a large methanol-moistened Kimwipe, wipe the material from the top of the cyclone/manifold assembly. This will prevent large particles from falling into the 1 and 2.5μ sample tubes when the manifolds are removed.

Remove the cyclones from the manifolds, remove the dust cup, and blow the cyclones clean with compressed air. Replace the dust cup, taking care not to lay the cyclone on its side, thus reintroducing large particles from the dust cup into the cyclone.

Remove the manifolds and blow the inside clean with compressed air. Set the manifolds and cyclones to one side.

Wipe the tops of the four rain caps with a large methanol-moistened Kimwipe. Again, this is merely to remove dirt which might fall into the sample tubes while they are open.

Remove the rain caps on the two 10μ sample tubes.

Using the wooden dowel, press the six impactors from the sample tubes.

Using the large spatula, push small dry Kimwipes across the top of the impactor plates to remove the captured dirt and impactor grease.

Regrease the impactor plates using the type M apiezon grease. Make sure that the grease is spread into a smooth, thin layer. Take care to avoid getting grease into the impactor jet.

Using a methanol-moistened Kimwipe, wipe the secondary impaction material from the inside of the 10μ sample tubes.

Press the impactors into their appropriate sample tubes. Note that the sample tubes, impactors, cyclones, and manifolds are all etched with an ID number. Make sure that these parts are reassembled in their appropriate places.

Replace the rain caps and cyclone/manifold assemblies.

Replace the chamber on top of the table. Seal the bottom of the chamber to the table using the wide masking tape. Using compressed air, blow the suspension flask clean.

Cleaning The Resuspension Chamber
Between Samples
Standard Operating Procedures

Note: A more thorough cleaning of the resuspension system is necessary between different samples. The following is a checklist for this level of cleaning:

Remove the masking tape sealing the resuspension chamber to the aluminum table.

Using a methanol-moistened Kimwipe, clean the inlet tubing at the top of the chamber. This is necessary because all the samples pass through this small area and the potential for cross-contamination is high.

Likewise, use a methanol-moistened Kimwipe to wipe clean the rubber stopper and glass tube.

Remove the chamber from the top of the table.

Remove the filter from the top of the chamber. Wipe around the lip of this opening to remove the accumulation of dust in this area.

Turn the chamber over, and using compressed air, blow the dust from the inside of the walls. **Note:** This is the dirtiest part of the cleaning procedure, as it results in blown dust in the suspension room. Make sure that the balance doors are shut, that no sample bags are open, and that the tackle boxes containing filters are closed and latched. This operation must be done before the remaining parts of the resuspension system are cleaned and set on the counter.

Turn the chamber over and tape a clean 8" by 5" glass fiber filter across the opening.

Using a large methanol-moistened Kimwipe, wipe the material from the top of the cyclone/manifold assemblies. Also wipe the rubber connecting sleeves.

Remove the cyclones from the manifolds, remove the dust cup, and blow the cyclones clean with compressed air. Dump the dirt from the dust cap, rinse with methanol, and dry with a Kimwipe.

Remove the manifolds and blow the inside clean with compressed air. Set the manifolds and cyclones to one side.

Wipe the tops of the four rain caps with a large methanol-moistened Kimwipe.

Remove all four rain caps and blow the insides clean with compressed air; pay particular attention to the plastic connector which tends to attract dust electrostatically.

Using large methanol-moistened Kimwipes, wipe the outside of the sample tubes and the tops of the tri-flanges.

Using large methanol-moistened Kimwipes, wipe the dirt from the top of the resuspension table. Note that black aluminum oxide will continue to appear on the Kimwipes despite considerable cleaning. This is not a problem provided that all loose dirt is removed. The dust from around the table top should be removed to avoid the possibility that small air leaks around the bottom of the chamber will resuspend the dust and contaminate the new sample.

Using the wooden dowel, press the impactors from the sample tubes.

Using the large spatula, push small dry Kimwipes across the top of the impactor plates to remove the captured dirt and impactor grease.

Wipe the tops of the impactors with a dry Kimwipe to remove any loose dust. This is most usually most noticeable on the 10μ impactors.

Regrease the impactor plates using the type M apiezon grease. Make sure that the grease is spread into a smooth, thin layer. Take care to avoid getting grease into the impactor jet.

If the rubber O-rings on the impactors are dry, regrease with a **light** coating of silicon vacuum grease. Avoid over-greasing the O-rings.

Using a methanol-moistened Kimwipe, wipe the secondary impaction material from the inside of the 10μ sample tubes.

Press the impactors into their appropriate sample tubes. Note that the sample tubes, impactors, cyclones, and manifolds are all etched with an ID number. Make sure that these parts are reassembled in their appropriate places.

Replace the rain caps and cyclone/manifold assemblies.

Replace the chamber on top of the table. Seal the bottom of the chamber to the table using the wide masking tape.

Appendix E
Point and Area Sources (non-resuspended)
Filter Summaries

Filter Summary
Diesel Emissions: Wheeler Ridge Weigh Station, November 1987

Run #	Filter ID	Filter Type	Size	Sample Duration (min)	Calibrated Flow (lpm)	Total Volume (m^3)
1	AT070025	Teflon	< 1 μ	145.7	8.5	1.2
1	AT070026	Teflon	< 2.5 μ	145.7	9.2	1.3
1	AT070027	Teflon	< 10 μ	145.7	9.5	1.4
1	AT070028	Teflon	TSP	145.7	9.4	1.4
1	AQ070025	Quartz	< 1 μ	147.1	8.8	1.3
1	AQ070026	Quartz	< 2.5 μ	147.1	8.8	1.3
1	AQ070027	Quartz	< 10 μ	147.1	9.2	1.4
1	AQ070028	Quartz	TSP	147.1	9.2	1.4
2	AT070029	Teflon	< 1 μ	168.2	8.5	1.4
2	AT070031	Teflon	< 2.5 μ	168.2	9.2	1.5
2	AT070032	Teflon	< 10 μ	168.2	9.5	1.6
2	AT070033	Teflon	TSP	168.2	9.4	1.6
2	AQ070029	Quartz	< 1 μ	168.0	8.8	1.5
2	AQ070031	Quartz	< 2.5 μ	168.0	8.8	1.5
2	AQ070032	Quartz	< 10 μ	168.0	9.2	1.5
2	AQ070033	Quartz	TSP	168.0	9.2	1.5
3	AT070034	Teflon	< 1 μ	79.7	8.5	0.68
3	AT070035	Teflon	< 2.5 μ	79.7	9.2	0.73
3	AT070036	Teflon	< 10 μ	79.7	9.5	0.76
3	AT070037	Teflon	TSP	79.7	9.4	0.75
3	AQ070034	Quartz	< 1 μ	79.7	8.8	0.70
3	AQ070035	Quartz	< 2.5 μ	79.7	8.8	0.70
3	AQ070036	Quartz	< 10 μ	79.7	9.2	0.73
3	AQ070037	Quartz	TSP	79.7	9.2	0.73
4	AT070043	Teflon	< 1 μ	57.8	8.5	0.49
4	AT070044	Teflon	< 2.5 μ	57.8	9.2	0.53
4	AT070045	Teflon	< 10 μ	57.8	9.5	0.55
4	AT070046	Teflon	TSP	57.8	9.4	0.54
4	AQ070043	Quartz	< 1 μ	57.8	8.8	0.51
4	AQ070044	Quartz	< 2.5 μ	57.8	8.8	0.51
4	AQ070045	Quartz	< 10 μ	57.8	9.2	0.53
4	AQ070046	Quartz	TSP	57.8	9.2	0.53
5	AT070047	Teflon	< 1 μ	58.6	8.5	0.50
5	AT070048	Teflon	< 2.5 μ	58.6	9.2	0.54
5	AT070049	Teflon	< 10 μ	58.6	9.5	0.56
5	AT070052	Teflon	TSP	58.6	9.4	0.55
5	AQ070047	Quartz	< 1 μ	58.4	8.8	0.52
5	AQ070048	Quartz	< 2.5 μ	58.4	8.8	0.52
5	AQ070049	Quartz	< 10 μ	58.4	9.2	0.54
5	AQ070052	Quartz	TSP	58.4	9.2	0.54
6	AT070053	Teflon	< 1 μ	64.1	8.5	0.54
6	AT070054	Teflon	< 2.5 μ	64.1	9.2	0.59
6	AT070055	Teflon	< 10 μ	64.1	9.5	0.61
6	AT070056	Teflon	TSP	64.1	9.4	0.60
6	AQ070053	Quartz	< 1 μ	64.1	8.8	0.56
6	AQ070054	Quartz	< 2.5 μ	64.1	8.8	0.56
6	AQ070055	Quartz	< 10 μ	64.1	9.2	0.59
6	AQ070056	Quartz	TSP	64.1	9.2	0.59

Filter Summary
Ambient Background: Wheeler Ridge Weigh Station, November 1987

Run #	Filter ID	Filter Type	Size	Sample Duration (min)	Calibrated Flow (lpm)	Total Volume (m^3)
1	AT070021	Teflon	< 1 μ	547.8	8.1	4.4
1	AT070022	Teflon	< 2.5 μ	547.8	8.0	4.4
1	AT070023	Teflon	< 10 μ	547.8	8.4	4.6
1	AT070024	Teflon	TSP	547.8	10.5	5.8
1	AQ070021	Quartz	< 1 μ	547.8	8.5	4.7
1	AQ070022	Quartz	< 2.5 μ	547.8	8.2	4.5
1	AQ070023	Quartz	< 10 μ	547.8	8.1	4.4
1	AQ070024	Quartz	TSP	547.8	8.4	4.6
2	AT070038	Teflon	< 1 μ	271.5	8.1	2.2
2	AT070039	Teflon	< 2.5 μ	271.5	8.0	2.2
2	AT070041	Teflon	< 10 μ	271.5	8.4	2.3
2	AT070042	Teflon	TSP	271.5	10.5	2.9
2	AQ070038	Quartz	< 1 μ	271.4	8.5	2.3
2	AQ070039	Quartz	< 2.5 μ	271.4	8.2	2.2
2	AQ070041	Quartz	< 10 μ	271.4	8.1	2.2
2	AQ070042	Quartz	TSP	271.4	8.4	2.3

Filter Summary
Oil-Fired Steam Generator: Santa Fe Energy Unit 118, November 1987

Run #	Filter ID	Filter Type	Size	Sample Duration (min)	Calibrated Flow (lpm)	Total Volume (m^3)	Mean Temperatures		
							Ambient	Stack	DSS
1	AT070057	Teflon	< 1 μ	180.0	10.0	1.80	65° F/18° C	170° F/77° C	73° F/23° C
1	AT070058	Teflon	< 2.5 μ	180.0	10.0	1.80	65° F/18° C	170° F/77° C	73° F/23° C
1	AT070059	Teflon	< 10 μ	180.0	10.0	1.80	65° F/18° C	170° F/77° C	73° F/23° C
1	AT070061	Teflon	TSP	180.0	10.0	1.80	65° F/18° C	170° F/77° C	73° F/23° C
1	AQ070057	Quartz	< 1 μ	180.0	10.0	1.80	65° F/18° C	170° F/77° C	73° F/23° C
1	AQ070058	Quartz	< 2.5 μ	180.0	10.0	1.80	65° F/18° C	170° F/77° C	73° F/23° C
1	AQ070059	Quartz	< 10 μ	180.0	10.0	1.80	65° F/18° C	170° F/77° C	73° F/23° C
1	AQ070061	Quartz	TSP	180.0	10.0	1.80	65° F/18° C	170° F/77° C	73° F/23° C
2	AT070062	Teflon	< 1 μ	61.8	10.0	0.618	55° F/13° C	166° F/74° C	66° F/19° C
2	AT070063	Teflon	< 2.5 μ	61.8	10.0	0.618	55° F/13° C	166° F/74° C	66° F/19° C
2	AT070064	Teflon	< 10 μ	61.8	10.0	0.618	55° F/13° C	166° F/74° C	66° F/19° C
2	AT070065	Teflon	TSP	61.8	10.0	0.618	55° F/13° C	166° F/74° C	66° F/19° C
2	AQ070062	Quartz	< 1 μ	61.8	10.0	0.618	55° F/13° C	166° F/74° C	66° F/19° C
2	AQ070063	Quartz	< 2.5 μ	61.8	10.0	0.618	55° F/13° C	166° F/74° C	66° F/19° C
2	AQ070064	Quartz	< 10 μ	61.8	10.0	0.618	55° F/13° C	166° F/74° C	66° F/19° C
2	AQ070065	Quartz	TSP	61.8	10.0	0.618	55° F/13° C	166° F/74° C	66° F/19° C
3	AT070066	Teflon	< 1 μ	89.0	10.0	0.890	70° F/21° C	176° F/80° C	82° F/28° C
3	AT070067	Teflon	< 2.5 μ	89.0	10.0	0.890	70° F/21° C	176° F/80° C	82° F/28° C
3	AT070068	Teflon	< 10 μ	89.0	10.0	0.890	70° F/21° C	176° F/80° C	82° F/28° C
3	AT070069	Teflon	TSP	89.0	10.0	0.890	70° F/21° C	176° F/80° C	82° F/28° C
3	AQ070066	Quartz	< 1 μ	89.0	10.0	0.890	70° F/21° C	176° F/80° C	82° F/28° C
3	AQ070067	Quartz	< 2.5 μ	89.0	10.0	0.890	70° F/21° C	176° F/80° C	82° F/28° C
3	AQ070068	Quartz	< 10 μ	89.0	10.0	0.890	70° F/21° C	176° F/80° C	82° F/28° C
3	AQ070069	Quartz	TSP	89.0	10.0	0.890	70° F/21° C	176° F/80° C	82° F/28° C
4	AT070071	Teflon	< 1 μ	183.1	10.0	1.83	66° F/19° C	177° F/81° C	76° F/24° C
4	AT070072	Teflon	< 2.5 μ	183.1	10.0	1.83	66° F/19° C	177° F/81° C	76° F/24° C
4	AT070073	Teflon	< 10 μ	183.1	10.0	1.83	66° F/19° C	177° F/81° C	76° F/24° C
4	AT070074	Teflon	TSP	183.1	10.0	1.83	66° F/19° C	177° F/81° C	76° F/24° C
4	AQ070071	Quartz	< 1 μ	183.1	10.0	1.83	66° F/19° C	177° F/81° C	76° F/24° C
4	AQ070072	Quartz	< 2.5 μ	183.1	10.0	1.83	66° F/19° C	177° F/81° C	76° F/24° C
4	AQ070073	Quartz	< 10 μ	183.1	10.0	1.83	66° F/19° C	177° F/81° C	76° F/24° C
4	AQ070074	Quartz	TSP	183.1	10.0	1.83	66° F/19° C	177° F/81° C	76° F/24° C

