

National Human Exposure Assessment Survey (NHEXAS)

Region 5 Study

Quality Systems and Implementation Plan for Human Exposure Assessment

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Title: NHEXAS Filter Handling, Weighing and Archiving Procedures for
Aerosol Samples

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TITLE: NHexas FILTER HANDLING, WEIGHING AND ARCHIVING
PROCEDURES FOR AEROSOL SAMPLES

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NHEXAS Filter Handling, Weighing and Archiving Procedures for Aerosol

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PREFACE

The weighing systems described in this protocol are still under development as these procedures are being prepared (June, 1994). Version series 1.X contains background material on measurements and control levels not found in the more specific operational Version 2.X series. The procedures described in the present version should be considered preliminary, but representative of the information to be contained in the final version.

1. Scope and Application

1.1 Study Description: The primary goal of NHEXAS is to reduce uncertainties in exposure and risk assessments, by (a) producing reliable estimates of the status and trends in total human exposures (via inhalation, ingestion and transdermal routes) to potentially harmful environmental agents, (b) determining the incidence and causes of high exposures, especially for biologically susceptible persons, and (c) establishing relationships between environmental concentrations, exposure, dose and health response. A specific objective of NHEXAS is to estimate the distribution of exposures of selected contaminants, especially the mean, median, and 90th percentile for the general population and subsets of special interest.

1.2 Data Use: The data collected under this protocol for the NHEXAS study will be used to develop concentration distributions for the affected populations. These distributions will be combined with personal activity information to estimate the study population exposure distributions. The precision and accuracy requirements for measurements in the NHEXAS data base are derived primarily from the study goals to define the mean, median and 90th percentile of these exposure distributions. The distributions will then be used to conduct hypothesis testing proposed as the bases for the program.

1.3 Target Analytes

The NHEXAS contaminants of interest, specifically relevant to this protocol, are size-specific aerosol mass concentrations from personal, indoor and outdoor sampling, with subsequent analyses to be conducted on selected samples for trace metals, including lead, cadmium, chromium and arsenic. The NHEXAS goal to estimate annual average exposures

using sample concentrations integrated over 6 to 7 day periods, places constraints on the aerosol sampling process that may affect the precision and accuracy for certain trace metals.

1.4 Limits of Detection

The mass concentration Quantitation Limits (QL) for the Total Inspirable and PM10 samplers are a function of a) the total integration interval, b) the sampling flowrate, c) the repeatability (standard deviation) of the weighing process used to perform mass measurements, and d) the allowable coefficient of variation. Using a) a total sampling window integration interval of six 24-hour days with an ON/OFF cycle ratio of 1/3 over the integration window to give a total sampling time of 2880 minutes, b) a sampling flowrates of 2.0 lpm, c) a "best-case" repeatability for weighing 25 mm Teflo[®] filter of 6.7 ug, and d) an allowable accuracy (coefficient of variation) of $\pm 10\%$, a LOD of 20 $\mu\text{g}/\text{m}^3$ is expected (see reference 2.2.5).

1.5 Responsibilities

1.5.1 Co-Principal Investigator (CPI) will be responsible for the final review and approval of this procedure. The Co-Principal Investigator (CPI) is responsible for a) providing equipment and supplies to the Field Coordinator (FC) to utilize this Standard Operating Protocol (SOP), and b) monitoring the field operating information relative to the quality assurance measures specified for this SOP and relaying corrective instructions to the FC as necessary.

1.5.2 Laboratory Coordinator (LC) will be responsible for a) training the Laboratory Technicians (LT's) in the application of this SOP and utilization of the described equipment and supplies, b) training the LT's in the proper recording of laboratory data, c) training the LT's in the quality assurance measures needed to support this SOP, d) corresponding with the CPI on equipment and supply needs to support continued application of this SOP, e) corresponding with the CPI on any problems encountered with this SOP.

1.5.3 Laboratory Technician (LT) will be responsible for a) understanding the following the procedures described in this SOP, b) understanding and following the quality assurance measures supporting this SOP, c) insuring that the laboratory and QC data are properly recorded, d) following the procedures for sample custody, handling and shipment of filters, as described in the most current version of "NHEXAS Filter Logging, Storage and Shipment Procedures for Aerosol Samples", and e) notifying the LC on any problems encountered with this SOP.

2. Summary of Procedures

2.1 Overview

The purpose of this NHEXAS Standard Operating Protocol is to describe the procedures for weighing, handling and archiving aerosol filters and management of the associated analytical and quality assurance data. The basic flow of samples and data is shown in Figure 1. Most of the operations described will take place at the RTI/RTP aerosol laboratory, with only the automated field sampling data transfer occurring at NHEXAS field sampling locations. Analytical and quality assurance information are entered, processed and stored using separate software packages, written specifically for each location. The specific operations covered in this protocol include: (a) filter quality examination, (b) filter and blank pre-weighing, (c) filter

loading into either IOM or PM10 aerosol sampling inlets, (d) shipment of unexposed samples to the field sampling locations, (e) field sample data collection and entry, (f) shipment of exposed samples and sampling data to the RTI/RTP aerosol laboratory, aerosol inlet unloading, (g) filter and blank post-weighing, (h) sample validation using collection quality assurance data, (i) computation of mass concentrations, (j) temporary archival of samples for subsequent analyses, and (k) shipment of samples to RTI/ACS laboratories for analysis. Separate protocols describe (a) the field sample storage and shipment procedures, (b) the field sample collection processes for the determination of mass concentration, and (c) substrate analyses, other than mass concentration.

2.2 Method References

2.2.1 British Health and Safety Executive, "General Methods for the Gravimetric Determination of Respirable and Total Inhalable Dust", Method MDHS 14, Occupational Medicine and Hygiene Laboratory, London NW2 6LN, October, 1989.

2.2.2 Gelman Sciences, Inc., "Laboratory Filtration - The Gelman Sciences Filter Book", PN 32378, Ann Arbor, MI, 1994.

2.2.3 Pellizzari, E., "Particle Team Exposure Assessment Methodology (PTEAM) Pilot Study, Volume II: Protocols for Environmental Sampling and Analysis, Addendum to the Workplan", report for EPA Contract 68-02-4544, Work Assignment 67, ARB Agreement No. A833-060, Research Triangle Institute, Research Triangle Park, NC 27709, February, 1992.

2.2.4 Rodes, C. E., "NHEXAS Filter Weighing Issues", internal NHEXAS project report, Research Triangle Institute, Center for Aerosol Technology, Research Triangle Park, NC 27709, November, 1993.

2.2.5 Rodes, C. E. and R.J. Newsome, "Moisture Uptake of Selected Sampling Substrates Proposed for the NHEXAS Program", internal NHEXAS project report, Research Triangle Institute, Center for Aerosol Technology, Research Triangle Park, NC 27709, February, 1994.

2.2.6 Rodes, C. E., Newsome, R.J. and P.K. Lawless, "Personal, Indoor and Outdoor Air Sampling Procedures for Total Inspirable and PM10 Aerosols", NHEXAS Protocol RTI/ACS-AP-209-010, Research Triangle Institute, Research Triangle Park, NC 27709, April, 1994.

2.2.7 U. S. Environmental Protection Agency, "Determination of Respirable Particulate Matter in Indoor Air Using Size Specific Impaction", Method IP-10, in **Compendium of Methods for the Determination of Pollutants in Indoor Air**, EPA publication EPA-600/S4-90/010, Research Triangle Park, NC 27711, 1990.

2.3 Definitions

EPA - Environmental Protection Agency

NHEXAS - National Human Exposure Assessment Study

RTI - Research Triangle Institute (Research Triangle Park [RTP], NC)

EOHSI - Environmental and Occupational Health Sciences Institute (Rutgers, NJ)

SOP - Standard Operating Protocol

PM10 - particulate matter meeting the Environmental Protection Agency definition for particles with an aerodynamic diameter less than or equal to 10 micrometers

Inspirable Particulate Matter - particulate matter meeting the criteria of Reference 2.2.8 for particles inspired through the oral and nasal entry planes

IOM - Institute for Occupational Medicine (Edinburgh, Scotland)

SKC - Commercial manufacturer (Eighty Four, PA) of a conductive plastic version of the IOM sampler to collect the inspirable particulate matter fraction

lpm - liters per minute

STP - standard temperature and pressure (25 °C and 760 mm Hg)

MSP - Commercial manufacturer (Minneapolis, MN) of the personal PM10 aerosol inlets proposed to be used in this program

LOD - Limit of Detection

ASSL - Aerosol Sampling Support Laboratory

CPI - Co-Principle Investigator

LC - Laboratory Coordinator

LT - Laboratory Technician

Participant - the primary respondent at each residence who carries the personal monitoring system

ID#- Identification Number

3. Filter Inspection/Aerosol Inlet Loading

Filter inspection and aerosol inlet loading activities will occur under controlled conditions at the RTI/RTP aerosol laboratory.

3.1 Filter Inspection

The expected tare weight for the 25 mm Teflo® filters is approximately 0.040 g (40 mg), which should be easily accommodated by analytical balances with either a 10 µg or a 1.0 µg resolution. The tare weight of a 37 mm Teflo® filter is approximately 0.110 g. Filter tare weight deviations greater than ± 10 mg (0.010 g) should be considered as questionable, and the filters rejected for entry into the data system. Stability tests of the 25 mm Teflo® filter tare weight over the range of typical relative humidities (20 to 80%) has shown less than a 10 µg change. Repeated weighings of a Teflo® filter with a Mettler AT261 Analytical balance with a 10 µg resolution, supported on a marble weighing table, showed a reproducibility of 6.7 µg.

