

Development of Quality Control Parameters and Electronic Data Recording for an Ambient Air Particle Inhalation Exposure System

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Research and Development at EPA



- 1,950 employees
- \$700 million budget
- \$100 million extramural research grant program
- 13 lab or research facilities across the U.S.
- Credible, relevant and timely research results and technical support that inform EPA policy decisions

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National Health and Environmental Effects Research Laboratory

- NHEERL is EPA's focal point for scientific research on the effects of contaminants and environmental stressors on human health and ecosystem integrity.
- NHEERL's Mission and goals help EPA identify and understand the processes that affect our health and environment, and evaluate the risks that pollution poses to humans and ecosystems.

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High Priority Research Areas



- Human Health
- Particulate Matter
- Drinking Water
- Clean Water
- Global Change
- Endocrine Disruptors
- Ecological Risk
- Pollution Prevention
- Homeland Security

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Experimental Toxicology Division



- ETD conducts multidisciplinary research to improve the scientific basis of risk assessment for pulmonary, cardiovascular, hepatic, renal, and immunotoxicity
- Research facilitates the use of pharmacokinetic data in the risk assessment process
- Animal Inhalation Exposure Facility allows researchers to study the health effects of gases, vapors, and aerosols

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Animal Inhalation Exposure Facility Team



- Performs inhalation exposure studies of gases, vapors, and aerosols including real world particulate matter (PM), and combustion emissions (e.g. fuel oil, natural gas, coal, diesel)
- Conducts air PM sampling, collection, and characterization
- Current projects include studies of concentrated real world PM air pollutants - and the role of size and components - on cardiopulmonary disease

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Inhalation Exposure Studies of Concentrated Air Particles



- Determine health effects of “real world, real time” PM on cardio-pulmonary systems of various animal models
- Simultaneous real time exposures using multiple modes of concentrated ambient air PM
 - UCAPs – Ultra-fine (μf) mode aerosols ($< 0.3 \mu\text{m}$)
 - CAPs - Fine mode aerosols ($0.3 < X < 2.5 \mu\text{m}$)

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AIEF UCAPs/CAPs Small Animal Exposure Systems

- 0.3 m^3 stainless steel and glass chambers for Ultra-fine PM, fine PM, and control groups
- Exposure chamber atmosphere monitoring, characterization, and control
- “Real time, real world” ambient air particles (not resuspended or laboratory generated)
- State-of-the-art particle growth and Slit Virtual Impactor particle concentrating technology

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AIEF UCAPs/CAPs Small Animal Exposure Systems

- PM size fractionating eliminates incoming particles larger than PM₁₀, PM_{2.5} and PM_{0.3}
- Specialized equipment concentrates remaining particles with minimal effect on particle chemistry
- Monitoring instruments measure and characterize atmosphere delivered to animals
- Repeated exposures, 6 hr/d, 5 d/wk, 13 wks
- Ambient weather station

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PM Delivery System

- 10,000 Lpm ambient air containing PM drawn into size selective inlet (SSI) impactors
- Entering ambient PM size fractionated by SSIs to <2.5 µm
- 5000 Lpm each delivered to 2 different particle concentrating systems
- Slit Virtual Impactor (SVI) technology used to concentrate PM for independent, simultaneous UCAPs and CAPs studies

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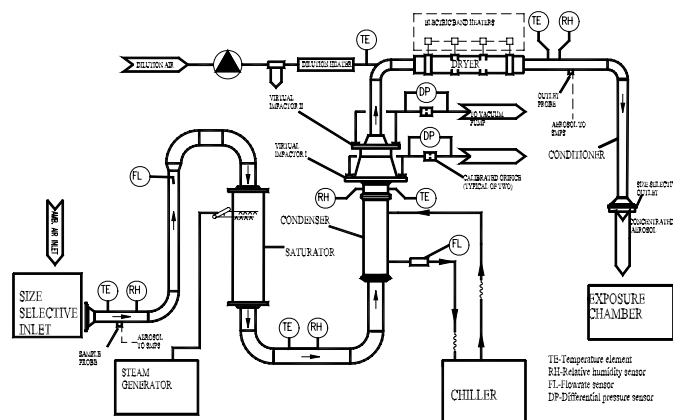
Ultra-fine PM Concentrating Equipment