Filter Summary
Wheeler Ridge Weigh Station and Santa Fe Energy Unit 118
Field Blanks, November 1987

Run #	Filter ID	Filter Type	Size
1	AT070030	Teflon	< 1 μ
1	AT070040	Teflon	< 2.5 μ
1	AT070051	Teflon	< 10 μ
1	AT070060	Teflon	TSP
1	AQ070030	Quartz	< 1 μ
1	AQ070040	Quartz	< 2.5 μ
1	AQ070051	Quartz	< 10 μ
1	AQ070060	Quartz	TSP

Filter Summary
Fireplace, Bakersfield Cordwood, Run 1, February 1988

Filter I.D.	Filter Type	Size Cut	Sample Duration*	Calibrated Flow	Total Volume
AT070079	Teflon	1 μ	35.8 minutes	10.0 lpm	358.0 liters
AT070080	Teflon	2.5 μ	35.8 minutes	10.0 lpm	358.0 liters
AT070081	Teflon	10 μ	35.8 minutes	10.0 lpm	358.0 liters
AT070082	Teflon	TSP	35.8 minutes	10.0 lpm	358.0 liters
AQ070079	Quartz	1 μ	32.6 minutes	10.0 lpm	326.0 liters
AQ070080	Quartz	2.5 μ	32.6 minutes	10.0 lpm	326.0 liters
AQ070081	Quartz	10 μ	32.6 minutes	10.0 lpm	326.0 liters
AQ070082	Quartz	TSP	32.6 minutes	10.0 lpm	326.0 liters
Ambient Background					
AT070075	Teflon	1 μ	401.1 minutes	8.9 lpm	3,569.8 liters
AT070076	Teflon	2.5 μ	401.1 minutes	8.9 lpm	3,569.8 liters
AT070077	Teflon	10 μ	401.1 minutes	9.3 lpm	3,730.2 liters
AT070078	Teflon	TSP	401.1 minutes	9.3 lpm	3,730.2 liters
AQ070075	Quartz	1 μ	401.0 minutes	8.6 lpm	3,449.5 liters
AQ070076	Quartz	2.5 μ	401.0 minutes	9.3 lpm	3,730.2 liters
AQ070077	Quartz	10 μ	401.0 minutes	9.6 lpm	3,850.6 liters
AQ070078	Quartz	TSP	401.0 minutes	9.5 lpm	3,810.5 liters

* The dilution sampler was run intermittently with one "on" cycle every 15 minutes. An "on" cycle for the Teflon filters was 52 seconds and for the quartz filters 49 seconds. The ambient background sampler ran continuously throughout the entire test.

Filter Summary
Fireplace, Bakersfield Cordwood, Run 2, February 1988

Filter I.D.	Filter Type	Size Cut	Sample Duration*	Calibrated Flow	Total Volume
AT070083	Teflon	1 μ	45.0 minutes	10.0 lpm	450 liters
AT070084	Teflon	2.5 μ	45.0 minutes	10.0 lpm	450 liters
AT070085	Teflon	10 μ	45.0 minutes	10.0 lpm	450 liters
AT070086	Teflon	TSP	45.0 minutes	10.0 lpm	450 liters
AQ070083	Quartz	1 μ	42.0 minutes	10.0 lpm	420 liters
AQ070084	Quartz	2.5 μ	42.0 minutes	10.0 lpm	420 liters
AQ070085	Quartz	10 μ	42.0 minutes	10.0 lpm	420 liters
AQ070086	Quartz	TSP	42.0 minutes	10.0 lpm	420 liters
Ambient Background					
AT070087	Teflon	1 μ	392.6 minutes	8.9 lpm	3,494.1 liters
AT070088	Teflon	2.5 μ	392.6 minutes	8.9 lpm	3,494.1 liters
AT070089	Teflon	10 μ	392.6 minutes	9.3 lpm	3,651.2 liters
AT070091	Teflon	TSP	392.6 minutes	9.3 lpm	3,651.2 liters
AQ070087	Quartz	1 μ	392.5 minutes	8.6 lpm	3,375.5 liters
AQ070088	Quartz	2.5 μ	392.5 minutes	9.3 lpm	3,650.3 liters
AQ070089	Quartz	10 μ	392.5 minutes	9.6 lpm	3,768.0 liters
AQ070091	Quartz	TSP	392.5 minutes	9.5 lpm	3,728.8 liters

* The dilution sampler was run intermittently with one "on" cycle every 15 minutes. An "on" cycle for the Teflon filters was 52 seconds and for the quartz filters 49 seconds. The ambient background sampler ran continuously throughout the entire test.

Filter Summary
Fireplace, Bakersfield Cordwood, Run 3, February 1988

Filter I.D.	Filter Type	Size Cut	Sample Duration*	Calibrated Flow	Total Volume
AT070096	Teflon	1 µ	43.9 minutes	10.0 lpm	439 liters
AT070097	Teflon	2.5 µ	43.9 minutes	10.0 lpm	439 liters
AT070098	Teflon	10 µ	43.9 minutes	10.0 lpm	439 liters
AT070099	Teflon	TSP	43.9 minutes	10.0 lpm	439 liters
AQ070096	Quartz	1 µ	41.2 minutes	10.0 lpm	412 liters
AQ070097	Quartz	2.5 µ	41.2 minutes	10.0 lpm	412 liters
AQ070098	Quartz	10 µ	41.2 minutes	10.0 lpm	412 liters
AQ070099	Quartz	TSP	41.2 minutes	10.0 lpm	412 liters
Ambient Background					
AT070101	Teflon	1 µ	386.1 minutes	8.9 lpm	3,436.3 liters
AT070102	Teflon	2.5 µ	386.1 minutes	8.9 lpm	3,436.3 liters
AT070103	Teflon	10 µ	386.1 minutes	9.3 lpm	3,590.7 liters
AT070104	Teflon	TSP	386.1 minutes	9.3 lpm	3,590.7 liters
AQ070101	Quartz	1 µ	386.3 minutes	8.6 lpm	3,322.2 liters
AQ070102	Quartz	2.5 µ	386.3 minutes	9.3 lpm	3,592.6 liters
AQ070103	Quartz	10 µ	386.3 minutes	9.6 lpm	3,708.5 liters
AQ070104	Quartz	TSP	386.3 minutes	9.5 lpm	3,669.9 liters

* The dilution sampler was run intermittently with one "on" cycle every 15 minutes. An "on" cycle for the Teflon filters was 52 seconds and for the quartz filters 49 seconds. The ambient background sampler ran continuously throughout the entire test.

Filter Summary
Fireplace, Mammoth Lakes Cordwood, Run 1, February 1988

Filter I.D.	Filter Type	Size Cut	Sample Duration*	Calibrated Flow	Total Volume
AT070105	Teflon	1 μ	47.4 minutes	10.0 lpm	474 liters
AT070106	Teflon	2.5 μ	47.4 minutes	10.0 lpm	474 liters
AT070107	Teflon	10 μ	47.4 minutes	10.0 lpm	474 liters
AT070108	Teflon	TSP	47.4 minutes	10.0 lpm	474 liters
AQ070105	Quartz	1 μ	44.3 minutes	10.0 lpm	443 liters
AQ070106	Quartz	2.5 μ	44.3 minutes	10.0 lpm	443 liters
AQ070107	Quartz	10 μ	44.3 minutes	10.0 lpm	443 liters
AQ070108	Quartz	TSP	44.3 minutes	10.0 lpm	443 liters
Ambient Background					
AT070109	Teflon	1 μ	414.0 minutes	8.9 lpm	3,684.6 liters
AT070111	Teflon	2.5 μ	414.0 minutes	8.9 lpm	3,684.6 liters
AT070112	Teflon	10 μ	414.0 minutes	9.3 lpm	3,850.2 liters
AT070113	Teflon	TSP	414.0 minutes	9.3 lpm	3,850.2 liters
AQ070109	Quartz	1 μ	413.9 minutes	8.6 lpm	3,559.5 liters
AQ070111	Quartz	2.5 μ	413.9 minutes	9.3 lpm	3,849.3 liters
AQ070112	Quartz	10 μ	413.9 minutes	9.6 lpm	3,973.4 liters
AQ070113	Quartz	TSP	413.9 minutes	9.5 lpm	3,932.1 liters

* The dilution sampler was run intermittently with one "on" cycle every 15 minutes. An "on" cycle for the Teflon filters was 52 seconds and for the quartz filters 49 seconds. The ambient background sampler ran continuously throughout the entire test.

Filter Summary
Fireplace, Mammoth Lakes Cordwood, Run 2, February 1988

Filter I.D.	Filter Type	Size Cut	Sample Duration*	Calibrated Flow	Total Volume
AT070114	Teflon	1 μ	43.2 minutes	10.0 lpm	432.0 liters
AT070115	Teflon	2.5 μ	43.2 minutes	10.0 lpm	432.0 liters
AT070116	Teflon	10 μ	43.2 minutes	10.0 lpm	432.0 liters
AT070117	Teflon	TSP	43.2 minutes	10.0 lpm	432.0 liters
AQ070114	Quartz	1 μ	40.5 minutes	10.0 lpm	405.0 liters
AQ070115	Quartz	2.5 μ	40.5 minutes	10.0 lpm	405.0 liters
AQ070116	Quartz	10 μ	40.5 minutes	10.0 lpm	405.0 liters
AQ070117	Quartz	TSP	40.5 minutes	10.0 lpm	405.0 liters
Ambient Background					
AT070118	Teflon	1 μ	380.1 minutes	8.9 lpm	3,382.9 liters
AT070119	Teflon	2.5 μ	380.1 minutes	8.9 lpm	3,382.9 liters
AT070121	Teflon	10 μ	380.1 minutes	9.3 lpm	3,534.9 liters
AT070122	Teflon	TSP	380.1 minutes	9.3 lpm	3,534.9 liters
AQ070118	Quartz	1 μ	380.3 minutes	8.6 lpm	3,270.6 liters
AQ070119	Quartz	2.5 μ	380.3 minutes	9.3 lpm	3,536.8 liters
AQ070121	Quartz	10 μ	380.3 minutes	9.6 lpm	3,650.9 liters
AQ070122	Quartz	TSP	380.3 minutes	9.5 lpm	3,612.9 liters

* The dilution sampler was run intermittently with one "on" cycle every 15 minutes. An "on" cycle for the Teflon filters was 52 seconds and for the quartz filters 49 seconds. The ambient background sampler ran continuously throughout the entire test.

Filter Summary
Fireplace, Mammoth Lakes Cordwood, Run 3, February 1988

Filter I.D.	Filter Type	Size Cut	Sample Duration*	Calibrated Flow	Total Volume
AT070123	Teflon	1 µ	32.4 minutes	10.0 lpm	324 liters
AT070124	Teflon	2.5 µ	32.4 minutes	10.0 lpm	324 liters
AT070125	Teflon	10 µ	32.4 minutes	10.0 lpm	324 liters
AT070126	Teflon	TSP	32.4 minutes	10.0 lpm	324 liters
AQ070123	Quartz	1 µ	29.3 minutes	10.0 lpm	293 liters
AQ070124	Quartz	2.5 µ	29.3 minutes	10.0 lpm	293 liters
AQ070125	Quartz	10 µ	29.3 minutes	10.0 lpm	293 liters
AQ070126	Quartz	TSP	29.3 minutes	10.0 lpm	293 liters
Ambient Background					
AT070127	Teflon	1 µ	366.8 minutes	8.9 lpm	3,264.5 liters
AT070128	Teflon	2.5 µ	366.8 minutes	8.9 lpm	3,264.5 liters
AT070129	Teflon	10 µ	366.8 minutes	9.3 lpm	3,411.2 liters
AT070131	Teflon	TSP	366.8 minutes	9.3 lpm	3,411.2 liters
AQ070127	Quartz	1 µ	367.0 minutes	8.6 lpm	3,156.2 liters
AQ070128	Quartz	2.5 µ	367.0 minutes	9.3 lpm	3,413.1 liters
AQ070129	Quartz	10 µ	367.0 minutes	9.6 lpm	3,523.2 liters
AQ070131	Quartz	TSP	367.0 minutes	9.5 lpm	3,486.5 liters

* The dilution sampler was run intermittently with one "on" cycle every 15 minutes. An "on" cycle for the Teflon filters was 52 seconds and for the quartz filters 49 seconds. The ambient background sampler ran continuously throughout the entire test.