Of concern for tare weight stability of the Teflo® filter are losses of shards of the PTFE and polymethylpentene materials left partially attached to the filter during manufacture. Brief visual examination under a lighted, 5 power magnifying lens (VWR 36934-005, or equiv.) must

be conducted on each filter prior to tare weighing. This examination must identify and remove the larger loosely held fragments that may become dislodged during subsequent handling, or, if the problem can't be corrected, reject the filter as unusable. This examination must include a "candling" step where the filter is held in front of a high intensity light (VWR 36519-149, or equiv.) and examined for pin holes or tears caused by the manufacturing process or rough handling. Suspect filters should be rejected. Filters that were noticeably deformed by the heat sealing process, to include more than 2.0 mm of out-of-roundness and annulus rings bowed (will not lie flat) by more than 5.0 mm are also to be rejected.

3.2 Aerosol Inlet Loading

Pre-weighing procedures are different for each aerosol inlet type and are described in section 4.3.

3.2.1 IOM Total Inspirable

The SKC, Inc. IOM aerosol inlet is shown in Figure 2. A Millipore 25 mm Dacron back-up support screen is used beneath the pre-weighed Gelman Teflo[®] filter. The filter and support screen are placed into the two-piece cassette prior to loading the assembly into the inlet. The screw pressure of the inlet cap forces the cassette to seal against the rubber O-ring located between the cassette and base. The degree of screw pressure is determined by a special torque screwdriver that attaches to the cap with a special adapter as shown in Figure 3. A pre-set torque value [*to be determined in June, 1994*] assures a proper seal.

The primary filter substrate for this aerosol inlet will be Gelman 25 mm diameter, 3.0 μ m Teflo[®] filters. This filter consists of a 3.0 μ m porosity PTFE Teflon layer stretched across a 25 mm diameter, polymethylpentene annulus ring and heat sealed. To add rigidity to the Teflon layer during sampling and minimize pattern blinding caused by the filter holder support grid, a 25 mm Dacron backup supporting screen is added under the Teflo[®] filter. The exposed area for aerosol collection is approximately 21 mm in diameter. Aerosol samples (Total Inspirable and PM10) will be collected at 2.0 lpm on filter substrates over a predetermined integration interval.

After the pre-weighed filter has been installed into the inlet, the leak-tightness of the inlet and the integrity of the filter must be tested by measuring the pressure drop across the inlet at 2.0 lpm. Every loaded aerosol inlet sent to the field locations will be tested. Within a pre-determined limit, the pressure drop for a clean filter should be repeatable. Units not meeting this pressure drop indicate that either an internal leak is present in the inlet, or the filter has a mechanical flaw.

The testing is accomplished using the apparatus shown in Figure 4, where a flow-controlled personal sampling pump with a digital flowrate display (MSA ELF pump, or equivalent) provides the suction, a water manometer measures the pressure drop and an automatic bubble flowmeter determines if the correct flowrate of 2.0 lpm is maintained. This apparatus uses a quick-connection on the outlet of the aerosol inlet to provide a leak-tight seal. The procedure consists of 1) connecting the inlet, 2) turning the pump ON to an adjusted flow of 2.0 lpm, 3) allowing the flow to stabilize for 20 seconds, 4) reading the bubble flowmeter to determine if the flow is 2.0 ± 0.1 lpm (1.9 to 2.1 lpm), and 5) reading the water manometer pressure drop to determine if the pressure drop is 1.3 ± 0.2 inches of water (1.1 to 1.5 inches of

H₂O). If the flowrate is outside the acceptance limit, readjust the pump and repeat the test procedure. If the flowrate is acceptable, but the pressure drop is outside the acceptance limit, the inlet should be disassembled to determine if a error was made in the assembly process. If no error is detected, the inlet assembly procedure should be repeated, and the pressure drop test repeated. If the pressure drop is still outside the acceptance limits, the inlet should be disassembled and the filter discarded and replaced with a new pre-weighed filter.

Exposure situations involving aerosols that tend to clog ("blind") the Teflon substrate, may require the use of fiber matrix filters that have a substantially greater aerosol capacity. As a point of reference, the 25 mm Teflo® filters will begin to blind (demonstrate a rapid increase in pressure drop with loading) after the collection of approximately 2.0 to 2.5 mg of tobacco smoke. Glass fiber filters of the same diameter will collect at least 4 times as much tobacco smoke without blinding. The Teflo® filter has a significantly lower initial pressure drop of 1.3 inches of H₂O at 2.0 lpm, as compared to a glass fiber filter with 4 inches of H₂O. If the Teflon filter cannot be utilized, the current tentative choice of fiber substrates is the Gelman AE glass fiber filter. Impurities in the glass fiber matrix may, however, limit subsequent analyses for selected trace metals, and background analyses are currently in progress [June, 1994].

3.2.2 PM10

The MSP, Corp. PM10 aerosol inlet is shown in Figure 5. A perforated stainless steel 37 mm back-up support screen with 40 % open area is used beneath the Gelman Teflo® filter. The filter is placed directly into the inlet assembly to rest on the support screen. Teflon ring gaskets above and below the filter and the support screen provide sealing surfaces. The pressure of the screws in the inlet cap forces the top to in turn press the greased impaction ring insert against the upper gasket to seal against the filter and support screen. The degree of pressure [to be determined in June, 1994] on the top during inlet closure is determined by a special torque screwdriver press that holds that top while the two security screws are tightened as shown in Figure 6.

The impaction ring consists of a stainless steel, sintered annulus ring permanently mounted on an annulus skirt. The sintered metal material has a 10 µm porosity and is coated with a grease to trap particles greater than a specified aerodynamic size (10 µm) that strike the ring surface after passing through one of the 5 inlet holes in the cover. Particles less than 10 µm follow the air stream past the sintered material to be trapped on the filter material below.

The grease used on the sintered surface is a high vacuum silicon (VWR 59344-055, or equiv.) and is spread in a light coating onto the sintered surface using a stainless steel spatula (VWR 57941-020, or equiv.). The process is much like buttering toast. Great care must be taken to remove any grease that is left on any surface other than the sintered material. Grease removal is accomplished with a paper lab wipe. After greasing, the freshly coated impaction surface should be protected from settled dust, by covering (e.g. in a disposable petri dish). Prior to the grease application, the impaction ring and sintered surface must be cleaned in a soap (Liqui-Nox [VWR 21837-005] or equivalent) and tap water solution in a sonicated bath at least three times for at least 15 minutes, before the soap and water solution are replaced. Following the final sonication, the plates will be rinsed in distilled water, and then dried for 2 hours at 120 °F in a drying oven. The remaining components of the PM10 inlet (excluding the Tygon sleeve and tubing disconnect on the hose barb) must also be washed in the soap and water solution

between field operations, but for only one 15 minute sonication cycle prior to oven drying for 2 hours at 120 °F.

Aerosol samples will be collected at 2.0 lpm on filter substrates over a predetermined integration interval. The primary filter substrate for PM10 sampling will be Gelman 37 mm diameter, 2.0 µm Teflo® filters, or equivalent (Reference 2.2.3). Note that the 37 mm filter is not currently available in a 3.0 µm porosity. These 37 mm filters consist of a 2.0 µm porosity PTFE Teflon layer stretched across a 37 mm diameter, polymethylpentene annulus ring and heat sealed. To add rigidity to the Teflon layer during sampling and minimize pattern blinding caused by the filter holder support grid, a backup supporting material may be added under the Teflo® filter. Only fixed location indoor and outdoor aerosol sampling are anticipated for the PM10 size fraction, and only at locations collocated with Total Inspirable sampling. Only Teflo® filter materials will be used for PM10 sampling.

After the pre-weighed filter has been installed into the inlet, the leak-tightness of the inlet and the integrity of the filter are tested by measuring the pressure drop across the inlet at 2.0 lpm. Every loaded aerosol inlet sent to the field locations will be tested. Within a pre-determined limit, the pressure drop for a clean filter should be repeatable. Units not meeting this pressure drop indicate that either an internal leak is present in the inlet, or the filter has a mechanical flaw.

The testing is accomplished using the apparatus shown in Figure 4, where a flow-controlled personal sampling pump with a digital flowrate display (MSA ELF pump, or equivalent) provides the suction, a water manometer measures the pressure drop and an automatic bubble flowmeter determines if the correct flowrate of 2.0 lpm is maintained. This apparatus uses a quick-connection on the outlet of the aerosol inlet to provide a leak-tight seal. The procedure consists of 1) connecting the inlet, 2) turning the pump ON to an adjusted flow of 2.0 lpm, 3) allowing the flow to stabilize for 20 seconds, 4) reading the bubble flowmeter to determine if the flow is 2.0 ± 0.1 lpm (1.9 to 2.1 lpm), and 5) reading the water manometer pressure drop to determine if the pressure drop is 1.3 ± 0.2 inches of water (1.1 to 1.5 inches of H₂O). If the flowrate is outside the acceptance limit, readjust the pump and repeat the test procedure. If the flowrate is acceptable, but the pressure drop is outside the acceptance limit, the inlet should be disassembled to determine if a error was made in the assembly process. If no error is detected, the inlet assembly procedure should be repeated, and the pressure drop test repeated. If the pressure drop is still outside the acceptance limits, the inlet should be disassembled and the filter discarded and replaced with a new pre-weighed filter.

4. Pre- and Post-Exposure Filter Weighing

Pre- and post-exposure filter weighing activities will occur under controlled conditions at the RTI/RTP aerosol laboratory.

4.1 Analytical Laboratory Environment

The analytical laboratory used for pre- and post-weighing of 25 and 37 mm Teflo filters should have temperature and humidity controls to assure that a) the analytical balance is maintained within the manufacturer's specifications, and b) the filter substrates are not subjected

to abnormal humidity levels that may affect the tare weights. The Mettler AT261 balance system is temperature compensated according to the manufacturer for the relatively broad room temperature range from 5 to 40 °C. The temperature range maintained at the RTI/RTP aerosol laboratory (Room 427A) is expected to be within the range of 20 to 25 °C, which is well within the manufacturer's requirements. The weighing temperature will be recorded for each weighing session to assure compliance.