- SVI technology is not directly effective on ultra-fine PM, therefore, requires particles $<0.3 \mu\text{m}$ be grown to $>1 \mu\text{m}$ for concentrating
- Concentrating effect optimized at vapor super saturation ratio, $S_r \sim 3.0$
- Supersaturated water vapor chamber grows μf PM to $>1.5 \mu\text{m}$ in seconds
- Multistage SVIs concentrate grown PM $>40 \times$
- Reshaper dries particles to original size and SSO eliminates PM $> 0.3 \mu\text{m}$ prior to delivery to chamber

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UCAPs Schematic



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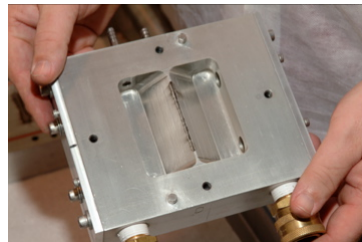
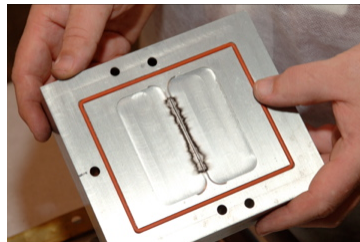
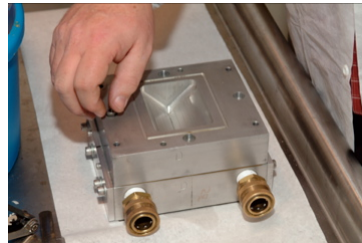
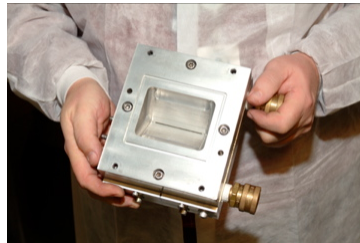
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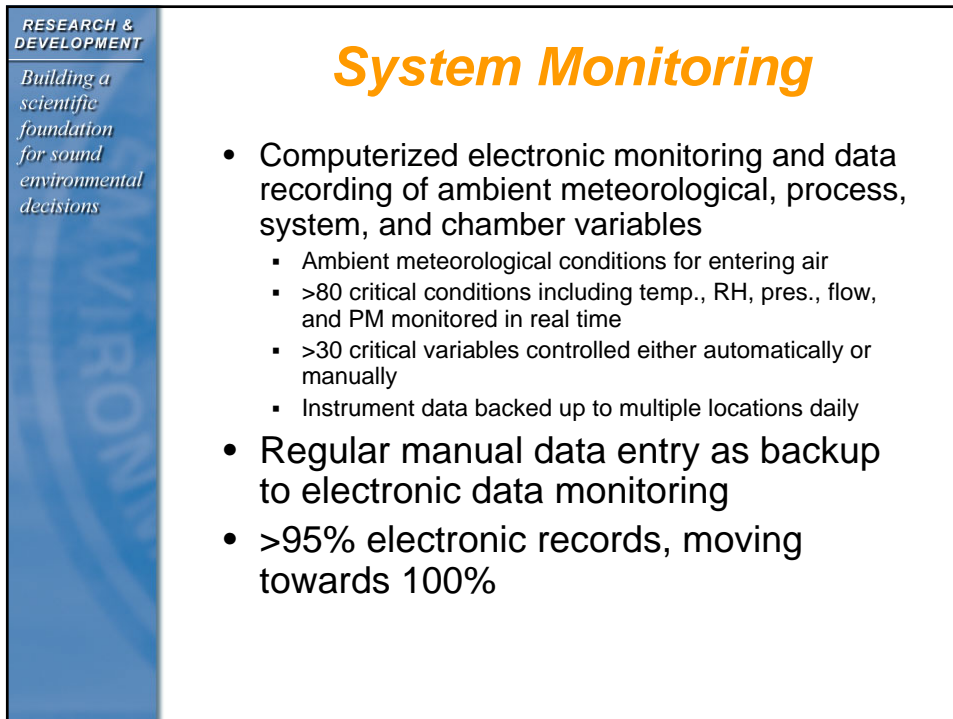
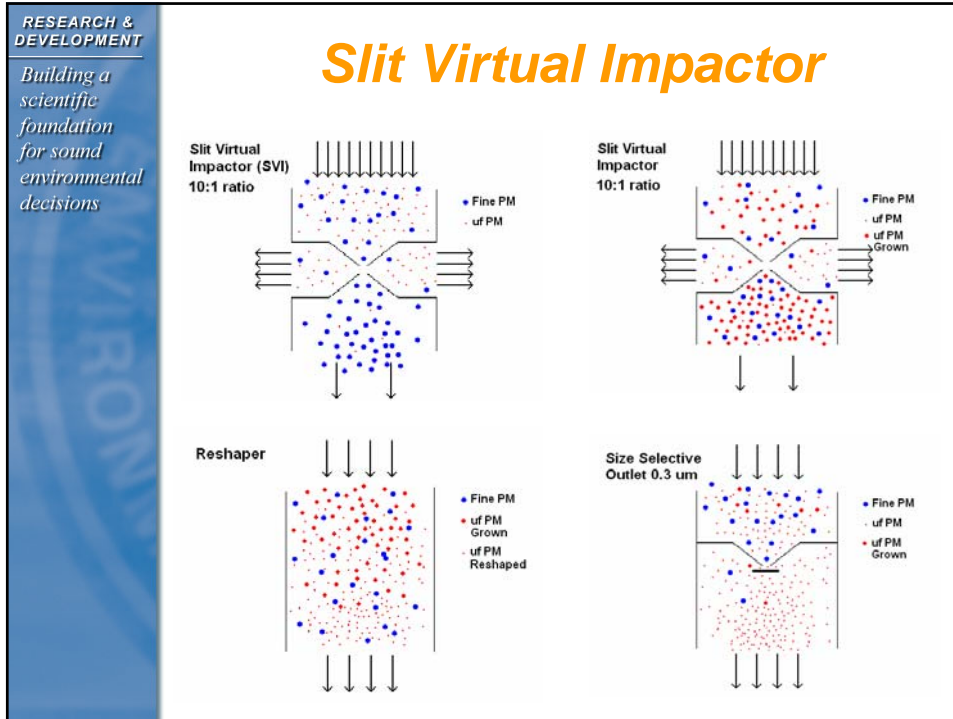