Filter Summary
Woodstove, Mammoth Lakes Cordwood, Run 1, February 1988

Set	Filter I.D.	Filter Type	Size Cut	Sample Duration*	Calibrated Flow	Total Volume
1	AT070136	Teflon	1 μ	16.1 min.	10.0 lpm	161 liters
1	AT070137	Teflon	2.5 μ	16.1 min.	10.0 lpm	161 liters
1	AT070138	Teflon	10 μ	16.1 min.	10.0 lpm	161 liters
1	AT070139	Teflon	TSP	16.1 min.	10.0 lpm	161 liters
1	AQ070136	Quartz	1 μ	15.0 min.	10.0 lpm	150 liters
1	AQ070137	Quartz	2.5 μ	15.0 min.	10.0 lpm	150 liters
1	AQ070138	Quartz	10 μ	15.0 min.	10.0 lpm	150 liters
1	AQ070139	Quartz	TSP	15.0 min.	10.0 lpm	150 liters
2	AT070141	Teflon	1 μ	20.3 min.	10.0 lpm	203 liters
2	AT070142	Teflon	2.5 μ	20.3 min.	10.0 lpm	203 liters
2	AT070143	Teflon	10 μ	20.3 min.	10.0 lpm	203 liters
2	AT070144	Teflon	TSP	20.3 min.	10.0 lpm	203 liters
2	AQ070141	Quartz	1 μ	19.1 min.	10.0 lpm	191 liters
2	AQ070142	Quartz	2.5 μ	19.1 min.	10.0 lpm	191 liters
2	AQ070143	Quartz	10 μ	18.2 min.	10.0 lpm	182 liters
2	AQ070144	Quartz	TSP	18.2 min.	10.0 lpm	182 liters
Ambient Background						
A	AT070132	Teflon	1 μ	631.4 min.	8.9 lpm	5,619.5 liters
A	AT070133	Teflon	2.5 μ	631.4 min.	8.9 lpm	5,619.5 liters
A	AT070134	Teflon	10 μ	631.4 min.	9.3 lpm	5,872.0 liters
A	AT070135	Teflon	TSP	631.4 min.	9.3 lpm	5,872.0 liters
A	AQ070132	Quartz	1 μ	631.3 min.	8.6 lpm	5,429.2 liters
A	AQ070133	Quartz	2.5 μ	631.3 min.	9.3 lpm	5,871.1 liters
A	AQ070134	Quartz	10 μ	631.3 min.	9.6 lpm	6,060.5 liters
A	AQ070135	Quartz	TSP	631.3 min.	9.5 lpm	5,997.4 liters

* The dilution sampler was run intermittently with one "on" cycle every 15 minutes. An "on" cycle for the Teflon filters was 52 seconds and for the quartz filters 49 seconds. The ambient background sampler ran continuously throughout the entire test.

Filter Summary
Woodstove, Mammoth Lakes Cordwood, Run 2, February 1988

Filter I.D.	Filter Type	Size Cut	Sample Duration*	Calibrated Flow	Total Volume
AT070149	Teflon	1 μ	35.7 minutes	10.0 lpm	357 liters
AT070151	Teflon	2.5 μ	35.7 minutes	10.0 lpm	357 liters
AT070152	Teflon	10 μ	35.7 minutes	10.0 lpm	357 liters
AT070153	Teflon	TSP	35.7 minutes	10.0 lpm	357 liters
AQ070149	Quartz	1 μ	33.4 minutes	10.0 lpm	334 liters
AQ070151	Quartz	2.5 μ	33.4 minutes	10.0 lpm	334 liters
AQ070152	Quartz	10 μ	33.4 minutes	10.0 lpm	334 liters
AQ070153	Quartz	TSP	33.4 minutes	10.0 lpm	334 liters
Ambient Background					
AT070145	Teflon	1 μ	619.1 minutes	8.9 lpm	5,510.0 liters
AT070146	Teflon	2.5 μ	619.1 minutes	8.9 lpm	5,510.0 liters
AT070147	Teflon	10 μ	619.1 minutes	9.3 lpm	5,757.6 liters
AT070148	Teflon	TSP	619.1 minutes	9.3 lpm	5,757.6 liters
AQ070145	Quartz	1 μ	619.1 minutes	8.6 lpm	5,324.3 liters
AQ070146	Quartz	2.5 μ	619.1 minutes	9.3 lpm	5,757.6 liters
AQ070147	Quartz	10 μ	619.1 minutes	9.6 lpm	5,943.4 liters
AQ070148	Quartz	TSP	619.1 minutes	9.5 lpm	5,881.5 liters

* The dilution sampler was run intermittently with one "on" cycle every 15 minutes. An "on" cycle for the Teflon filters was 52 seconds and for the quartz filters 49 seconds. The ambient background sampler ran continuously throughout the entire test.

Filter Summary
Woodstove, Mammoth Lakes Cordwood, Run 3, February 1988

Filter I.D.	Filter Type	Size Cut	Sample Duration*	Calibrated Flow	Total Volume
AT070154	Teflon	1 μ	32.5 minutes	10.0 lpm	325 liters
AT070155	Teflon	2.5 μ	32.5 minutes	10.0 lpm	325 liters
AT070156	Teflon	10 μ	32.5 minutes	10.0 lpm	325 liters
AT070157	Teflon	TSP	32.5 minutes	10.0 lpm	325 liters
AQ070154	Quartz	1 μ	30.5 minutes	10.0 lpm	305 liters
AQ070155	Quartz	2.5 μ	30.5 minutes	10.0 lpm	305 liters
AQ070156	Quartz	10 μ	30.5 minutes	10.0 lpm	305 liters
AQ070157	Quartz	TSP	30.5 minutes	10.0 lpm	305 liters
Ambient Background					
AT070158	Teflon	1 μ	568.0 minutes	8.9 lpm	5,055.2 liters
AT070159	Teflon	2.5 μ	568.0 minutes	8.9 lpm	5,055.2 liters
AT070161	Teflon	10 μ	568.0 minutes	9.3 lpm	5,282.4 liters
AT070162	Teflon	TSP	568.0 minutes	9.3 lpm	5,282.4 liters
AQ070158	Quartz	1 μ	568.0 minutes	8.6 lpm	4,884.8 liters
AQ070159	Quartz	2.5 μ	568.0 minutes	9.3 lpm	5,282.4 liters
AQ070161	Quartz	10 μ	568.0 minutes	9.6 lpm	5,452.8 liters
AQ070162	Quartz	TSP	568.0 minutes	9.5 lpm	5,396.0 liters

* The dilution sampler was run intermittently with one "on" cycle every 15 minutes. An "on" cycle for the Teflon filters was 52 seconds and for the quartz filters 49 seconds. The ambient background sampler ran continuously throughout the entire test.

Filter Summary
DSS and PISD Field Blanks
Woodstove and Fireplace Tests, February 1988

Sampling System	Filter I.D.	Filter Type
Dilution tunnel	AT070090	Teflon
Dilution tunnel	AT070110	Teflon
Dilution tunnel	AT070120	Teflon
Dilution tunnel	AT070130	Teflon
Dilution tunnel	AQ070090	Quartz
Dilution tunnel	AQ070110	Quartz
Dilution tunnel	AQ070120	Quartz
Dilution tunnel	AQ070130	Quartz
PISD	AT070140	Teflon
PISD	AT070160	Teflon
PISD	AT070170	Teflon
PISD	AT070180	Teflon
PISD	AQ070140	Quartz
PISD	AQ070160	Quartz
PISD	AQ070170	Quartz
PISD	AQ070180	Quartz

FILTER SUMMARY
DIESEL EMISSIONS: SKI BUSES, MAMMOTH LAKES – FEBRUARY 1988

Run	Filter I.D.	Filter Type	Size	Sample Duration	Flow	Total Volume
1	AT07163	Teflon	TSP	24 min.	9.4 lpm	226 liters
1	AT07164	Teflon	< 10 μ	24 min.	9.5 lpm	228 liters
1	AT07165	Teflon	< 2.5 μ	24 min.	9.2 lpm	221 liters
1	AT07166	Teflon	< 1 μ	24 min.	8.5 lpm	204 liters
1	AQ07163	Quartz	TSP	24 min.	9.2 lpm	221 liters
1	AQ07164	Quartz	< 10 μ	24 min.	9.2 lpm	221 liters
1	AQ07165	Quartz	< 2.5 μ	24 min.	8.8 lpm	211 liters
1	AQ07166	Quartz	< 1 μ	24 min.	8.8 lpm	211 liters
2	AT07167	Teflon	TSP	103 min.	9.4 lpm	968 liters
2	AT07168	Teflon	< 10 μ	103 min.	9.5 lpm	978 liters
2	AT07169	Teflon	< 2.5 μ	103 min.	9.2 lpm	948 liters
2	AT07171	Teflon	< 1 μ	103 min.	8.5 lpm	876 liters
2	AQ07167	Quartz	TSP	103 min.	9.2 lpm	948 liters
2	AQ07168	Quartz	< 10 μ	103 min.	9.2 lpm	948 liters
2	AQ07169	Quartz	< 2.5 μ	103 min.	8.8 lpm	906 liters
2	AQ07171	Quartz	< 1 μ	103 min.	8.8 lpm	906 liters
3	AT07172	Teflon	TSP	131 min.	9.4 lpm	1231 liters
3	AT07173	Teflon	< 10 μ	131 min.	9.5 lpm	1244 liters
3	AT07174	Teflon	< 2.5 μ	131 min.	9.2 lpm	1205 liters
3	AT07175	Teflon	< 1 μ	131 min.	8.5 lpm	1114 liters
3	AQ07172	Quartz	TSP	131 min.	9.2 lpm	1205 liters
3	AQ07173	Quartz	< 10 μ	131 min.	9.2 lpm	1205 liters
3	AQ07174	Quartz	< 2.5 μ	131 min.	8.8 lpm	1153 liters
3	AQ07175	Quartz	< 1 μ	131 min.	8.8 lpm	1153 liters

FILTER SUMMARY

WHEAT AND BARLEY STUBBLE BURN – BAKERSFIELD AREA, JUNE/JULY 1988

Run #[1]	Filter ID	Filter Type	Size	Sample Duration (min)	Calibrated Flow (lpm)	Total Volume (l)
1	AT070996	Teflon	TSP	4.8	9.5	45.6
1	AT070995	Teflon	< 10 μ	4.8	9.6	46.1
1[2]	AT070994	Teflon	< 2.5 μ	4.8	9.3	44.6
1	AT070992	Teflon	< 1 μ	4.8	8.6	41.3
1	AQ070996	Quartz	TSP	5.3	8.5	45.1
1	AQ070995	Quartz	< 10 μ	5.3	8.2	43.5
1[2]	AQ070994	Quartz	< 2.5 μ	5.3	8.3	44.0
1	AQ070992	Quartz	< 1 μ	5.3	8.6	45.6
2	AT071001	Teflon	TSP	44.4	9.5	421.8
2	AT070999	Teflon	< 10 μ	44.4	9.6	426.2
2	AT070998	Teflon	< 2.5 μ	44.4	9.3	412.9
2	AT070997	Teflon	< 1 μ	44.4	8.6	381.8
2	AQ071001	Quartz	TSP	44.4	9.3	412.9
2	AQ070999	Quartz	< 10 μ	44.4	9.3	412.9
2	AQ070998	Quartz	< 2.5 μ	44.4	8.9	395.2
2	AQ070997	Quartz	< 1 μ	44.4	8.9	395.2
3	AT071005	Teflon	TSP	65.2	9.5	619.4
3	AT071004	Teflon	< 10 μ	65.2	9.6	625.9
3	AT071003	Teflon	< 2.5 μ	65.2	9.3	606.4
3	AT071002	Teflon	< 1 μ	65.2	8.6	560.7
3	AQ071005	Quartz	TSP	65.2	9.3	606.4
3	AQ071004	Quartz	< 10 μ	65.2	9.3	606.4
3	AQ071003	Quartz	< 2.5 μ	65.2	8.9	580.3
3	AQ071002	Quartz	< 1 μ	65.2	8.9	580.3

[1] Runs 1 and 2 were wheat stubble; run 3 was barley stubble.

[2] AT070993 and AQ070993 were not used because the Teflon filter ring was warped.