The relative humidity in the weighing laboratory environment has been shown to affect the tare weight of the filter substrate, depending on the hygroscopic nature of the material. The 25 mm and 37 mm Gelman Teflo® filters to be used have been tested for moisture stability in the range of relative humidities from 20 to 80 %. No statistically detectable weight changes from humidity were detected, within the analytical precision of the AT261 balance ($\pm 15 \mu\text{g}$). The filter storage and weighing relative humidity will be monitored and recorded to assure that the Rh stays within the 20 to 80 % range. The humidity must not only be maintained during the weighing operation, but during a filter equilibration period of 24 hours prior to the weighing.

4.2 Analytical Balance Description

The analytical balance to be used in weighing the aerosol samples collected in the NHEXAS program will be a model Mettler AT261. This balance resides in the RTI/RTP aerosol laboratory (room 427A), on a standard marble balance table, resting on a concrete slab floor. The balance utilizes an electronic pan sensor to determine the filter weight, with a tare range of 0 to 205 g and a reproducibility of $\pm 15 \mu\text{g}$. Internal calibration weights at 100 and 200 g are used to set the full-scale linearity, while an electronic auto-zero establishes the reference on demand. The draft shield doors are electrically opened and closed. The balance includes electronically adjustable pan damping levels to accommodate external vibrational frequencies that may be transmitted through the balance table.

All balance operations, including door opening and closing, can be controlled through an internal bi-directional data interface connected to an external computer utilizing the appropriate software. This interface also allow the digital display on the balance to provide instructional operator prompts to assure consistent operation. The operations of zeroing, internal calibration, door opening, prompting to load the filter or an audit weight, closing the door, waiting specific time intervals for pan stabilization, displaying the reading and transferring the number to a computer, opening the door, prompting for filter or weight removal, closing the door, and rechecking the zero prior to initiating another weighing sequence are all addressable.

A personal computer will be used to control all weighing operations and store weighing and quality assurance data. The Aerosol Weighing Laboratory Software will be written [*by mid-June, 1994*] to not only assimilate the pre- post-weighing data for the filters, but to collect the necessary validation information against which to set validation flags. The data management structure must be consistent with that at the NHEXAS field sampling locations for seamless integration of field operational data into the subsequent concentration computation process and final data validation.

4.3 Balance Zero/Calibration Procedures

The Mettler AT261 analytical balance will be auto-zeroed immediately prior to and zero checked immediately after each weighing operation. This will assure that the electronic state of "zero" is consistent across balance loading and unloading operations. The stabilization period prior to auto-zeroing will be controlled by the software. If the post weighing after filter or weight removal from the balance pan is not identically indicated as zero (to five places), the weighing process must be repeated.

Internal balance calibration/linearization using the built-in standard weights will be conducted prior to each weighing session, and after each 20 weighings (filters or audit weights). NTIS-traceable Class 3 audit weights of 50.0 mg (Troemner 7030-3, or equiv.) will be placed on the pan and weighed immediately after each internal calibration. Indicated weighing must be within $\pm 15 \mu\text{g}$, or the balance must be re-zeroed and re-linearized with the internal weights. If the balance still does not meet the tolerance limit for the audit weight, the balance must be serviced and re-calibrated by a qualified balance technician. The results of zero checks and audit calibrations will be stored by the computer software as part of the quality assurance program.

4.4 Filter Weighing Procedure

4.4.1 Filter Equilibration

Prior to pre- or post-weighing, all Gelman Teflo® filters must be equilibrated to a consistent relative humidity level between 20 and 80 % for at least 24-hours. This equilibration requires that the filters be placed on a tray in petri dishes with the top removed and covered with a single thickness lab wipe (Kimwipe EX-L or equiv.) for the entire period. Filters in uncovered petri dishes must not be allowed to be. This allows the laboratory air to readily reach the filter surface for equilibration. Filter for pre-weighing will already be properly identified petri dishes prior to equilibration. Exposed filters returning from field locations, must be removed from the aerosol inlets and placed in properly marked petri dishes for equilibration. In order to assure that the relative humidity and temperature remain within acceptable ranges during equilibration and weighing, the T (°C) and Rh (%) will be monitored routinely to determine hourly averages using a Solomat MPM4013 data system with thermistor and relative humidity sensor probes.

The static charge accumulated by the filters during storage or sampling must be removed prior to pre- or post-weighing. This is accomplished by holding the filter with forceps and passing the filter back and forth three times between two opposing Staticmaster ionizer units (VWR 58580-041 or equiv.) mounted in positioning units (VWR 58582-003 or equiv.) and separated by a gap of 2.5 cm. This must be done immediately prior to placing the filter on the balance pan. The ionizer units have only a 4 month active life and must be replaced periodically, based on the date stamped on the back of each unit. **SPECIAL NOTE:** Each ionizer contains 500 μC of Polonium 210 and must be treated according to a radioactive materials handling and disposal protocol.

4.4.2 Weighing Procedures

The weighing procedures are essentially identical for pre- and post-weighed filters, with the exception that post weighed filters must be placed on the balance pan with the exposed surface facing upward. The general weighing scheme for filters in chronological order, involves connection of the computer to the analytical balance, start-up of the weighing lab software, entry

of preliminary data, analytical balance checking, audit weighing, sample weighing, and validating the transfer and storage of sample and QC data. Filters must always be handled using special filter forceps (VWR 30033-042 or equiv.), grasping the filter only on the annulus plastic ring. The Teflon filter surface is extremely fragile, and sharp objects, including the forceps edges, must not be allowed to contact the Teflon membrane.

The entire weighing process and the assimilation of data are coordinated and implemented by the computer software prepared to operate the analytical balance. The computer is connected from the COM1 port to the special RS-232 connector on the back of the balance using the cable provided. The bar-code reader signal cable is connected to the COM2 port on the computer. The weighing software is written in QuickBasic and is run from the DOS prompt by typing "ALWP" (Aerosol Laboratory Weighing Program), followed by pressing the Enter key. The program is menu driven and provides for entry of information (a) manually from the keyboard, (b) electronically as weights from the balance interface, and (c) electronically as identifying numbers from the bar-code reader. Upon start-up, the program is initialized and displays the Main Menu. The structures of the Main Menu and the sub-menus are shown in Figure 7.

A weighing session (pre- or post-) is initiated by accessing the software Set-Up menu and entering the requested information, including: date and time (if incorrectly set by the computer clock), operator initials, current laboratory temperature and relative humidity. The Audit menu is then accessed and the balance checkout routine initiated. This includes automatic, computer controlled zero, setting of the balance variables, and internal linearization calibration. After checkout, the program requests the operator to place a certified, NIST-traceable, 50 mg audit weight on the balance as a calibration check. If this check is acceptable the filter samples are ready to be weighed.

The Sample Weighing menu is accessed to initiate the weighing procedures. Indicate whether the session is to pre-weigh or post-weigh filters. Pre-weighing involves assigning a NHEXAS-compatible number to the filter, associated with a previously printed, bar-coded, pressure-sensitive label. Post-weighing involves reading the bar-coded label and recalling the filter data file indicating the pre-weight. The post-weighing session will also require loading of the field sampling data into the computer for subsequent computation of mass concentration and overall assessment of all quality control checks. All weighings are preceded by an Auto-Zero. Note that the computer software controls the stabilization time between all weighing activities and actual data collection, for consistency.

If the balance is performing properly (meets software QC requirements) the access door will automatically open and the display request a sample to be weighed. If not, additional steps will be required. The filter is then removed from the petri dish (and handled at all times) using special filter forceps (Gelman 51147, or equiv.) and placed in the center of the balance pan. Other types of forceps with sharp points can easily damage the Teflon filter substrate. Grasp the filter with the forceps only on the outside annulus ring. Press the "PRINT" button on the balance to close the access door and start the weighing process. After the weight value has been transferred to the computer, the access door will automatically open for removal of the filter. Again press "PRINT" to close the access door and initiate a subsequent zero check to determine stability. If the post-zero is acceptable, door will open and request another sample. If the zero is not acceptable, the Auto-Zero cycle will be repeated and the filter sample must be re-weighed.

At the completion of sample weighing, the computer software, accessible thru the Summary menu, prepares a displayed or printed (selectable) description of the weighing session. If the samples have been pre- and post-weighed, and the field sampling data have been loaded (via diskette), the software automatically computes the sample mass concentrations. A summary display or printout (selectable) is then accessible on the Summary menu that lists by either filter number or participant ID#, all identifying information and quality control check information. A final check is made by the software to validate that the data are properly stored on the hard disk, and a back-up diskette is requested to be loaded for data storage.

4.5 Weighing Data Retrieval/Storage

The laboratory pre- and post-weighing data are stored directly on the hard disk of the aerosol laboratory computer. Back-up copies of the data are stored on diskette. The formatting, access and transfer of data are controlled by the ALWP computer software. Laboratory weighing and field sampling data are displayed to the screen and printed in summary formats using the Concentration Computation menu. Quality control data are displayed and printed using the Sample Validation menu. A security code system is employed within the software to limit data access.

4.6 Temporary Filter Archival

Pre-weighed filters are stored either in petri dishes or pre-loaded into an aerosol inlet. These samples must be stored in the laboratory under acceptable temperature and relative humidity conditions, prior to field shipment. Unexposed filter samples that have not been shipped to a field location for exposure within 90 days of pre-weighing should be re-weighed.

Post-exposure filter samples should be post-weighed promptly - within 5 working days - upon receipt from the field location. Since samples are normally express-shipped from each field sampling location (county) after each 12-day sampling period, no samples arriving at the RTI/RTP laboratory should be older than 18 calendar days since exposure. This schedule should be maintained to minimize sample degradation. All exposed samples must be carefully identified at all times with the associated filter ID#, and maintained at the weighing laboratory temperature and relative humidity. After removal from the aerosol inlets and post-weighing, filter samples must be stored in properly identified and capped petri dishes.