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Slit Virtual Impactor

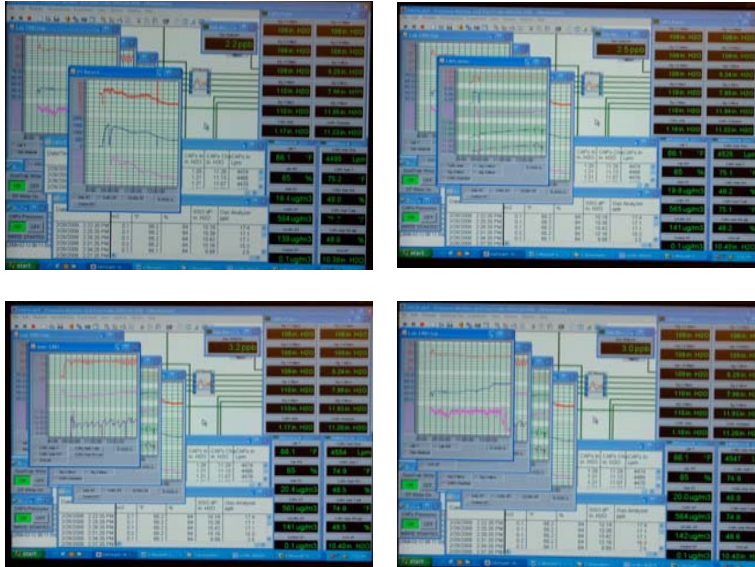




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DASYLab Monitoring



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HUCAPs Monitoring



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Particle Monitoring



- Ambient and Chamber atmospheres monitored by continuous filter samples and electronic instruments for meteorological conditions and pollutant levels
- Filters provide mass concentration in $\mu\text{g}/\text{m}^3$
- SMPS, APS, DustTrak, P-Trak provide PM sizing (μm) and concentration ($\#/ \text{cc}$ and $\mu\text{g}/\text{m}^3$)