FILTER SUMMARY

WHEAT STUBBLE BURN – EL CENTRO AREA, JUNE/JULY 1988

Run #	Filter ID	Filter Type	Size	Sample Duration (min)	Calibrated Flow (lpm)	Total Volume (l)
1	AT071009	Teflon	TSP	77.0	9.3	716.1
1	AT071008	Teflon	< 10 μ	77.0	9.3	716.1
1	AT071007	Teflon	< 2.5 μ	77.0	8.9	685.3
1	AT071006	Teflon	< 1 μ	77.0	8.9	685.3
1	AQ071009	Quartz	TSP	77.1	8.5	655.4
1	AQ071008	Quartz	< 10 μ	77.1	8.2	632.2
1	AQ071007	Quartz	< 2.5 μ	77.1	8.3	639.9
1	AQ071006	Quartz	< 1 μ	77.1	8.6	663.1
2	AT071013	Teflon	TSP	26.0	9.3	241.8
2	AT071012	Teflon	< 10 μ	26.0	9.3	241.8
2	AT071011	Teflon	< 2.5 μ	26.0	8.9	231.4
2	AT071010	Teflon	< 1 μ	26.0	8.9	231.4
2	AQ071013	Quartz	TSP	25.9	8.5	220.2
2	AQ071012	Quartz	< 10 μ	25.9	8.2	212.4
2	AQ071011	Quartz	< 2.5 μ	25.9	8.3	215.0
2	AQ071010	Quartz	< 1 μ	25.9	8.6	222.7
3	AT071017	Teflon	TSP	15.5	9.3	144.2
3	AT071016	Teflon	< 10 μ	15.5	9.3	144.2
3	AT071014	Teflon	< 2.5 μ	15.5	8.9	138.0
3	AT071015	Teflon	< 1 μ	15.5	8.9	138.0
3	AQ071016	Quartz	TSP	15.4	8.5	130.9
3	AQ071017	Quartz	< 10 μ	15.4	8.2	126.3
3	AQ071014	Quartz	< 2.5 μ	15.4	8.3	127.8
3	AQ071015	Quartz	< 1 μ	15.4	8.6	132.4

FILTER SUMMARY

WHEAT STUBBLE BURN – VISALIA AREA, JUNE/JULY 1988

Run #	Filter ID	Filter Type	Size	Sample Duration (min)	Calibrated Flow (lpm)	Total Volume (l)
1	AT071021	Teflon	TSP	36.5	9.3	339.5
1	AT071020	Teflon	< 10 μ	36.5	9.3	339.5
1	AT071019	Teflon	< 2.5 μ	36.5	8.9	324.9
1	AT071018	Teflon	< 1 μ	36.5	8.9	324.9
1	AQ071021	Quartz	TSP	36.5	8.5	310.3
1	AQ071020	Quartz	< 10 μ	36.5	8.2	299.3
1	AQ071019	Quartz	< 2.5 μ	36.5	8.3	303.0
1	AQ071018	Quartz	< 1 μ	36.5	8.6	313.9
2	AT071025	Teflon	TSP	21.9	9.3	203.7
2	AT071024	Teflon	< 10 μ	21.9	9.3	203.7
2	AT071023	Teflon	< 2.5 μ	21.9	8.9	194.9
2	AT071022	Teflon	< 1 μ	21.9	8.9	194.9
2	AQ071025	Quartz	TSP	21.9	8.5	186.2
2	AQ071024	Quartz	< 10 μ	21.9	8.2	179.6
2	AQ071023	Quartz	< 2.5 μ	21.9	8.3	181.8
2 [1]	AQ071022	Quartz	< 1 μ	21.9	8.6	188.3
3 [2]	AT071029	Teflon	TSP	17.4	9.3	161.8
3	AT071028	Teflon	< 10 μ	17.4	9.3	161.8
3	AT071027	Teflon	< 2.5 μ	17.4	8.9	154.9
3	AT071026	Teflon	< 1 μ	17.4	8.9	154.9
3	AQ071029	Quartz	TSP	17.4	8.5	147.9
3	AQ071028	Quartz	< 10 μ	17.4	8.2	142.9
3	AQ071027	Quartz	< 2.5 μ	17.4	8.3	144.4
3	AQ071026	Quartz	< 1 μ	17.4	8.6	149.6
4	AT071058	Teflon	TSP	47.0	8.7	408.9
4	AT071057	Teflon	< 10 μ	47.0	8.6	404.2
4	AT071056	Teflon	< 2.5 μ	47.0	10.5	493.5
4	AT071055	Teflon	< 1 μ	47.0	9.1	427.7
4	AQ071058	Quartz	TSP	47.0	9.5	446.5
4	AQ071057	Quartz	< 10 μ	47.0	9.6	451.2
4	AQ071056	Quartz	< 2.5 μ	47.0	9.3	437.1
4	AQ071055	Quartz	< 1 μ	47.0	8.6	404.2

[1] Two filters stuck together; used and loaded as one.

[2] Filter not connected; sample invalid.

FILTER SUMMARY

WHEAT STUBBLE BURN – STOCKTON AREA, JUNE/JULY 1988

Run #	Filter ID	Filter Type	Size	Sample Duration (min)	Calibrated Flow (lpm)	Total Volume (l)
1	AT071037	Teflon	TSP	24.3	9.3	226.0
1	AT071036	Teflon	< 10 μ	24.3	9.3	226.0
1	AT071035	Teflon	< 2.5 μ	24.3	8.9	216.3
1	AT071034	Teflon	< 1 μ	24.3	8.9	216.3
1	AQ071037	Quartz	TSP	24.3	8.5	206.6
1	AQ071036	Quartz	< 10 μ	24.3	8.2	199.3
1	AQ071035	Quartz	< 2.5 μ	24.3	8.3	201.7
1	AQ071034	Quartz	< 1 μ	24.3	8.6	209.0
2	AT071041	Teflon	TSP	63.9	9.3	594.3
2	AT071040	Teflon	< 10 μ	63.9	9.3	594.3
2	AT071039	Teflon	< 2.5 μ	63.9	8.9	568.7
2	AT071038	Teflon	< 1 μ	63.9	8.9	568.7
2	AQ071041	Quartz	TSP	64.0	8.5	544.0
2	AQ071040	Quartz	< 10 μ	64.0	8.2	524.8
2	AQ071039	Quartz	< 2.5 μ	64.0	8.3	531.2
2	AQ071038	Quartz	< 1 μ	64.0	8.6	550.4
3	AT071045	Teflon	TSP	73.2	9.3	680.8
3	AT071044	Teflon	< 10 μ	73.2	9.3	680.8
3	AT071043	Teflon	< 2.5 μ	73.2	8.9	651.5
3	AT071042	Teflon	< 1 μ	73.2	8.9	651.5
3	AQ071045	Quartz	TSP	73.2	8.5	622.2
3	AQ071044	Quartz	< 10 μ	73.2	8.2	600.2
3	AQ071043	Quartz	< 2.5 μ	73.2	8.3	607.6
3	AQ071042	Quartz	< 1 μ	73.2	8.6	629.5

FILTER SUMMARY
VISALIA AREA DAIRY, JUNE/JULY 1988

Run #	Filter ID	Filter Type	Size	Sample Duration (min)	Calibrated Flow (lpm)	Total Volume (l)
1	AT071049	Teflon	TSP	1407.6	10.6	14920.6
1	AT071048	Teflon	< 10 μ	1407.6	8.5	11964.6
1	AT071047	Teflon	< 2.5 μ	1407.6	8.1	11401.6
1	AT071046	Teflon	< 1 μ	1407.6	8.2	11542.3
1	AQ071049	Quartz	TSP	1407.6	9.5	13372.2
1	AQ071048	Quartz	< 10 μ	1407.6	9.6	13513.0
1	AQ071047	Quartz	< 2.5 μ	1407.6	9.3	13090.7
1	AQ071046	Quartz	< 1 μ	1407.6	8.6	12105.4
2	AT071054	Teflon	TSP	1324.4	10.6	14038.6
2	AT071053	Teflon	< 10 μ	1324.4	8.5	11257.4
2	AT071052	Teflon	< 2.5 μ	1324.4	8.1	10727.6
2	AT071051	Teflon	< 1 μ	1324.4	8.2	10860.1
2	AQ071054	Quartz	TSP	1324.5	9.5	12582.8
2	AQ071053	Quartz	< 10 μ	1324.5	9.6	12715.2
2	AQ071052	Quartz	< 2.5 μ	1324.5	9.3	12317.9
2	AQ071051	Quartz	< 1 μ	1324.5	8.6	11390.7
3	AT071062	Teflon	TSP	928.9	10.6	9846.3
3	AT071061	Teflon	< 10 μ	928.9	8.5	7895.7
3	AT071060	Teflon	< 2.5 μ	928.9	8.1	7524.1
3	AT071059	Teflon	< 1 μ	928.9	8.2	7617.0
3	AQ071062	Quartz	TSP	928.7	9.5	8822.7
3	AQ071061	Quartz	< 10 μ	928.7	9.6	8915.5
3	AQ071060	Quartz	< 2.5 μ	928.7	9.3	8636.9
3	AQ071059	Quartz	< 1 μ	928.7	8.6	7986.8

FILTER SUMMARY

FRESNO CONSTRUCTION – HIGHWAY 40, JUNE/JULY 1988

Run #	Filter ID	Filter Type	Size	Sample Duration (min)	Calibrated Flow (lpm)	Total Volume (l)
1	AT071066	Teflon	TSP	439.2	10.6	4655.5
1	AT071065	Teflon	< 10 μ	439.2	8.5	3733.2
1	AT071065	Teflon	< 2.5 μ	439.2	8.1	3557.5
1	AT071063	Teflon	< 1 μ	439.2	8.2	3601.4
1	AQ071066	Quartz	TSP	439.0	9.5	4170.5
1	AQ071065	Quartz	< 10 μ	439.0	9.6	4214.4
1	AQ071064	Quartz	< 2.5 μ	439.0	9.3	4082.7
1	AQ071063	Quartz	< 1 μ	439.0	8.6	3775.4
2	AT071070	Teflon	TSP	439.1	9.3	4083.6
2	AT071069	Teflon	< 10 μ	439.1	9.3	4083.6
2	AT071068	Teflon	< 2.5 μ	439.1	8.9	3908.0
2	AT071067	Teflon	< 1 μ	439.1	8.9	3908.0
2	AQ071070	Quartz	TSP	438.9	8.5	3730.7
2	AQ071069	Quartz	< 10 μ	438.9	8.2	3599.0
2	AQ071068	Quartz	< 2.5 μ	438.9	8.3	3642.9
2	AQ071067	Quartz	< 1 μ	438.9	8.6	3774.5
3	AT071074	Teflon	TSP	128.0	10.6	1356.8
3	AT071073	Teflon	< 10 μ	128.0	8.5	1088.0
3	AT071072	Teflon	< 2.5 μ	128.0	8.1	1036.8
3	AT071071	Teflon	< 1 μ	128.0	8.2	1049.6
3	AQ071074	Quartz	TSP	128.0	9.5	1216.0
3	AQ071073	Quartz	< 10 μ	128.0	9.6	1228.8
3	AQ071072	Quartz	< 2.5 μ	128.0	9.3	1190.4
3	AQ071071	Quartz	< 1 μ	128.0	8.6	1100.8

FILTER SUMMARY
DILUTION SAMPLING SYSTEM (DSS) FIELD BLANK, JUNE/JULY 1988

Run #	Filter ID	Filter Type	Size
1	AT070991	Teflon	$< 1\ \mu$
1	AT070990	Teflon	$< 2.5\ \mu$
1	AT070989	Teflon	$< 10\ \mu$
1	AT070988	Teflon	TSP
1	AQ070991	Quartz	$< 1\ \mu$
1	AQ070990	Quartz	$< 2.5\ \mu$
1	AQ070989	Quartz	$< 10\ \mu$
1	AQ070988	Quartz	TSP

FILTER SUMMARY

OIL-FIRED STEAM GENERATOR: CHEVRON RACETRACK STEAM PLANT, JUNE/JULY 1988

Run #	Filter ID	Filter Type	Size	Sample Duration (min)	Calibrated Flow (lpm)	Total Volume (l)	Mean Temperatures Ambient	Mean Temperatures Stack	Mean Temperatures DSS
1	AT070979	Teflon	TSP	158.9	10.0	1589.0	86°F/30°C	130°F/54°C	104°F/40°C
1	AT070978[1]	Teflon	< 10 μ	158.9	10.0	1589.0	86°F/30°C	130°F/54°C	104°F/40°C
1	AT070977	Teflon	< 2.5 μ	158.9	10.0	1589.0	86°F/30°C	130°F/54°C	104°F/40°C
1	AT070976	Teflon	< 1 μ	158.9	10.0	1589.0	86°F/30°C	130°F/54°C	104°F/40°C
1	AQ070979	Quartz	TSP	158.8	10.0	1588.0	86°F/30°C	130°F/54°C	104°F/40°C
1	AQ070978	Quartz	< 10 μ	158.8	10.0	1588.0	86°F/30°C	130°F/54°C	104°F/40°C
1	AQ070977	Quartz	< 2.5 μ	158.8	10.0	1588.0	86°F/30°C	130°F/54°C	104°F/40°C
1	AQ070976	Quartz	< 1 μ	158.8	10.0	1588.0	86°F/30°C	130°F/54°C	104°F/40°C
2	AT070983	Teflon	TSP	135.1	10.0	1351.0	93°F/34°C	129°F/54°C	104°F/40°C
2	AT070982	Teflon	< 10 μ	135.1	10.0	1351.0	93°F/34°C	129°F/54°C	104°F/40°C
2	AT070981	Teflon	< 2.5 μ	135.1	10.0	1351.0	93°F/34°C	129°F/54°C	104°F/40°C
2	AT070980	Teflon	< 1 μ	135.1	10.0	1351.0	93°F/34°C	129°F/54°C	104°F/40°C
2	AQ070983	Quartz	TSP	135.2	10.0	1352.0	93°F/34°C	129°F/54°C	104°F/40°C
2	AQ070982	Quartz	< 10 μ	135.2	10.0	1352.0	93°F/34°C	129°F/54°C	104°F/40°C
2	AQ070981	Quartz	< 2.5 μ	135.2	10.0	1352.0	93°F/34°C	129°F/54°C	104°F/40°C
2	AQ070980	Quartz	< 1 μ	135.2	10.0	1352.0	93°F/34°C	129°F/54°C	104°F/40°C
3	AT070987	Teflon	TSP	149.0	10.0	1490.0	74°F/23°C	128°F/53°C	94°F/34°C
3	AT070986	Teflon	< 10 μ	149.0	10.0	1490.0	74°F/23°C	128°F/53°C	94°F/34°C
3	AT070985	Teflon	< 2.5 μ	149.0	10.0	1490.0	74°F/23°C	128°F/53°C	94°F/34°C
3	AT070984	Teflon	< 1 μ	149.0	10.0	1490.0	74°F/23°C	128°F/53°C	94°F/34°C
3	AQ070987	Quartz	TSP	148.9	10.0	1489.0	74°F/23°C	128°F/53°C	94°F/34°C
3	AQ070986	Quartz	< 10 μ	148.9	10.0	1489.0	74°F/23°C	128°F/53°C	94°F/34°C
3	AQ070985	Quartz	< 2.5 μ	148.9	10.0	1489.0	74°F/23°C	128°F/53°C	94°F/34°C
3	AQ070984	Quartz	< 1 μ	148.9	10.0	1489.0	74°F/23°C	128°F/53°C	94°F/34°C