4.7 Filter and Data Transfer for Subsequent Analysis

A Custody Log must be kept on exposed samples arriving and leaving the weighing laboratory. This log identifies the sample ID#'s and the receiving individual and arrival date and time of each shipment from the field, and the transferring individual and the date and time of each shipment to the RTI analytical laboratory for subsequent analyses. The individual in the analytical laboratory receiving the filters must also be identified. No exposed, post-weighed filters and/or associated data are to be released from the weighing laboratory, unless specifically approved by a NHEXAS Co-Principal Investigator, as part of the overall NHEXAS analytical scheme.

5. Sample Validation

5.1 Valid Sample Requirements

The sample validation scheme for NHEXAS aerosol samples consists of a series of quality control checks conducted from the point of initial inspection of the unexposed filter through the post-weighing process. These binary flags are identified by the "Q number" code in Figures 8 through 12. They indicate that the specific flag was acceptable (value of 1), unacceptable (value of 2) or, in some case, unknown (value of 3). The acceptance criteria for each flag are based on the specific numeric variable ranges, also given in Figures 8 through 12, except for flag Q17, which is a subjective field operator judgment. If all previous flags have a value of 1 (acceptable on all counts) the overall flag, Q18, is set to a value of 1. The data validation flags for each sample are stored along with the mass concentration for each filter sample to assist in subsequent data interpretations.

5.2 Validation Procedures

The sample validation procedures for NHEXAS aerosol samples intuitively follow the requirements described in section 5.1. The validation checks are programmed into the ALWP software, such that at specific points in the sample flow scheme, a reading is compared with a target range to determine acceptability. The direct comparisons with the target ranges are performed by the software and the results flag value automatically stored in a data flag array with the associated filter ID#. The target ranges were established, based on the data quality necessary to meet the NHEXAS objectives. These objectives were correspondingly developed based on previous experience from studies such as the PTEAM Study described in reference 2.2.3.

6. Laboratory Aerosol Data Management

6.1 Filter Coding

[The filter coding formats had not been completely established at the time of this protocol preparation (June, 1994).] All filters used for the NHEXAS program that meet the required mechanical attributes (see section 4.1) will be assigned a unique identification code number that will be permanently maintained. The basic sample identification coding scheme for the RTI NHEXAS program is given in protocol RTI/ACS-AP-209-070. The small size of both the 25 mm and 37 mm filters precludes placing a number on the filter, necessitating that it be printed on an adhesive label and attached to the transporting container. In previous studies this container was typically a petri dish that was sent to the field location, where the filter was loaded by the field technician. The NHEXAS program will utilize laboratory personal at RTI to tare-weigh the filter, temporarily store it in a petri dish, and then pre-load tared filters into the appropriate size-fractionating aerosol inlet, prior to shipment to the field location. After tare weighing, the

unexposed filters are assigned a number which is printed on triplicate labels. One of these labels is removed from the backing paper and placed on the base tab of a filter petri dish (Gelman 7231 or equivalent) as shown in Figure 14, while the duplicate labels are attached (still on their backing paper) to the petri dish with a rubber band.

The aerosol inlets will be individually numbered with an engraved ID#'s on the back. This aerosol inlet ID# is linked to the filter ID# in the computer filter tracking program after the filter is removed from the storage petri dish and loaded into the inlet at the RTI laboratory. The duplicate filter ID# label is then placed on the back of the aerosol inlet as shown in Figures 13. The label remaining on the petri dish can be removed and discarded to permit reuse of the petri dish. To preserve the integrity of the filter tare weighing and collected aerosol, the entire IOM and PM10 aerosol inlet assemblies will be shipped to and from the field sampling location as sealed units.

6.2 Bar Code Identification

[The bar code identification system and software were not completely identified at the time of this protocol preparation (June, 1994)] An adhesive label will be located on the petri dish tab after filter tare weighing, and on the back of the aerosol inlet (see Figures 13 and 14). Labelworks® software by American Microsystems will be used from within Windows® to print the labels on a laser jet printer. This code label displays the filter ID#, and includes a standard bar code (Code 128 format) that can be read by a standard light pen attached to the serial port of either the NHEXAS lab or field lap top computers. Code 128 format includes an internal check-sum computation character to validate the accuracy of the optical reading. The light pen data entry is controlled by custom prepared software for the identification and tracking of filters and aerosol inlets at the RTI/RTP laboratory. The light pen produces ASCII characters from the bar code, representing the filter ID# (and check-sum number), which is then read into a selected data file.

6.3 Computer Data Entry/Retrieval

The identification and tracking of filter samples and aerosol inlets during pre- and post-weighing at the RTI/RTP laboratory will be accomplished with the aid of a custom-prepared weighing laboratory software program. This program will operate from the DOS prompt and be written in QuickBasic. It will accomplish several functions, including: (a) reading the filter numbers from the bar code labels, (b) controlling the Mettler analytical balance functions (e.g. auto-zero, auto-calibration, stabilization time, etc.) to assure proper operation and providing operator prompts on the balances digital display screen, (c) providing error-free electronic input of the weighing data from the balance to the computer data files, (d) storing and retrieving weighing and quality control data, (e) computing the mass collections between pre- and post-exposure of the filters, (f) assimilating the diskette-based field sampling data (e.g. flowrate, integration time, sampling temperature, etc.) with the weighing data to compute mass concentration data of known quality, and (g) summarizing the measurement and quality assurance data and assigning validation codes for subsequent processing. Data management

functions of the weighing laboratory software include data storage and retrieval of 1) the status and locations of the IOM and PM10 aerosol inlets, 2) the ID#'s of the filters and the assigned aerosol inlet, 3) the pre-weights of the filters, lab and field blanks, 4) the post-weights of the filters, lab and field blanks, 5) supporting information relative to the weighing process (e.g. balance operator's name, dates and times, room temperature and humidity, etc.), and 6) the validation codes assigned to each parameter. These data will be stored on the computer hard disk as well as utilizing daily data backup routines to diskette.

The weighing laboratory software will be directly compatible with the data formats and file structures of the field sampling software. This is required for aerosol mass concentration calculations as well as summarizing the quality control checks. The weighing data, field sampling data, quality control data and computed concentrations will be summarized for each county (every 2 weeks) in a data file for transmittal to the designated NHEXAS database manager. This transmittal will consist of a summary printout of the data and a 3-1/2" diskette in the data format requested by the database manager.

Mass concentrations will be computed within the software based upon the sampled volume, with the ambient flowrate (\bar{Q}_{amb}) at field conditions, corrected to standard flowrate (\bar{Q}_{std}) at standard conditions ($P_{STD} = 760$ mm Hg, $T_{STD} = 298$ °K). The correction equation is:

$$\bar{Q}_{std} = \bar{Q}_{amb} (P_s / T_s) (298 / 760) \quad (1)$$

where: P_s = mean field site atmospheric pressure over sampling period, mm Hg
 T_s = mean field site temperature over sampling period, °K

The total air volume sampled at standard conditions is calculated as:

$$V_{std} = (\bar{Q}_{std}) t \quad (2)$$

where: V_{std} = sampled volume corrected to standard conditions, m³
 t = total sampling time, minutes

The mean aerosol mass concentration is calculated as:

$$C = (W_f - W_i) (10^6) / V_{std} \quad (3)$$

where: C = mass concentration, µg/m³
 W_f = final filter weight, g
 W_i = initial filter weight, g

7. QA/QC Procedures

7.1 Sample ID Codes

As noted in section 6.1, the ID codes for aerosol samples are designed to uniquely identify the samples and sampling systems. The addition of specific leading identifiers as part of the ID codes, such as 25xxxxx and 37xxxxx for 25 and 37 mm filters, should minimize the misinterpretation of results. The addition of bar-coded labels will minimize errors in data transcription and help to link filter numbers to specific sampling systems in cases of suspected sampler malfunctions.

7.2 Sample Custody

The custody of exposed aerosol filter samples will be documented at all times, from the point of removal of the exposed filter from the sampling inlet to the final archive for subsequent analysis. This custody will be documented in permanent logs at both the field sampling mobile laboratory and the RTI aerosol analysis laboratory at RTP, NC. The Field Coordinator will be responsible for the field log, while the Laboratory Coordinator will be responsible for the laboratory log. These logs will identify the sample custodian, and current location and the date and itinerary of any transfer shipments. This will also permit an assessment of storage conditions prior to post-weighing and subsequent analysis. Samples will not be released for analyses beyond mass concentration, without approval from a NHEXAS Co-Principal Investigator. The subsequent analyses must be part of an overall NHEXAS analysis plan, since most analyses either degrade the sample or reduce the quantity of sample available for additional analyses.

7.3 Shipment/Field Blank Stability

The tare weighed filter substrates and the collected aerosol material deposited on the filters must be protected during handling, sampling and shipping operations. Shipment of the pre-weighed filters to and from the field in the aerosol inlets provides a substantial degree of protection. Utilization of express shipments (overnight), minimize the time the sample shipments are not under the direct control of project personnel, and hence under controlled-environment conditions. Outside influences that may affect the tare weight or the aerosol collected on the filter and must be avoided, include: excessive temperatures ($>35^{\circ}\text{C}$), moisture extremes ($<20\%$ and $>80\%$ Rh), and severe shocks from dropping the aerosol inlet on a hard surface (e.g. concrete floor). If samples are exposed to these conditions in the field, the operators should note the conditions in the operator comment field, to possibly explain concentration data that are extreme.