---

[1] Filter dropped while unloading

FILTER SUMMARY
PARALLEL IMPACTOR SAMPLING DEVICE (PISD) FIELD BLANK, JUNE/JULY 1988

Run #	Filter ID	Filter Type	Size
1	AT071030	Teflon	$< 1\ \mu$
1	AT071031	Teflon	$< 2.5\ \mu$
1	AT071032	Teflon	$< 10\ \mu$
1	AT071033	Teflon	TSP
1	AQ071030	Quartz	$< 1\ \mu$
1	AQ071031	Quartz	$< 2.5\ \mu$
1	AQ071032	Quartz	$< 10\ \mu$
1	AQ071033	Quartz	TSP

Appendix F
Analytical Quality Assurance Plan

QUALITY ASSURANCE PROJECT PLAN
for the
DETERMINATION OF PARTICLE SIZE DISTRIBUTIONS AND CHEMICAL COMPOSITION OF PARTICULATE MATTER FROM SELECTED SOURCES IN CALIFORNIA

DRI Document No. 8069.1F1
September 28, 1987

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1.0 INTRODUCTION

This Quality Assurance Project Plan outlines the laboratory analytical methods which will be followed to specify the precision, accuracy, and validity of measurement values acquired in the "Determination of Particle Size Distribution and Chemical Compositions of Particulate Matter from Selected Sources in California". The objectives of this plan are:

- To prescribe analytical procedures which will be followed in the measurement process.
- To set data quality goals and reporting structures.
- To identify the quality assurance responsibilities of project participants.

This plan is structured according to the "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" which was issued by the U.S. Environmental Protection Agency in December, 1980 (U.S. EPA, 1980). The laboratory procedures used in this project have been developed by the California Air Resources Board (ARB) and the Energy and Environmental Engineering Center (EEEC) of the Desert Research Institute (DRI). These procedures are described briefly in the following sections and will be included in their entirety in an appendix to the final report for this project.

1.1 Project Description

Significant portions of the San Joaquin Valley, Southeast Desert, and Great Basin Valley air basins of California are expected to be in non-attainment of the National Ambient Air Quality PM_{10} standard which was recently promulgated by the U.S. Environmental Protection Agency (Federal Register, 1987a, 1987b, 1987c, 1987d). The California Air Resources Board expects to use source and receptor modeling techniques (Pace and Watson, 1987; Axetell and Watson, 1987) to attribute ambient PM_{10} concentrations to pollutant sources. The source attributions and emissions rates in each size sub-fraction of PM_{10} will be used for the preparation revisions to the State Implementation Plan for suspended particulate matter.

The objectives of this project are:

- To identify source sampling periods and locations which represent chemical source profiles as they would be perceived at PM_{10} receptors.

- To obtain representative samples of these particulate emissions in less than 1 μm, 1 to 2.5 μm, 2.5 to 10 μm, and greater than 10 μm particle size ranges.
- To chemically characterize these samples for species which will allow their identification in PM_{10} receptor samples.
- To document the source characterization methods, the source operating parameters, and the accuracy, precision, and validity of source composition data.
- To create a source profile data base which is compatible with existing source libraries, emissions inventories, and PM_{10} assessment models.

A variety of sources such as geological material (i.e. agricultural tillage, paved/unpaved road dust, wind blown dust), vegetative combustion (i.e. forest fires, agricultural burning, wood stoves and fireplaces), vehicular diesel combustion, oil-field steamers and internal combustion engines are to be sampled. The laboratory operations relevant to source sampling and analysis are:

- Substrate Preparation and Sample Chain-of-Custody
- Bulk Sample Resuspension in Four Size Ranges
- Gravimetric Analysis for Mass
- X-Ray Fluorescence Analysis for Elements
- Ion Chromatographic Analysis for Sulfate and Nitrate
- Atomic Absorption Spectrophotometric Analysis for Soluble Sodium and Potassium
- Thermal/Optical Combustion Analysis for Organic, Elemental and Carbonate Carbon

A flow diagram of the source sampling and chemical analysis measurement process is presented in Figure 1-1. Up to 150 source samples in the four specified size fractions will be taken on Teflon-membrane and quartz-fiber filter substrates. These particle deposits will be analyzed following the procedures specified in this plan.

1.2 Project Organization and Responsibility

A quality assurance (QA) officer has been appointed within DRI's Energy and Environmental Engineering Center and reports directly to the Executive Director of this Center. The QA officer determines the extent and methods of quality assurance to be applied to each project, estimates the level of effort involved in this quality assurance, identifies the appropriate personnel to perform QA tasks, and ascertains that these tasks are budgeted and carried out as part of the measurement process.

The QA officer also develops and carries out the QA project plan. The results of these QA efforts are summarized by the QA officer and included in the final report without modification by the project co-principal investigators. The QA officer has the authority to state the results of QA tasks without further review within DRI. The co-principal investigators of the project may take issue with the QA officer's conclusions, and a summary of these issues may be appended to the formal QA report. The QA officer may call upon expertise from other groups within or outside of DRI in order to complete his QA functions.

Drs. James Houck and Judith Chow are co-principal investigators for this project. Dr. John Watson is quality assurance officer. Mr. Clifton Frazier is laboratory supervisor. Chemical analysis and data processing activities will be overseen by both Mr. Clifton Frazier and Dr. Judith Chow. Figure 1-2 presents the project organization chart.

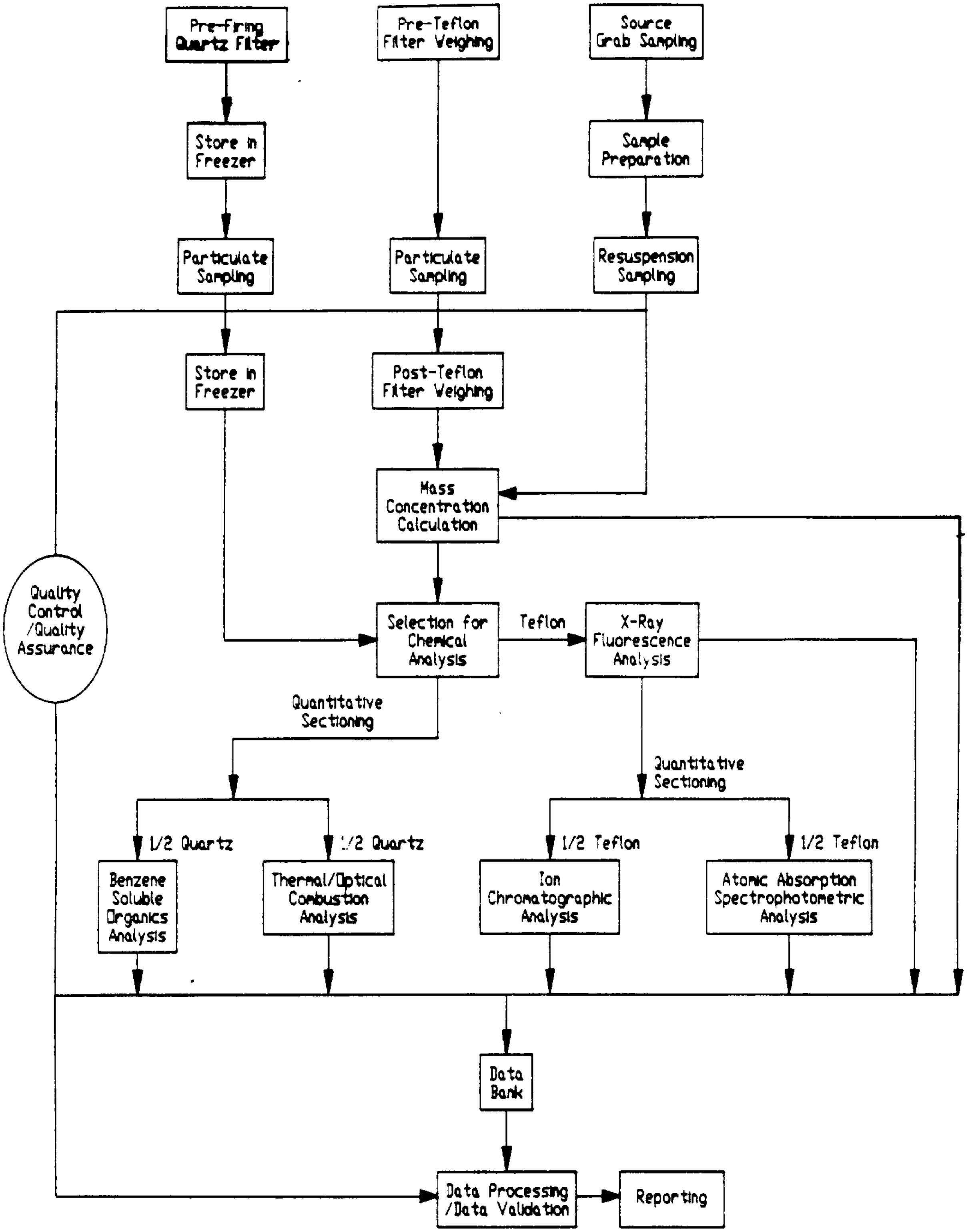

Figure 1-1. Flow Diagram of the ARB Source Sampling and Analysis Techniques

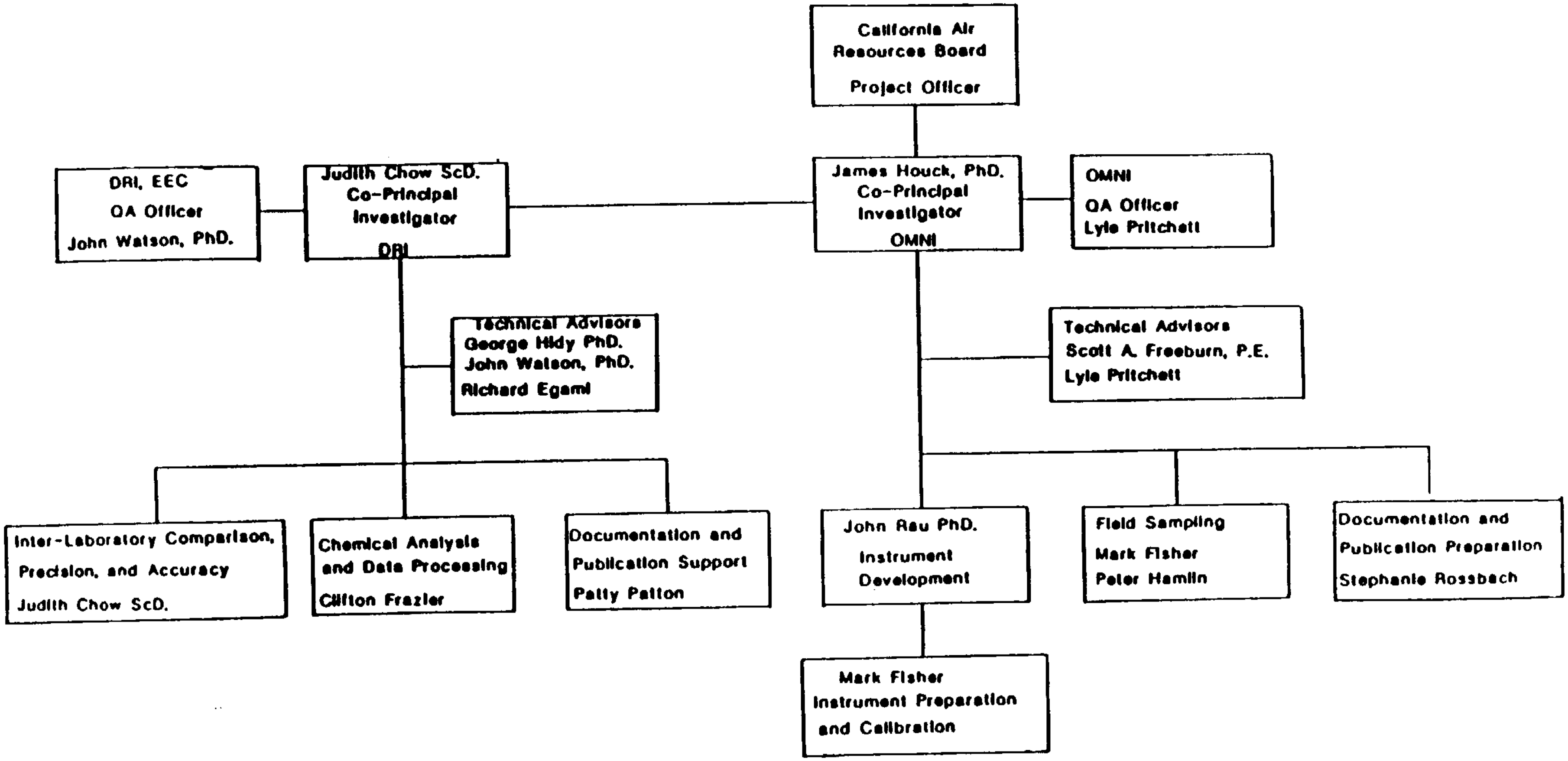

Figure 1-2. Project Management Organization for the Determination of Particle Size Distribution and Chemical Compositions of Particulate Matter from Selected Sources in California