In order to assess the stability of the tare weight, a blank, pre-weighed filter will be loaded into an inlet and treated as a sampled filter - without actually drawing air through the filter - for each filter batch shipped (1 per county). This inlet will be shipped to the field, taken to a randomly selected field sampling location, clipped outside the bluff body form of either the indoor or outdoor sampler adjacent to the other inlets, and returned the RTI/RTP laboratory for post-weighing. The comparison of pre- and post-weights for this field blank will permit estimation of the tare weight stability.

7.4 External Balance Auditing

Randomly, during each 5 month period the RTI aerosol laboratory at RTP, NC is weighing samples for the NHEXAS program [15 months total are expected], an external (to RTI) auditor will visit the laboratory to check the performance of the analytical balance. This auditor will be selected by EPA and will provide NIST-traceable audit weights against which to judge the accuracy of the balance. During the first audit visit, this auditor will also review the handling, weighing and archival procedures be used, and subjectively address the expected quality and adequacy of the procedures.

7.5 Standard Weights

The internal audit weights used to check the accuracy of the balance used to weigh NHEXAS samples will be NIST-traceable, Class 3 weights. The weight range of the audit weights should be within 25% of the expected sample weights (30 to 50 mg for 25 mm filters, and 80 to 120 mg for 37 mm filters).

7.6 Exposed Filter Re-weighing

In order to assess the precision of the pre- and post-weighing processes, 5 % (1 out 20) filters will be randomly selected at the end of each weighing session, for re-weighing. The expected precision of the re-weighing is $\pm 5\%$, or better. Re-weights that exceed this limit, suggest that the weighing process may have problems. If the re-weight is $> \pm 5\%$ of the original weight, the sample should be re-weighed a 3rd time for verification. If the re-weights are $> \pm 10\%$ of the original weight, a problem has occurred that must be identified and the entire session of samples re-weighed.

7.7 QA Data Entry/Tracking

The QA data entry for the weighing process will occur during both laboratory and field sampling operations, as part of the aerosol laboratory and field data collection software applications. These QA data checks (described in section 5.2) are computed and tracked by the software. Summary reports can be printed by the ALWP software for the QA data representing the samples from each NHEXAS sampling county. This summary presents the QC checks compared with established control limits in tabular and graphical formats.

Figure 1. NHEXAS Aerosol Sample / Data Flow

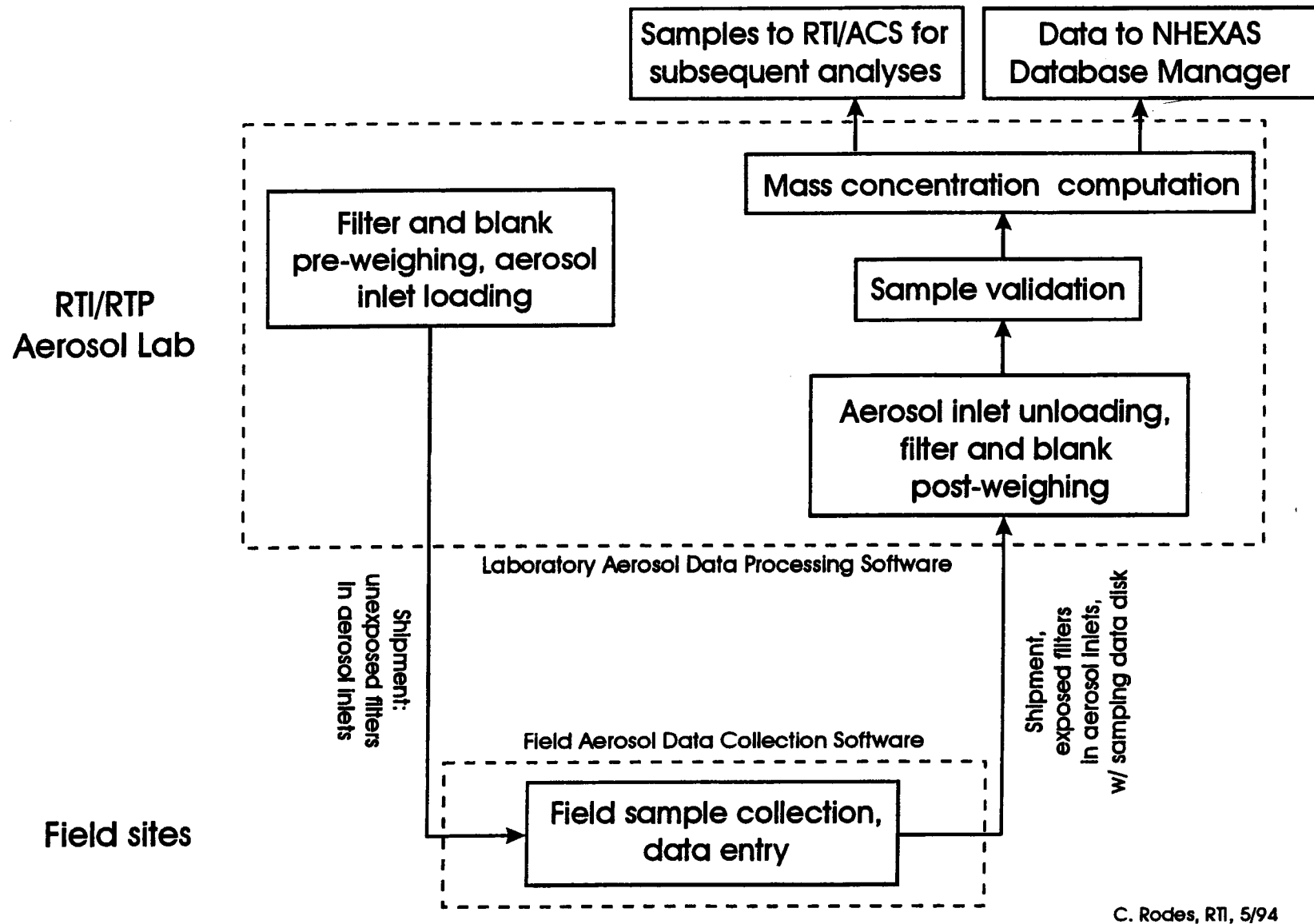


Figure 2. IOM Total Inspirable Aerosol Sampling Inlet and Cassette, SKC, Inc.