2.0 PRECISION, ACCURACY, AND VALIDITY OBJECTIVES

Every measurement consists of four attributes: a value, a precision, an accuracy, and a validity (e.g., Hidy, 1985). The source sampling and analysis methods described in this plan are used to obtain the value. Quality control (QC) and quality assurance (QA) are the complementary parts of the measurement process which provide the precision, accuracy, and validity estimates, and guarantee that these attributes are within acceptable ranges. Precision and accuracy are quantitative and can be estimated for in situ and laboratory measurements from replicate analyses and audits as described in Mueller and Hidy et al. (1983) and Watson et al. (1983). Measurement validity is more qualitative, and its specification is variable-dependent.

Two types of validity are associated with each measurement process. The first is measurement method validity, which quantifies the effects of deviations from measurement method assumptions that are likely to occur in practical sampling and analysis. The second type of validity is sample validity which identifies the significant deviations from measurement method assumptions that occurred for a specific sample.

An important, but often neglected, aspect of QC is the feedback from the QC results back to the routine operations. Several past experiences in major air quality studies have found correctable biases as a result of quality audits, but these corrections were not applied to the data base. In other instances, QC performance tests were dutifully performed, but their results were not evaluated in a manner which as timely enough to identify and correct the problems they revealed. The laboratory manager will review all QC data as soon as it becomes available, and will initiate and document changes to the data base when they are needed.

The following activities are carried out as part of the quality assurance program:

- Issue, verify, and periodically revise standard operating procedures (SOPs) for each measurement process.
- Provide training in SOPs to measurement staff.
- Implement calibration, performance testing, maintenance, and data validation portions of each SOP. Implement auditing and interlaboratory comparison portions of each SOP.
- Conduct periodic systems and performance audits.
- Investigate concerns regarding measurement method validity and recommend corrective actions.

- Assure the completion of QA procedures within the project specifications.
- Document the accuracy, precision, and validity of each measurement.

The precision, accuracy, and validity objectives for this project are:

- Obtain analytical precisions within ± 10% for chemical species which are present at levels greater than ten times lower quantifiable limits.
- Obtain accuracies within ± 10% as determined from interlaboratory comparisons and analyses of standard reference materials.
- Obtain in excess of ± 95% valid analyses for the measured species.

3.0 SAMPLE HANDLING AND CHAIN-OF-CUSTODY

Eight major tasks are required to fulfill the laboratory operations of this project which cover the entire lifespan of a sample from acquisiton of sampling media through sample archival. They are:

- Substrate procurement and acceptance testing.
- Preparation, labeling, and shipment of sample media (plus associated supplies) to source samplers.
- Receipt and log-in of samples from source samplers.
- Bulk sample resuspension.
- Extraction and storage prior to analysis.
- Chemical analyses.
- Data processing and data validation.
- Archival of samples.

The following sections describe the sample handling and documentation that will be applied to all the source samples acquired from this project.

3.1 Acceptance Testing of Filter Substrates

Filters are procured from one vendor in a single lot whenever it is practical. At least one filter from every 100 is analyzed for all species to verify that pre-established specifications have been met. All filters are inspected for imperfections. Rejected filters are returned for credit.

Testing of sample media continues throughout the course of the project. In addition to 5% laboratory blanks, field blank samples are analyzed at a rate of 5% of all shipments to the field.

3.2. Pre-firing of Quartz-fiber Filters

Excessive organic and elemental carbon levels on quartz-fiber filters raise the lower quantifiable limits (LQLs) of organic and elemental carbon measurements. These blank carbon levels can be lowered from 1 to 3 $\mu g/cm^2$ to less than 0.5 $\mu g/cm^2$ by baking the filters at a high temperature prior to sampling (Fung, 1986). Baking at 400 $^{\circ}C$ decreases the organic carbon content, but it is insufficient to oxidize elemental carbon to CO_2. A temperature of approximately 700 to 800 °C is required to adequately remove both organic and elemental carbon from quartz-fiber filter substrates.

In order to obtain a lower quantifiable limit of 0.5 $\mu g/cm^2$, quartz-fiber filters are heated for at least three hours at 700 to 800 °C. A sample of each batch of 100 pre-fired filters is tested for carbon blank levels prior to sampling, and sets of filters which fail this test (i.e. having excessive blank levels) are re-fired or rejected. All pre-fired filters are stored in a freezer prior to sampling. This baking procedure has been found to make quartz-fiber filters more fragile, however.

3.3 Sample Chain-of-Custody and Filter Processing

After the acceptance testing, Teflon-membrane filters are labeled, then weighed in a temperature and relative humidity controlled environment. Pre-fired quartz-fiber filters are stored under low temperature before shipment to the field. The pre-weighed Teflon and pre-fired quartz filters are stored in separate Petri-slides prior to sample shipment.

After field sampling, labeled filters in Petri-slide trays are shipped to the laboratory by the sampling technicians with the source sampling data sheets. Upon receipt from the field, the laboratory technician logs in the samples prior to chemical analyses. A log book is maintained in which the condition of each sample is recorded along with the following information: 1) date and time of sample arrival; 2) site location, sample-type, and corresponding sample number; and 3) physical appearance of the filter samples.

Teflon filters are equilibrated in a temperature and relative humidity controlled environment for 24 hours. They are then weighed and stored under low temperature. Quartz filters are stored at low temperature.

4.0 LABORATORY PROCEDURES

Laboratory procedures associated with this project are: 1) bulk sample resuspension; 2) gravimetric analysis; 3) x-ray fluorescence; 4) filter extraction; 5) anion chromatography; 6) atomic absorption spectrophotometry; and 7) thermal/optical carbon analysis. The procedures followed to accomplish these operations are described below.

4.1 Bulk Sample Resuspension Procedures

The experimental procedures for resuspension sampling are (Chow, 1985):

- Transfer the grab samples from the sampling bag to a glass beaker, cover with a paper towel and label with the sample identification.
- Place the beakers in an oven with the temperature set at less than 40 °F (to eliminate potential volatilization of the sampled material) until the sample is completely dried.
- Perform size segregation by sieving the sample under the laboratory hood. Sieve each sample through a series of 30, 50, 100, 200 and 400 Tyler mesh screens (Fisher Scientific Products) which correspond to particle diameters of 600, 300, 150, 75 and 38 μm, respectively.
- Transfer the smallest particles to a labelled glass storage jar.
- Discard the coarse particles and clean the sieves by blowing with compressed air.
- Repeat the sieving procedure for each source sample.
- Transfer about 5 mg of each sieved sample to a glass container.
- Cover the impactor sampling system being used to collect the resuspended material with the resuspension chamber.
- Resuspend particles from the glass container with air puffs generated by a diaphragm pump. Sampling is usually conducted for a period of 15 to 20 minutes.
- Inspect the areal density on the filters after sampling to ensure that a homogeneous particle distribution is obtained. Proper mass loading (ideally 0.5 to 1.0 mg) can be ensured by a test weighing. Dissasemble the sampling system and and clean with 95 percent alcohol when changing from one type of sample to another in order to eliminate cross-contamination. Similarly, the inside of the

resuspension chamber is wiped out with Kaydry towels and 95 percent alcohol.

4.2 Gravimetric Analysis Procedures for Mass Determination

Unexposed and exposed samples are equilibrated at 20 ± 5 °C temperature and 30 ± 5% relative humidity for 24 hours prior to weighing. Weighing is performed on a Cahn 31 electromicrobalance with ± 0.001 mg sensitivity. Ten percent of all weights are checked by an independent technician and all samples are re-weighed if these check-weights do not agree with the original weights within ± 0.015 mg. All pre- and post-weights, check weights, and re-weights (if applied) are recorded on data sheets for later entry into the data-base management system.

4.3 X-Ray Fluorescence Analysis Procedures for Elements from Aluminum to Lead

X-ray fluorescence analysis is performed on Teflon-membrane filters for Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Br, Rb, Sr, Zr, Cd, Sn, Sb, Ba, Hg and Pb on a Kevex Corporation Model 700/8000 x-ray fluorescence analyzer.

Five separate excitation conditions are used to analyze each filter, as summarized in Table 4-1. A combination of direct-mode and secondary-target excitation methods is used which provides lower detection limits than could be achieved by any single excitation scheme. Low atomic number filters at the exit of the germanium (Ge) and titanium (Ti) secondary targets decrease background and thereby improve the minimum detection limits in key regions of the spectrum.

Interference-free minimum detection limits (MDLs) provided by the system are listed in Table 4-2. These are three sigma (i.e. three times the standard deviation of the blank) interference-free minimum detection limits for a Teflon membrane filter of 1 $\mu g/cm^2$ areal density. This is a very conservative method of calculating detection limits, and the values reported in Table 4-1 are not necessarily directly comparable to MDLs reported by other laboratories without appropriate conversion.

The elemental x-ray peaks reside on a background of scattered radiation. Background removal is accomplished by using the spectrum obtained from analysis of a blank filter as a model background. This model background has the same shape and features of the sample spectra if the deposit mass is small relative to the filter mass, except for the presence of characteristic lines due to particulate matter on the filter (Russ, 1977). The model background is normalized to each sample spectrum to correct for the difference in scatter intensity due to different filter masses. This is done by multiplying the counts

Table 4-1

Excitation Conditions of Kevex/DRI X-Ray Fluorescence Analyzer

Parameter	Condition Number				
	1	2	3	4	5
Tube Voltage, KV	60	30	30	30	8
Tube Current, ma	1.5	3.3	3.3	3.3	1.0
Direct Mode Filter	Mo	Mo	NA	NA	Whatman 41
Thickness	0.2 mm	0.2 mm	NA	NA	3 layers
Secondary Target	NA	NA	Ge	Ti	NA
Filter	NA	NA	Whatman 41	Mylar	NA
Thickness	NA	NA	1 layer	3.8 μm	NA
Analysis Time, sec.	100	400	400	100	100
Energy Range, KeV	0-40	0-20	0-10	0-10	0-10
Elements Measured	Pd, Ag, Cd, In, Sn, Sb, Ba, La	Fe, Co, Ni, Cu, Zn, Ga, As, Se, Br, Rb, Sn, Y, Zr, Hg, Pd	K, Ca, Ti, V, Crg, Mn, Fe, Co, Ni, Cu, Zn	Al, Si, P, S, Cl, K, Ca, Mo	Al, Si, P, S

Table 4-2

X-Ray Fluorescence Air Filter Analysis Interference-Free Minimum Detectable Limits* Using DRI Standard Analysis Protocol

Element	Condition Number	MDL ng/cm^2
Al	5	12
Si	5	6
P	4	18
S	5	3
Cl	4	9
K	4	5
Ca	4	5
Ti	3	2
V	3	2
Cr	3	2
Mn	3	2
Fe	3	1
Co	3	1
Ni	3	1
Cu	3	1
Zn	3	2
Ga	2	3
As	2	3
Se	2	2
Br	2	2
Rb	2	3
Sr	2	4
Y	2	8
Zr	2	14
Mo	4	100
Pd	1	20
Ag	1	20
Cd	1	25
In	1	25
Sn	1	30
Sb	1	30
Ba	1	90
La	1	100
Hg	2	9
Pb	2	9

* MDL defined as 3 times the standard deviation of the blank for a filter of 1 mg/cm^2 areal density and analysis times of 100 sec. for Conditions 1, 4 and 5, and 400 sec. for Conditions 2 and 3.

in each channel of the model background by the ratio of the sample-to-background scatter peak counts.

Experience has shown that use of a scatter peak for blank normalization from too low an energy region, e.g. the Ti secondary target scatter peak from Condition 2, can lead to unreliable background estimation in some circumstances. Therefore, the Mo scatter peak is used for Conditions 1 and 2, and the Ge scatter peak is used for Conditions 3, 4 and 5.