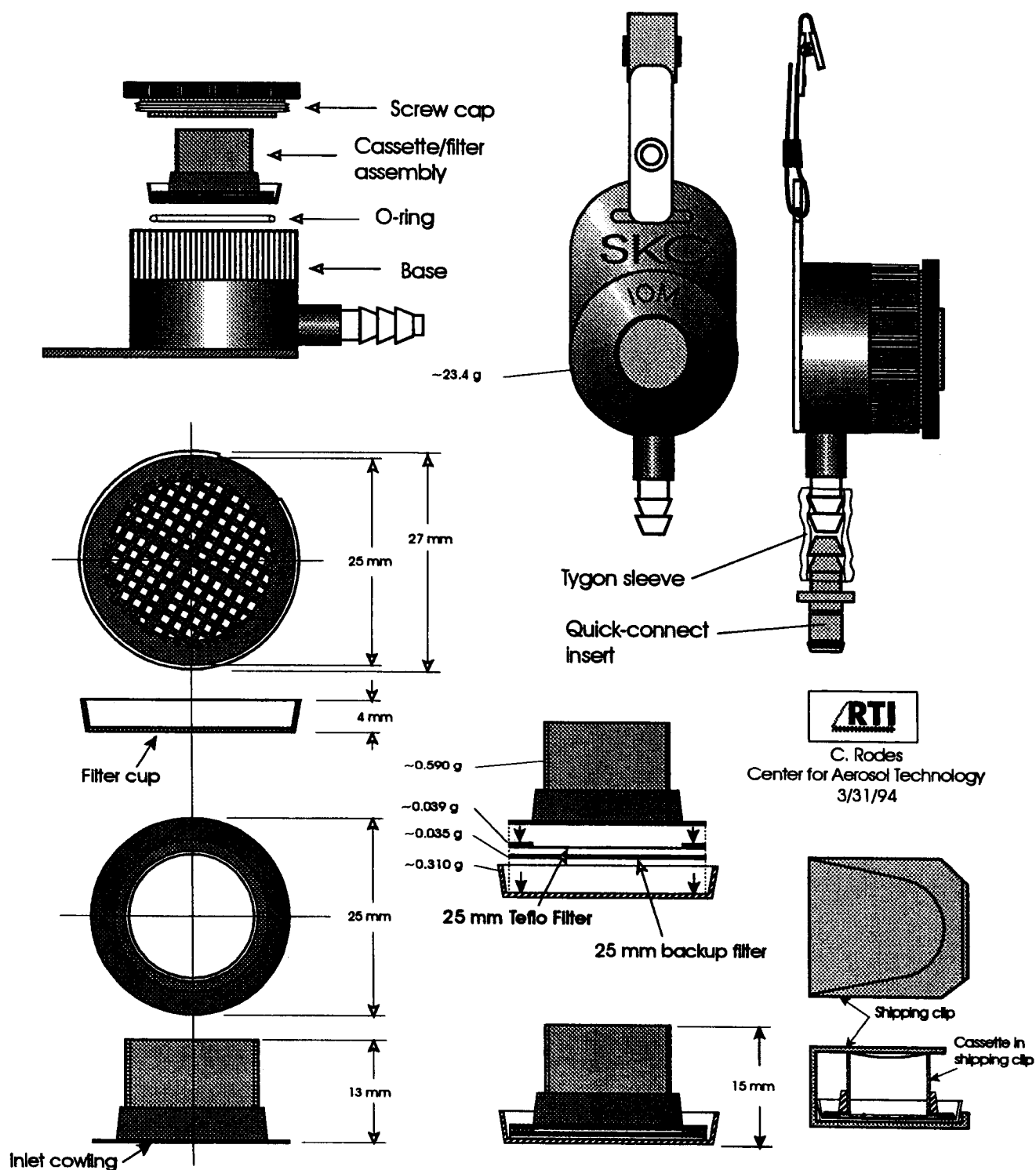


Figure 3. IOM Total Inspirable Aerosol Sampling Inlet Screw Cap Closure

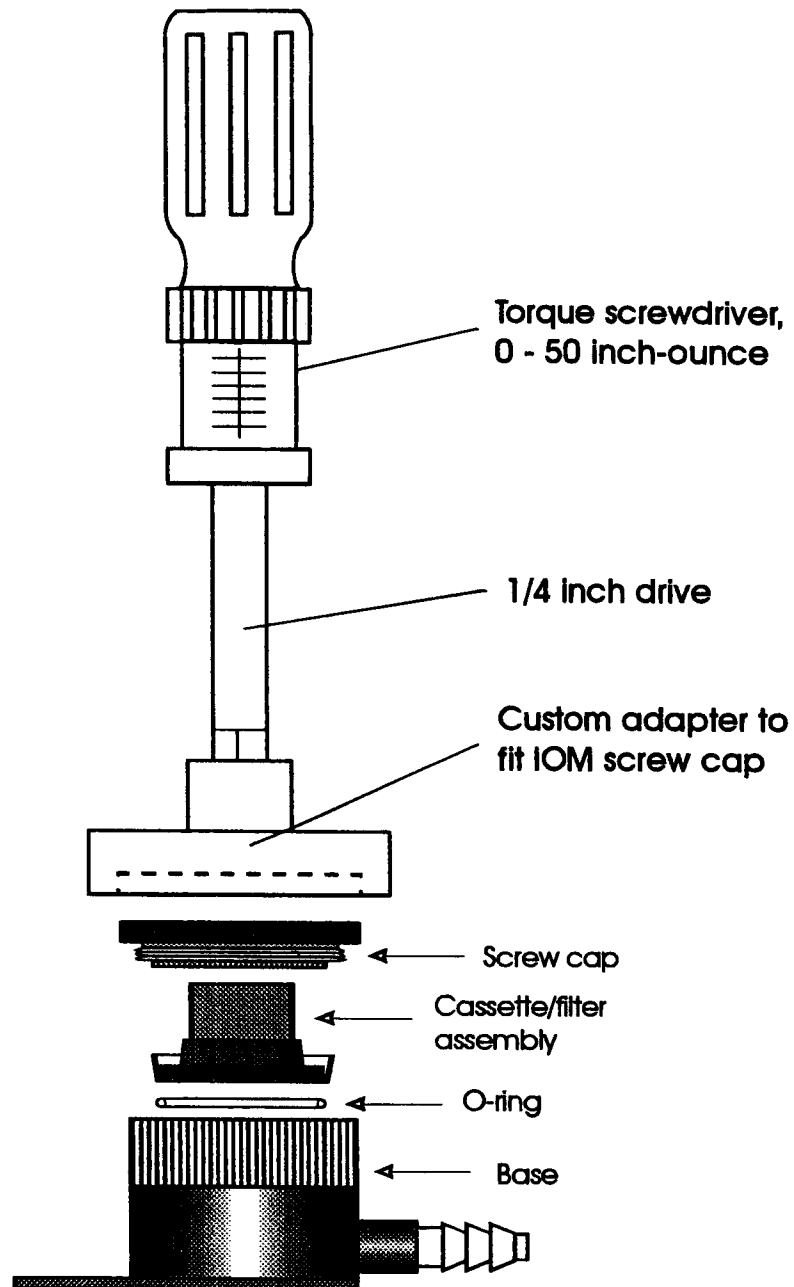


Figure 4. Filter/Aerosol Inlet Initial Pressure Drop Test Apparatus

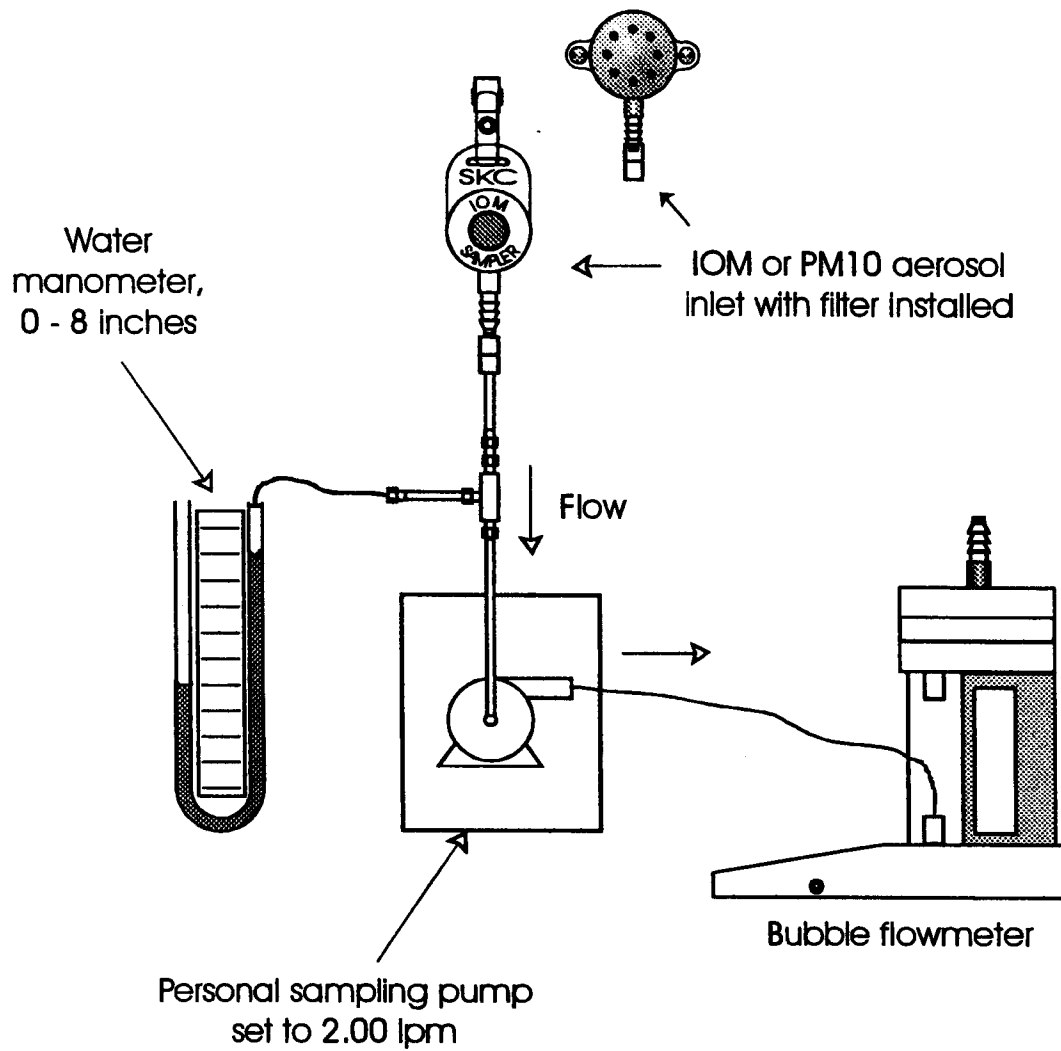


Figure 5. PM10 Aerosol Sampling Inlet, MSP, Corp.