Each excited element generates a set of x-ray lines that are characteristic with respect to energy. Each element from atomic number 13 (aluminum) to 57 (lanthanum) produces a pair of lines, termed K-alpha and K-beta, that are of interest in aerosol filter analysis. The K lines generally increase in energy with atomic number and range in energy from 1.4 to 33 KeV. Elements from atomic number 35 (bromine) to 57 (lanthanum) yield a series of L lines in addition to their K lines. These L series lines range in energy from 1.4 to 4.6 KeV. The elements mercury (Hg) and lead (Pb) produce a series of M lines in addition to their L lines.

Because of the number and spacing of the characteristic x-ray lines relative to detector resolution, peak overlaps must be considered (Dzubay, 1986). A variety of methods has been used to resolve the peak overlaps (Arinc et al., 1977; Parkes et al., 1979; Drane et al., 1983), including least squares fitting to library spectra, fitting to Gaussian and other mathematical functions, and the use of peak overlap coefficients.

Using peak overlap coefficients to successively subtract interferences from peak integrals is a reliable method of determining net peak areas, particularly when concentrations approach the detection limit, as is often the case with air particulate samples. The most important of these overlaps are the K-beta to K-alpha overlaps of elements from potassium (K) to zirconium (Zr), the lead (Pb) L-alpha to arsenic (As) K-alpha interference, and the lead (Pb) M line to sulfur (S) K line interference.

The ability of an x-ray to penetrate matter depends on the energy of the x-ray and the composition and thickness of material. In general, lower energy x-rays, characteristic of light elements, are absorbed in matter to a much greater degree than higher energy x-rays. Larger particles collected during aerosol sampling have sufficient size to cause absorption of x-rays within the particles. Attenuation factors for fine particles (particles with aerodynamic diameter less than 2.5 microns) are generally negligible (Criss, 1976), even for the lightest elements, but these attenuations can be significant for coarse fraction particles (particles with aerodynamic diameter from 2.5 to 10 microns or 2.5 to 15 microns). A variety of approaches has been taken to this problem, including calibration for light elements using particle standards of known particle size (Rhodes, 1975), calculation of attenuation factors based on assumed particle size distributions and compositions (Dzubay and Nelson, 1975), and reporting results as thin film equivalent concentrations (Harrison and Eldred, 1973). The methods that correct the data are based on assumptions of particle size distribution and composition of the sample. Variation in these factors normally encountered in ambient and source aerosol samples can yield significantly different attenuation corrections. For this reason, concentrations should be calculated without correction to ensure that they can be traced unambiguously to NBS standards. A range of correction factors which is typical of the chemical composition and particles sizes of the samples being analyzed should then be examined. Corrections calculated by Dzubay (1983) for PM_{10} size distributions and typical mineral compositions are used in this study.

The x-ray fluorescence (XRF) spectrometer is calibrated using thin film standards containing known quantities of one or two elements. In this approach, a sensitivity factor for element i is obtained from the level relationship: $I = S_i m_i$ (Giauque et al., 1977), where I is x-ray intensity in counts/sec, S_i is the sensitivity in counts/sec per $\mu g/cm^2$, and m_i is the mass of element i in the standard in $\mu g/cm^2$. The S_i are used to convert net peak intensities obtained from spectral analysis of a sample to concentrations.

Three types of XRF standards are available: 1) vacuum-deposited thin film elements and compounds (Micromatter), 2) polymer films (Dzubay et al., 1981), and 3) NBS thin-glass films. Before any standards are used for calibration, their concentrations are verified as described below and corrections to thin film equivalent concentrations are made if necessary.

Validation of concentrations is accomplished by determining the ratio of instrument response (counts/sec) to concentration ($\mu g/cm^2$), and plotting those ratios as a function of atomic number. Since response of the instrument varies smoothly as a function of x-ray energy, the plot should yield a smooth curve. Thin film Micromatter standards are analyzed for a variety of elements and are fit to this smooth curve. Most of the standards obtained fall within ± 10% of the smooth curve, but occasionally a standard will yield results significantly in error. These standards with values that do not match the curve are either replaced or their concentrations are adjusted.

A quality control standard is analyzed routinely with each run of samples as a check of instrument calibration and performance. This is a specially fabricated multi-element thin film standard containing appropriate concentrations of key elements. An NBS standard reference material thin-glass film standard is analyzed with every set of 15 filters, providing assurance of accurate calibration. The XRF analyzer is stable over many months.

4.4 Filter Extraction Procedures

The Teflon membrane filter is cut in half after x-ray fluorescence analysis for ion chromatographic analysis and atomic absorption spectrophotometric analyses. These sections are extracted in 20 ml of distilled deionized water (DDW) for both anion chromatographic analysis of nitrate and sulfate and atomic absorption spectrophotometric analysis of soluble sodium and potassium.

After filter sectioning, the remaining filter is placed back to the original Petri-slide and archived. The sections taken from the filter are placed in a clean, labeled extraction vial with sterile forceps. This procedure takes place in filtered air with gloved hands to prevent contamination. The filter sections are curled rather than folded in the extraction vial so that the entire deposit is exposed to the DDW solvent. Twenty milliliters (ml) of the DDW are added to the vial which is covered with a polyethylene cap. Each extraction tray with 32 extraction vials is placed in an ultrasonic bath for 30 minutes of extraction. The bath water is continually replaced to prevent temperature increases from the dissipation of ultrasonic energy in the water. After extraction, these solutions are stored under refrigeration prior to analysis.

4.5 Ion Chromatographic Analysis Procedures for Nitrate and Sulfate

Ion chromatography (IC) is a liquid chromatographic technique using an ion exchange mechanism and suppressed conductivity detection for he separation and determination of anions and cations. It offers a unique means of quantifying these ions as well as others in a multi-element mode. This method is subject to fewer interferences from substances commonly found in aerosol samples than the other two techniques. This chromatographic approach was developed by Small et al. (1975) and has been applied to aerosol measurements since the mid-1970's (Mulik et al., 1976; 1977; Butler et al., 1978; Mueller et al., 1978; Rich et al., 1978; Small, 1978).

The Dionex 4000i Ion Chromatographic system for the analysis of nitrate and sulfate contains an anion separator column (AS4 column Cat No 38019) with a strong basic anion exchange resin, and an anion micro-membrane suppressor column (250 x 6 mm ID) with a strong acid ion exchange resin. The anion eluent consists of 0.0045 M $NaHCO_3$ and 0.0018 M Na_2CO_3 prepared in distilled-deionized water. This water is verified to have a conductivity of less than 10^{-5} ohm^{-1} cm^{-1} prior to preparation of the eluent. For quantitative determinations, the chromatograph is operated at a flow rate of 1.5 ml/min provided by a 1000 psi pressure. Theoretically, the higher the pressure applied, the more rapid is the separation that can be achieved. Approximately 5 ml of extract are required for each analysis.

Standard solutions of KNO_3 and $(NH_4)_2SO_4$ are prepared with reagent grade salts which are dehydrated in a desiccator several hours prior to weighing. These anhydrous salts are weighed to the nearest 0.010 mg on a regularly-calibrated analytical balance under controlled temperature (~20 °C) and relative humidity (± 30%) conditions. These salts are diluted in precise volumes of distilled deionized water. A standard solution is prepared monthly and stored in a refrigerator. Calibration solutions are prepared daily by diluting the standard solution to concentrations covering the range of concentrations expected in the filter extracts. The calibration concentrations prepared are at 0%, 10%, 50%, 75% and 100% of instrument full-scale setting. These transfer standards are also be traceable to NBS weights and volumes via the mass and volume measurements from which the standardized values were derived.

A portion of each calibration solution is stored under refrigeration for re-verification in external audits and interlaboratory comparisons. Calibration is required before the sample run and performance tests are conducted every 20 samples. The ions are identified by matching the retention times of each peak in the unknown sample with each peak in the chromatogram of the standard.

A distilled deionized water blank and a performance test standard are alternated every tenth specimen as quality control checks on base-line and calibration, respectively. If the values obtained for these standards do not coincide within a pre-specified uncertainty level (typically three standard deviations of the baseline level or ± 5%), all of the samples between that standard and the previous performance test standard are reanalyzed. The Dionex IC system is capable of measuring ions with 5 to 10 ppb detection limits for small sample volumes.

4.6 Atomic Absorption Spectrophotometric Analysis Procedures for Sodium da Potassium

Atomic absorption spectrometry (AAS) is used to measure sodium and potassium. It is based on the principle of absorption of radiation from the source by an atomic species when the energy of source radiation closely coincides with the energy change in the transition of absorbing species. With the application of Beer-Lambert's law, the amount of absorption is a function of the number of atoms which is proportional to analyte concentration.

One of the distinguishing features of AAS is that the analyte must be in the elemental ground state. This is accomplished by aspirating the sample into a chamber where it is "atomized" into small droplets and mixed with the fuel and air. The major components of the AAS system consist of a light source with a hollow cathode tube containing a chemical element which is able to output that element's UV-visible spectrum. The line spectrum passes through the sample chamber (where the sample is heated with a burner) to a monochromator where an appropriate resonate wavelength is focused on a photomultiple tube, the detector. The electronic signals undergo processing and are output through the readout device.

A Perkin-Elmer Model 2380 Double Beam Atomic Absorption Spectrophotometer is used to analyze filter extract samples by comparison with standards of known composition. A standard calibration curve is established for each element before performing sample analysis. The ACS reagent grade sodium and potassium standards are used as stock standard solution. The dilutions of the stock solution to be used as calibration standards (0, 0.5, 1.0, 3.0 and 5.0 μg Na^+/ml; 0, 2, 5, 7.5 and 10.0 μg K^+/ml) are prepared daily prior to sample analysis. Extracts are filtered to eliminate clogging of the nebulizer by suspended solids. An oxidizing flame with air and acetylene is required with a sodium hollow cathode tube to measure the sodium absorption at 589.0 nm and a potassium hollow cathode tube to measure the potassium absorption at 766.5 nm. Ionization interference in sodium and potassium determination is eliminated by the addition of cesium chloride (CsCl) to samples and standard solutions. The chemical interference can be minimized with the addition of lanthanum in the sample extract. Approximately 5 ml of extract are submitted for each analysis.

4.7 Thermal/Optical Carbon Analysis Procedures for Organic, Elemental da Carbonate Carbon

The thermal/optical method developed at the Oregon Graduate Center (OGC) is used for the analysis of carbonaceous aerosol on quartz fiber filters (Johnson, 1981; Johnson et al., 1981; Shah, 1981; Huntzicker et al., 1982; Rau, 1986). This method consists of a thermal system and an optical system. Its fundamental operating principle relies on the fact that organic compounds can be volatilized from the filter in a helium (He) atmosphere while elemental carbon is not oxidized. This principle involves: 1) conversion of organic and elemental carbon to carbon dioxide (CO_2) under different temperature and oxidation environments; 2) reduction of CO_2 to methane (CH_4); and 3) quantification of CH_4 by a flame ionization detector (FID).

The principal function of the laser reflectance system is to continuously monitor the filter reflectance throughout an analysis cycle. This reflectance is proportional to the degree of pyrolytic conversion from organic to elemental carbon which takes place during organic carbon analysis. The correction for pyrolytic conversion of organic to elemental carbon is essential for an unbiased measurement of both carbon fractions, as discussed above.

For organic and elemental carbon analysis, a 0.5 cm^2 circular punch is removed from a quartz-fiber filter. Procedures for the determination of organic carbon are:

1. Place the filter disks into the quartz boat.
2. Purge the oven with a helium (He) atmosphere for 2 to 3 minutes.
3. Insert the quartz boat into the combustion zone where oxidation and volatilization of organic carbon into the flowing helium (He) stream

occurs. The temperature programming ramps from 250 oC, to 450 °C, to 650 °C, to 750 °C.

4. The flow moves through the oxidation zone (MnO_2 at 950 °C) where all volatilized carbon is converted to CO_2.

5. The CO_2 is reduced to CH_4 in the methanator and detected by the FID.
For the determination of elemental carbon:

6. Introduce a 2% oxygen (O_2) in helium (He) atmosphere.

7. Increase the oven temperature in steps to 700 °C and to 800 °C. This slow oxidation of the elemental carbon is applied in order to facilitate the correction for pyrolytically generated elemental carbon.

The lower quantifiable limits (LQLs) of carbon combustion methods depend upon the variable carbon content of the blank quartz filters rather than the analysis method. As noted in Section 3, the unexposed filter is pre-fired in an oven at 700 to 800 °C for several hours to remove any residual carbon contamination (Fung, 1986; Huntzicker, 1986; Rau, 1986). For well-cleaned quartz filters, the standard deviation of the blanks for both organic and elemental carbon is on the order of 0.2 to 0.3 $\mu g/cm^2$ (Fung, 1986).

Calibration is carried out by analyzing known amounts of methane, carbon dioxide, and potassium hydrogen phthalate (KHP). The FID response is ratioed to a reference level of methane injected at the end of each sample analysis. Performance tests of instrument calibration are conducted at the beginning and end of daily operation, as well as at the end of each sample run. All intervening samples are reanalyzed if calibration changes of more than ± 5% are found. Known amounts of American Chemical Society (ACS) certified reagent grade crystal sucrose and potassium hydrogen phthalate (KHP, 99.95 to 100.05%) are combusted as a verification of the organic carbon fractions. A total of 15 different standards are used for each calibration. Widely accepted primary standards for elemental and/or organic carbon are still lacking.

Carbonate analysis involves the acidification of a filter segment with 20 ul of 1% phosphoric acid at 50 °C which results in the conversion of carbonate to gaseous CO_2, followed by methanation and FID detection of methane, as discussed in the previous section. The carbonate analysis system is an integral part of the DRI/OGC thermal/optical carbon analyzer. The instrument is calibrated with separate standards of soluble potassium carbonate and the analyses are carried out as described above as a separate step. The carbonate-free sample is then submitted to the normal thermal/optical carbon analysis cycle.

4.8 Automated Colorimetric Analysis

The Technicon (Tarrytown, NY) TRAACS 800 Automated Colorimetric System is used to measure ammonium concentrations by the indophenol method. Ammonium in the extract is reacted with alkaline, phenol, and sodium hypochlorite to produce indophenol, a blue dye. The reaction is catalyzed by the addition of sodium nitroprusside. The absorbance of the solution is measured at 630 nm.

Approximately two milliliters of extract are placed in an autosampler which is controlled by a computer. Seven standard concentrations (0.05, 0.1, 0.3, 0.5, 1.0, 2.0, and 3.0 μg/ml) are prepared from ACS reagent-grade $(NH_4)_2SO_4$ following the same procedure as that for IC standards. Each set of samples consists of two distilled water blanks to establish a baseline, seven calibration standards and a blank, then sets of ten samples followed by analysis of one of the standards and a replicate from a previous batch. The computer control allows additional analysis of any filter extract to be repeated without the necessity of loading the extract into more than one vial. Analyzer performance is checked using 2 dilutions of NBS traceable ion chromatography ammonium standard solution (Fisher #SC349-100, 1000 μg/ml ammonium) and NBS rainwater standard (#2694-II, 1.0 μg/ml).

The system determines carry-over by analysis of a high standard followed by 2 blanks. The percent carry-over is then automatically calculated and can be applied to the samples analyzed during the run. Technicon software operating on an IBM/XT microcomputer controls the sample throughput, calculates concentrations, and records data.

Formaldehyde has been found to interfere when present in an amount which exceeds 20% of the ammonium content and hydrogen sulfide interferes in concentrations which exceed 1 mg/ml. Nitrate and sulfate are also potential interferents when present at levels which exceed 100 times the ammonium concentration. These levels are rarely exceeded in ambient samples. The precipitation of hydroxides of heavy metals such as calcium and magnesium is prevented by the addition of disodium ethylenediamine-tetracetate (EDTA) to the sample stream (Chow, 1981).

5.0 DATA REDUCTION, VALIDATION, AND REPORTING

5.1 Source Sampling Data Processing

The contents of source sampling data sheets are entered into computerized data forms. These forms have limits which do not allow entry of values which lie outside of a certain range. Every data item which is entered is verified by the data processing supervisor against the original data sheet.

A dummy data file which contains fields for sampling site, date, and all chemical species is formed by a dBaseIII+ program from these field data records. The IDs, flow rates, sampling sites, dates, and elapsed times have values in their appropriate fields. All other fields contain the missing data default value. These defaults are replaced by laboratory analysis data as they become available. In this way, it is always possible to determine which analyses have been completed and which have not.

The laboratory chain-of-custody data base records the disposition of each sample, and this data base can be consulted to determine the fate of missing values in the master data base. This independent tracking is needed to prevent sample IDs from being mixed up.

Each DRI laboratory analysis instrument is being linked to IBM-PC compatible computers and data are recorded in ASCII text files. All data are keyed to the filter IDs, and dBaseIII+ programs have been written to replace the defaults in the master data file with these values. Separate flags are entered at the time of analysis to indicate that a sample is an ambient sample, a source sample, a field blank, a laboratory blank, a replicate, a re-run, a performance test standard, or an audit standard. These flags are used to separate these quality control values from the individual data bases to generate quality control charts and precision estimates.

5.2 Data Base Management

Data from these various chemical analyses must be unified as they become available. The data base management system is needed to fulfill the following requirements:

- Quantitative and descriptive information must be accommodated.
- Data from a number of sources must be merged in an efficient and cost-effective manner.
- Input data required by models should be easily accessible directly from the data base.

The dBaseIII+ (Ashton-Tate, 1985) relational data base has been selected for this data base management task because:

- It is commonly available, reasonably priced, has good third party documentation and support, and provides interfaces to many other software packages.
- Non-proprietary software is available to access dBaseIII+ files directly without the need for the dBaseIII+ software itself. This software will be incorporated into Version 7 of the EPA/DRI Chemical Mass Balance (CMB) software.
- The EPA source composition library, the state of Nevada's source composition library and ambient data, and California's source composition library will be compiled in dBaseIII+ files.
- dBaseIII+ can handle 128 fields of 4000 characters per record and up to one billion records per file. A memo field is available which can be used to record data validation and ambient or source description data for each set of test results.
- Data entry, data validation, reformatting, and data reporting programs can be written.

5.3 Sample Validation

Sample validation consists of procedures which identify deviations from measurement assumptions and procedures. Three levels of validation will be applied which will result in the assignment of a: 1) valid, 2) valid but suspect, or 3) invalid rating to each measurement.

Level I sample validation takes place in the field or in the laboratory and consists of: 1) review of instrument outputs and data sheets and flagging of samples when significant deviations from measurement assumptions have occurred, 2) verifying computer file entries against data sheets, 3) eliminating values for measurements which are known to be invalid because of instrument malfunctions, 4) replacement of data from a backup data acquisition system in the event of failure of the primary system, and 5) adjustment of measurement values of quantifiable calibration or interference biases.

Level II sample validation takes place after data from various measurement methods have been assembled in the master data base. Level II applies consistency tests based on known physical relationships between variables to the assembled data. Level II Validation tests are: 1) the sum of all chemical species in a particulate matter sample is compared to the gravimetric mass of that sample; 2) mass and chemical concentrations in smaller size fractions are compared to corresponding values in larger size fractions; and 3) analyses of the same species by different methods (e.g. K by XRF and AA, S by IC and XRF). Samples are designated for re-analysis if they do not meet these criteria.

Data adjustments for quantifiable biases (e.g., large particle absorption corrections for aluminum) can be made in Level II validation if they are discovered after assembly of the master data base.

Level III sample validation is really a part of the data interpretation process. The first assumption upon finding a measurement which is inconsistent with physical expectations is that the unusual value is due to a measurement error. If, upon tracing the path of the measurement, nothing unusual is found, the value can be assumed to be a valid result of an environmental cause. Unusual values are identified during the data interpretation process as: 1) extreme values, 2) values which would otherwise normally track the values of other variables in a time series, and 3) values for observables which would normally follow a qualitatively predictable spatial or temporal pattern. No Level III sample validation will be applied within the scope of this project.

All data validation actions at each level are recorded in a data validation summary which accompanies the data base. Data base records contain flags to identify the level of validation which they have received at any point in their existence.

6.0 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

Approximately 10% of all samples and all dynamic blank filters are submitted for replicate analysis. These replicate and blank performance tests serve two purposes: 1) to identify possible problems with the measurement process; and 2) to calculate the precision of each measurement.

To serve the first purpose, when replicates exceed initial analysis results by more than pre-set tolerances (typically ± 10%), all analysis ceases until the reason for the discrepancy is discovered. All samples analyzed since the previous performance test are re-analyzed.

To serve the second purpose, the precision for samples analyzed over selected time periods is calculated by the methods described in Watson et al., (1983).

For replicate analyses, the precision of each chemical species, σ_C, on individual filters is:

$$\sigma_C = \left[\frac{1}{2} \sum_{i=1}^{N} (C_i - C_r)^2 \right]^{1/2}$$

Where N is the number of replicates, C_i is the chemical species content derived from the routine measurement, and C_r is the chemical species derived from the replicate measurement. When different sections of the same sample are extracted and analyzed, this precision contains the uncertainty due to the inhomogeneity of the deposit across the filter surface as well as the uncertainty of each chemical analysis.

For the dynamic blanks, the uncertainty of blank subtraction, σ_B, is:

$$\sigma_B = \left[\frac{1}{M-1} \sum_{j=1}^{M} (B_j - \bar{B})^2 \right]^{1/2}$$

Where M is the number of blanks, B_j is the value of the j^{th} blank, and $\bar{B}$, is the average blank value:

$$\bar{B} = \frac{1}{M} \sum_{j=1}^{M} B_j$$

The average blank value $\bar{B}$, is normally subtracted from each measurement if it exceeds σ_B. Both σ_C and σ_B are combined with the precision of the sample volume measurement to determine the precisions of the sample concentrations.

Accuracy is expressed as the distribution of percent deviations from the values of the reference materials submitted to routine analytical procedures. If the average percent deviation does not exceed twice the precision of a measurement, then the values will be considered accurate.

7.0 PERFORMANCE AND SYSTEM AUDITS

It is expected that ARB will retain an independent auditor for all measurements. This auditor will examine the standard operating procedures, will observe routine operations to assure compliance with those procedures, and will submit standard reference materials to the analysis process over the course of this project.

The DRI project co-principal investigator will cooperate with the external auditors and will supply all access and information necessary to complete the audit tasks. Both the DRI quality assurance officer and the DRI project co-principal investigator will examine auditor recommendations and will implement those which will enhance the measurement process. The laboratory manager will analyze performance audit discrepancies and implement procedural and/or traceability changes to correct them.

Table 7-1 summarizes the laboratory calibration, performance test, and audit observables, standards, and frequencies.

Table 7-1

Laboratory Performance Audit Observables

Measurement	Measurement Device	Performance Test Frequency	Performance Standard	Calibration Frequency	Calibration Standard	Audit Frequency	Audit Standard	Primary Standard
Fine Particle Mass	Cahn 31 Electro-microbalance	At beginning of each weighing session	NBS Calss M Standard Weights	Daily	NBS Calss M Standard Weights	Once/Study Period	NBS-Traceable Weights	NBS-Traceable Weights
Fine Particle Elements	Kevex 700/8000 XRF Analyzer	1/15 Samples	NBS Thin Film Standards	Quarterly	Micromatter Thin Film Standards	Once/Study Period	Independent NBS Thin Film Standard	NBS-Standard Filter
Fine Particle Ions ($SO_4^=$ NO_3^-	Dionex 4000i Ion Chromatographic Analyzer	At beginning of each measurement session	NBS Certified Simulated Standard Material	Daily	ACS Certified Standard Solution	Once/Study Period	Spiked Filters Standard Solutions	ACS Certified Chemicals
Soluble Fine Particle Sodium and Potassium	Perkin Elmer Model 2380 Atomic Absorption Spectrophotometry	At beginning of each measurement session	NBS Certified Standard Material	Daily	ACS Certified Standard Solution	Once/Study Period	Spiked Filters Standard Solutions	ACS Certified Chemicals
Fine Particle Organic, Elemental, and Carbonate Carbon	DRI/OGC Thermal/-Optical Carbon Analyzer	Each set of Analyses	Methane Gas	Daily	Methane, CO_2 Gas and ACS Certified KHP	Once/Study Period	Standard KHP Solutions	ACS Certified Chemicals

8.0 STANDARD OPERATING PROCEDURES

Standard Operating Procedures (SOPs) codify the actions which are taken to implement a measurement process over a specified time period. State-of-the-art scientific information is incorporated into the SOP with each revision. The SOPs followed in this ARB study include the following elements:

- A brief summary of the measurement method, its principles of operation, its expected accuracy and precision, and the assumptions which must be met for it to be valid.
- A list of materials, equipment, reagents, and suppliers. Specifications are given for each expendable item and its storage location is listed.
- Designation of the individual responsible for each part of the procedure.
- A general traceability path, the designation of primary standards or reference materials, tolerances for transfer standards, and a schedule for transfer standard verification.
- Start-up, routine, and shutdown operating procedures and an abbreviated checklist.
- Copies of all data forms with examples of filled out forms.
- Routine maintenance schedules, maintenance procedures, and troubleshooting tips.
- Internal calibration and performance testing procedures and schedules.
- External performance auditing schedules.
- References to relevant literature and related standard operating procedures.

The standard operating procedures required for the laboratory operation of this project are the following:

- Sample Shipping and Chain-of-Custody Procedures
- Filter Acceptance Testing and Processing
- Quartz Filter Pre-firing Procedures

- Gravimetric Analysis Procedures
- Filter Sectioning Procedures
- Filter Extraction Procedures
- Ion Chromatographic Analysis Procedures of Nitrate and Sulfate
- Atomic Absorption Spectrophotometric Analysis Procedures of Soluble Sodium and Potassium
- X-ray Fluorescence Analysis Procedures of Teflon Membrane Aerosol Deposits
- Thermal/Optical Carbon and Carbonate Analysis Procedures

Standard operating procedures specific to this project will be adapted from existing standard operating procedures currently in use at DRI.

9.0 QUALITY ASSURANCE REPORT TO MANAGEMENT

All procedures, precisions, accuracy estimates derived from independent audits, and sample validation results will be presented in the Quality Assurance Report. Portions of this report will be suitable for direct use in ARB measurements reports.

This QA report combines data and data interpretation specific to ARB studies with more generic information which is continuously being systematized as part of DRI's on-going air quality studies. In this way, ARB's final measurement reports will present the most accurate and comprehensive information on ARB and other measurements which are available at the time.

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