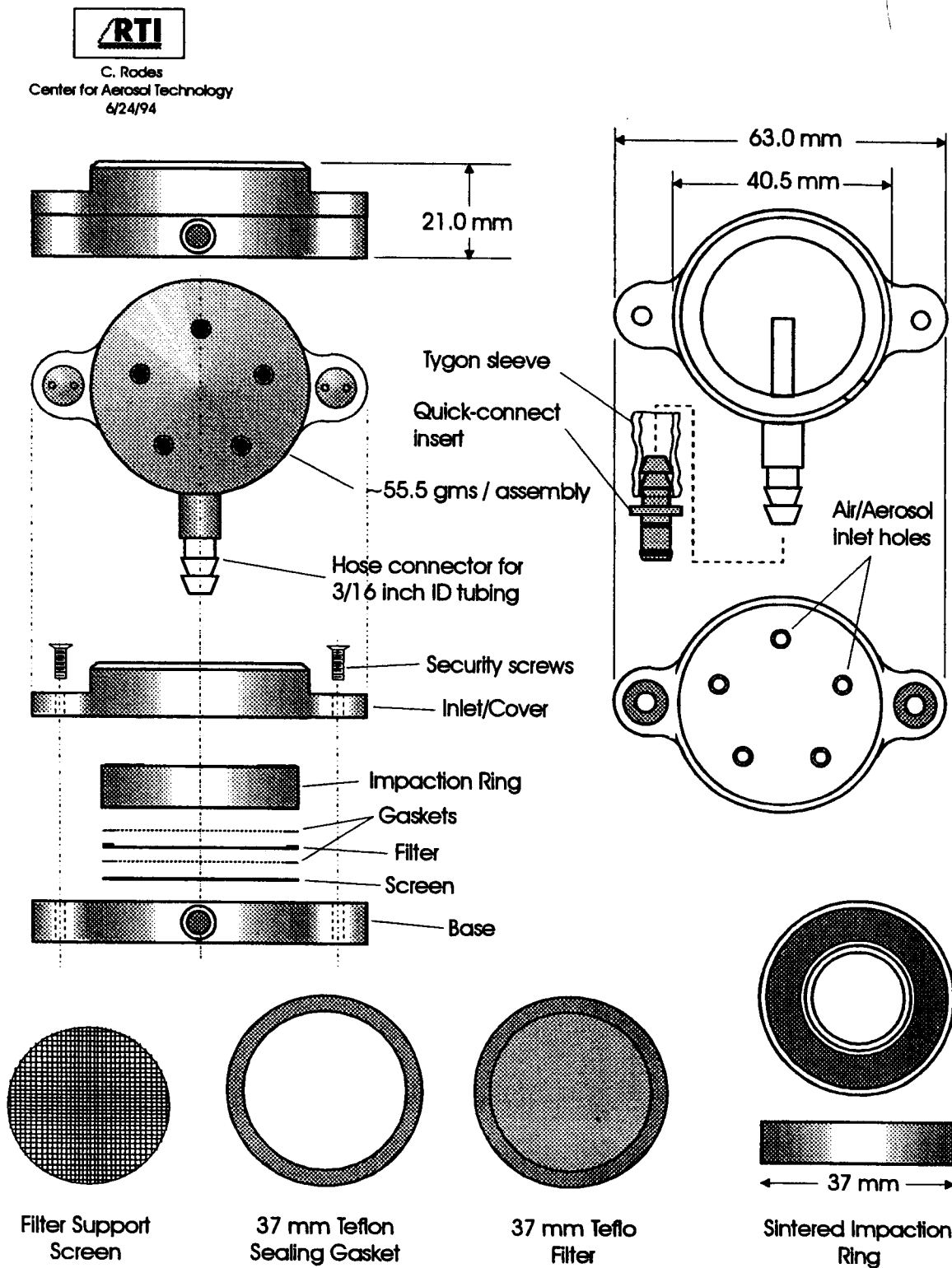


Figure 6. PM10 Aerosol Sampling Inlet, Cap Closure

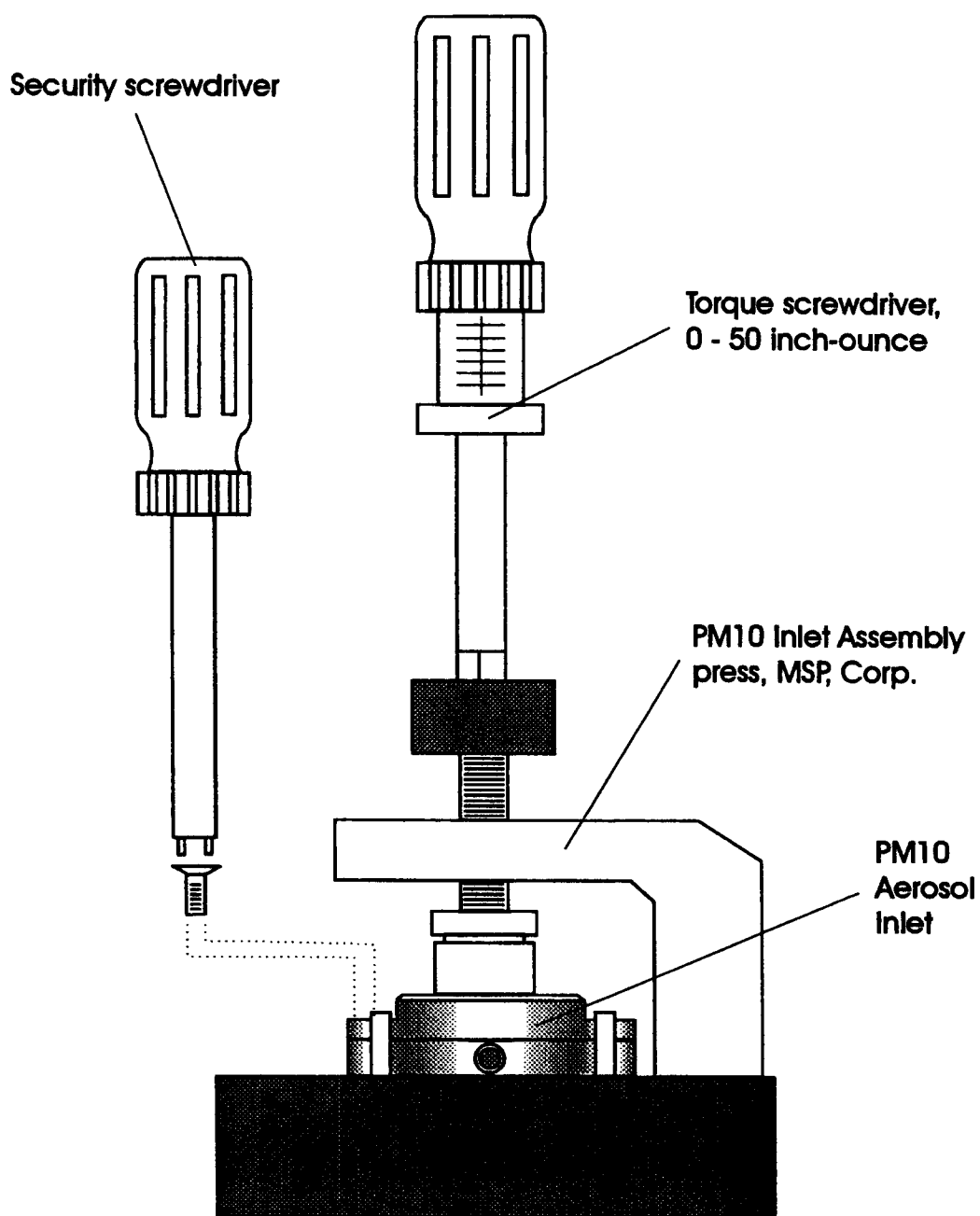
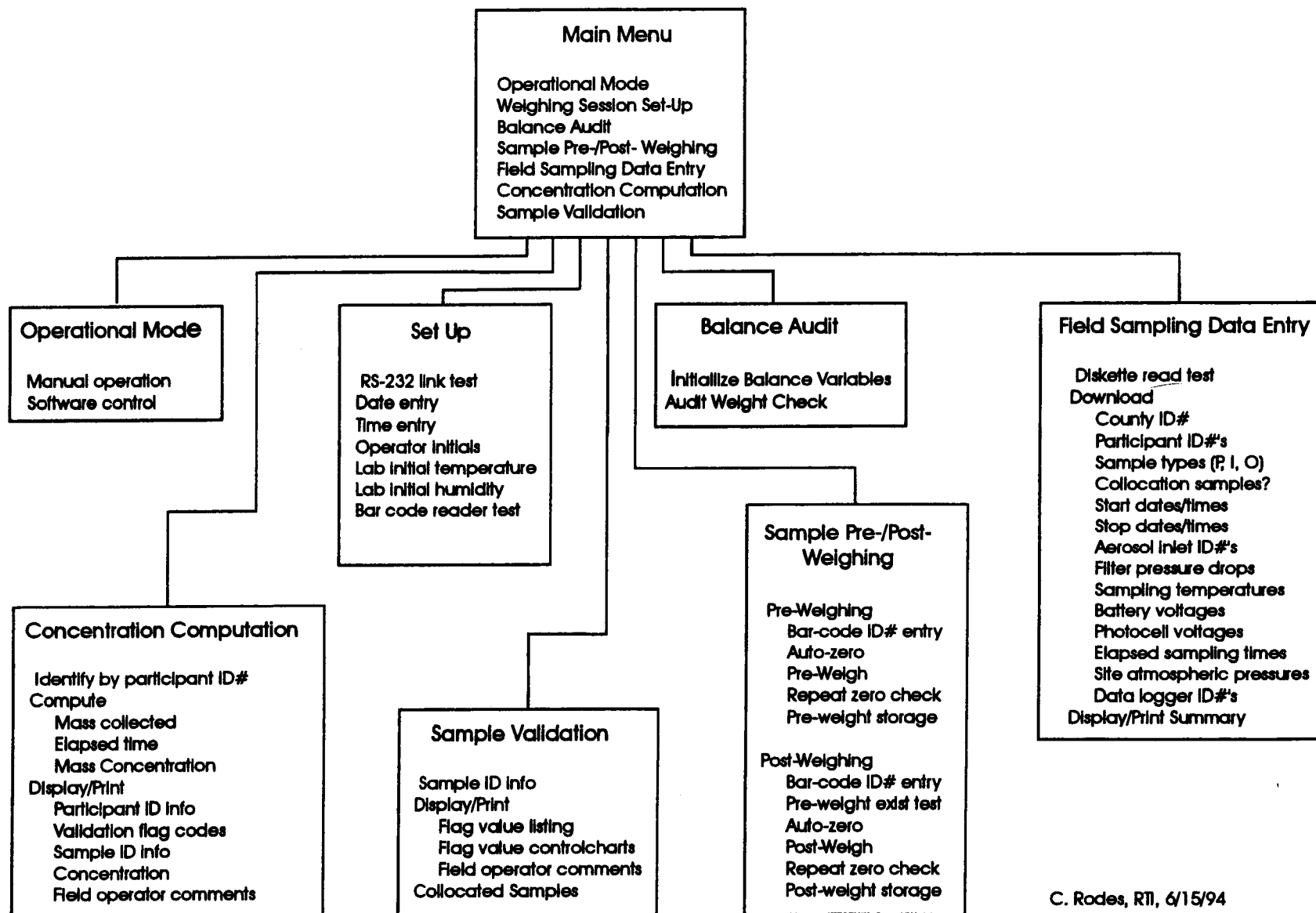


Figure 7. Aerosol Laboratory Weighing Program
Menu Structure / Variable Processing [proposed]



C. Rhodes, RTI, 6/15/94

**Figure 8. NHEXAS DATA MANAGEMENT:
Filter Pre-Weighing and Aerosol Inlet Loading**

ACTIVITY: FILTER PRE-WEIGHING

Pre-weigh date (mo/da/yr): xx/xx/xx
 Balance operator's initials: xxx
 Pre-weigh lab temp: xx.x deg C
 Pre-weigh lab Rh: xx.x %
 Standard reference weight: x.xxxxx g
 Expected reference weight range (low, high): x.xxxxx g, x.xxxxx g
 Reference weight within range? (Y - 1, N - 2): x **Q 1**
 25 mm filter ID#: 25xxxxx
 Filter pre-weight: x.xxxxx g
 Expected pre-weight range (low, high): x.xxxxx g, x.xxxxx g
 Pre-weight within range? (Y - 1, N - 2): x **Q 2**
 37 mm filter ID#: 37xxxxx
 Filter pre-weight: x.xxxxx g
 Expected pre-weight range (low, high): x.xxxxx g, x.xxxxx g
 Pre-weight within range? (Y - 1, N - 2): x **Q 3**

ACTIVITY: AEROSOL INLET LOADING

Loading date (mo/da/yr): xx/xx/xx
 Loader's initials: xxx
 25 mm filter ID#: 25xxxxx
 IOM Inlet ID#: IMxxxxx
 Filter type - sample (S), lab blank (L), field blank (F): x
 37 mm filter ID#: 37xxxxx
 PM10 inlet ID#: PMxxxxx
 Filter type - sample (S), lab blank (L), field blank (F): x

**Figure 9. NHEXAS DATA MANAGEMENT:
Field Data Entry and Collection**

ACTIVITY: FIELD DATA ENTRY/COLLECTION

Field Operator Keyboard data entry:

County ID#: xxxxxx

Participant ID#: xxxxxx

Sample type, Personal (P), Indoor (I) or Outdoor (O)?: x

Field sample (F) of collocated sample (C)?: x

Atmospheric pressure, mm Hg?: xxx.x

Portable Computer Internal data entry:

Sampling start date

Sampling start time (24 hr clock)

Sampling stop date

Sampling stop time (24 hr clock)

Light Pen data entry:

Aerosol Inlet ID #

Data Logger Download:

Filter pressure drop, inches of water

Sampling temperature, deg. C

Battery pack voltage, VDC

Photocell voltage, mVDC

Elapsed sampling time, min.

Data logger serial number

**Figure 10. NHEXAS DATA MANAGEMENT:
Aerosol Inlet Unloading and Filter Post-Weighing**

ACTIVITY: AEROSOL INLET UNLOADING

Unloading date (mo/da/yr): xx/xx/xx

Unloader's initials: xxx

25 mm filter ID#: 25xxxxx

IOM inlet ID#: IMxxxxx

Filter type - sample (S), lab blank (L), field blank (F): x

Filter ID# matches stored inlet ID# (Y - 1, N - 2): x Q4

Filter visual inspection code (OK - 1, Void - 2, ? - 3): x Q5

Inspection comment: xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx

37 mm filter ID#: 37xxxxx

PM10 inlet ID#: PMxxxxx

Filter type - sample (S), lab blank (L), field blank (F): x

Filter ID# matches stored inlet ID# (Y - 1, N - 2): x Q6

Filter visual inspection code (OK - 1, Void - 2, ? - 3): x Q7

Inspection comment: xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx

ACTIVITY: FILTER POST-WEIGHING

Post-weigh date (mo/da/yr): xx/xx/xx

Balance operator's initials: xxx

Post-weigh lab temp: xx.x deg C

Post-weigh lab Rh: xx.x %

Standard reference weight: x.xxxxx g

Expected reference weight range (low, high): x.xxxxx g, x.xxxxx g

Reference weight within range? (Y - 1, N - 2): x Q8

25 mm filter ID#: 25xxxxx

IOM inlet ID#: IMxxxxx

Filter type - sample (S), lab blank (L), field blank (F): x

Filter post-weight: x.xxxxx g

Expected post-weight range (low, high): x.xxxxx g, x.xxxxx g

Post-weight within range? (Y - 1, N - 2): x Q9

37 mm filter ID#: 37xxxxx

PM10 inlet ID#: PMxxxxx

Filter type - sample (S), lab blank (L), field blank (F): x

Filter post-weight: x.xxxxx g

Expected post-weight range (low, high): x.xxxxx g, x.xxxxx g

Post-weight within range? (Y - 1, N - 2): x Q10

Figure 11. NHEXAS DATA MANAGEMENT: Collected Filter Validation

ACTIVITY: COLLECTED SAMPLE (FILTER) VALIDATION

County ID#: xxxxxx

Participant ID#: xxxxxxxx

Sampling start date (mo/da/yr), stop date (mo/da/yr): xx/xx/xx, xx/xx/xx

Sampling 24-hr clock start time (hr:mn), stop time (hr:mn): xx:xx, xx:xx

25 mm filter ID#: 25xxxxx

Personal (P), Indoor (I) or Outdoor (O)?: x

Field sample (F) of collocated sample (C)?: x

37 mm filter ID#: 37xxxxx

Personal (P), Indoor (I) or Outdoor (O)?: x

Field sample (F) of collocated sample (C)?: x

Maximum allowable pressure drop: xx.x inches water

Pressure drops less than max? (Y - 1, N - 2): x Q 1 1

Maximum short duration pressure drop > 25 inches water: x.x min.

Duration not exceeded? (Y - 1, N - 2): x Q 1 2

Expected temperature range (low, high): xx.x deg C, xx.x deg. C

Within temperature range? (Y - 1, N - 2): x Q 1 3

Expected minimum battery voltage: x.xxx VDC

Battery voltage > minimum? (Y - 1, N - 2): x Q 1 4

Expected elapsed time range (low, high): xxx.x min, xxx.x min

Within elapsed time range? (Y - 1, N - 2): x Q 1 5

Expected flowrate range (low, high): x.xx lpm, x.xx lpm

Mid-period and final flowrate checks within range? (Y - 1, N - 2): x Q 1 6

Field operator sample assessment (Valid - 1, Invalid - 2): x Q 1 7

Field operator comment: xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx

Data validation flag (Q 1 thru Q 1 7 = 1 - 01, 1 or more = 2 - 02, ? - 03): x,

Q 1 8

Figure 12. NHEXAS DATA MANAGEMENT: Aerosol Concentration Computation

ACTIVITY: CONCENTRATION COMPUTATION

Sample Identification:

County ID#: xxxxxx

Participant ID#: xxxxxxx

Sampling start date (mo/da/yr), stop date (mo/da/yr): xx/xx/xx, xx/xx/xx

Sampling 24-hr clock start time (hr:mn), stop time (hr:mn): xx:xx, xx:xx

25 mm filter ID#: 25xxxxx

Personal (P), Indoor (I) or Outdoor (O)?: x

Field sample (F) of collocated sample (C)?: x

37 mm filter ID#: 37xxxxx

Personal (P), Indoor (I) or Outdoor (O)?: x

Field sample (F) of collocated sample (C)?: x

Data validation flag = 01 (Y - 1, N - 2): xx

Concentration Computation [see equations (1), (2) & (3), Section 6.3]:

Filter pre-weight: x.xxxxx g

Filter post-weight: x.xxxxx g

Computed weight change: xxx micrograms

Sampling elapsed time: xxx.x min.

Computed sampling volume (sampling conditions): x.xxx cubic meters

Average sampling temperature: xx.x deg. C

Average sampling atmospheric pressure: xxx.x mm Hg

Computed sampling volume (standard conditions): x.xxx cubic meters

Computed mass concentration: micrograms / standard cubic meter

Figure 13. Bar Coded ID Labels on IOM and PM10 Inlets

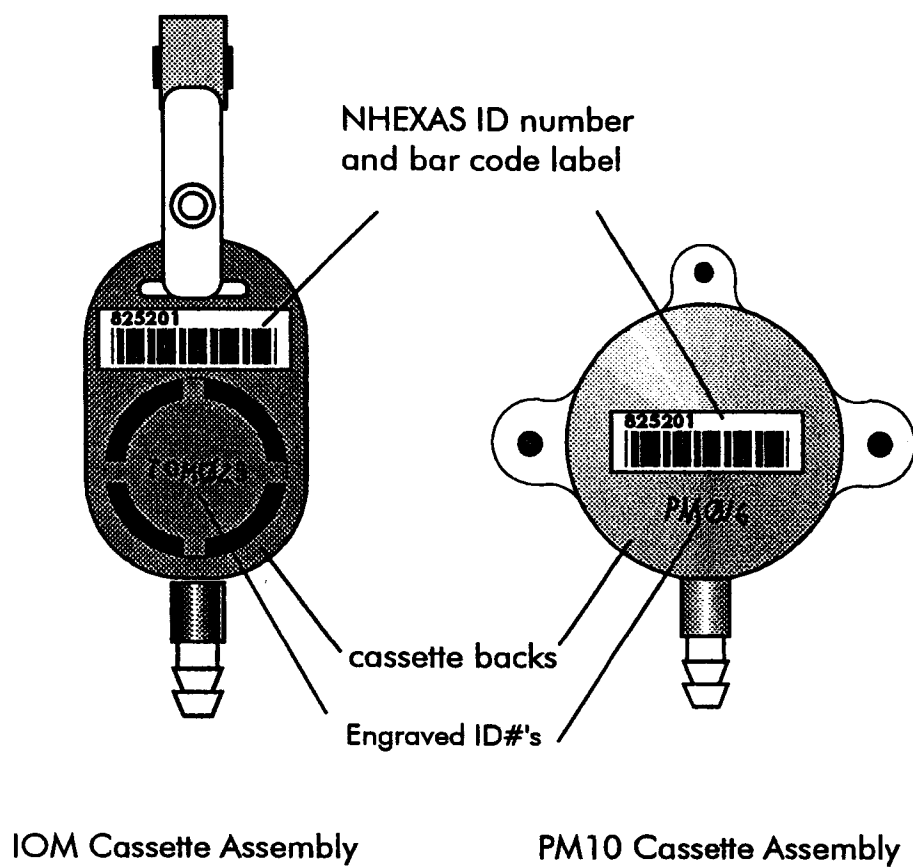


Figure 14. Filter Storage Petri Dish for
NHEXAS Aerosol Filters