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System Validation

- Devices including T, RH, flow, particle sizing and concentration monitors, and balances were audited and performance was routinely verified
- Chamber aerosol distribution testing insured consistent dosing of all test animals
- Certified weights, standard and field blank filters validated balances daily



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System Validation

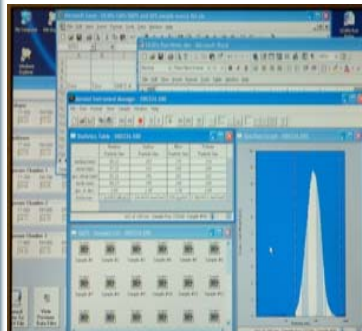


- Instrument checks recommended by manufacturers performed daily to verify zero and span
- Collocated filters and chemical analysis procedures validate PM instruments
- Concentrating equipment was extensively characterized by preliminary testing

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Electronic Records



- Electronic records developed where possible
- Notes and activities directly entered, recorded in files
- HUCAPs ADP controls allowed operators to monitor and adjust system parameters in real time to maintain optimal S_r and PM concentrating effects
- Process variables and chamber conditions recorded every 3 min.

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Electronic Records



- DASYS Lab process monitoring software tracked and recorded T, RH, flow, press, and PM concentrations at 24 locations
- 9 Aerosol instruments tracked and recorded chamber and inlet PM parameters every 3 min.
- All electronic files were backed up to multiple locations including network servers daily

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Redundancy

- Consistent exposures, reliable operations, minimal lost exposure time, equipment and process safeties, recoverable data
- Critical systems or measurements backed up with redundant equipment or alternative measurement techniques
- Examples include backup vacuum pumps, recoverable data such as chemical analysis of filters to backup balance measurements, DustTrak backup for filters, P-Trak backup for SMPS and APS
- Manual data entry as automatic data backup

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Exposure Results

- Completed 6 hr/d, 5 d/wk, 13wks of exposures with just 2 hrs of 1170 hrs scheduled exposure time lost
- UCAPs concentrations up to $5.0 \times e^5 \text{ \#}/\text{cc}$, $500 \text{ ug}/\text{m}^3$, $\sim 80 \text{ nm \# size}$, $\sim 170 \text{ nm mass size}$, Avg. Mass X factor $\sim 42.6 \text{ X}$
- CAPs concentrations up to $600 \text{ ug}/\text{m}^3$, $\sim 900 \text{ nm}$, Avg. Mass X factor $\sim 25.5 \text{ X}$
- Post exposure chemical analysis of ~ 1000 filter samples and health effects results pending

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Effective QC Practices

- Preliminary systems validation
- Survey of lab practices by QAM
- OPs backed up by detailed check lists completed daily
- Daily instrument QC checks
- Daily double checks of filter data sheets and activities entries
- Filter systems (sample port, holder, vacuum line, meter, valve, data sheet) color coded for location and sample ID

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Effective QC Practices

- Multiple alternative measurement techniques
- Daily file backups, multiple locations
- Ongoing routine systems maintenance based on system performance tracking
- Excellent staff communications and routine planning meetings/discussions
- Backup staffing coverage through cross training of staff in all critical areas and tasks

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Check lists



- Operations Status – allowed staff to track when various processes were completed so exposures could start
- Filter Sampling – insured filter samples were properly setup, and documentation completed
- Instrument Setup and QC checks – verified instrument performance daily prior to exposures
- Exposure Operations - ensured all aspects of OPs were followed, completed, and data were properly recorded and backed up

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Operations Status

CAPS/UCAPS DAILY EXPOSURE STATUS CHECK LIST

DATE: _____ RUN DAY # _____

	COMPLETE (Y)	TIME	INITIALS	COMMENTS
INSTR. DOWN LOAD				
BOILER FLUSH				
INSTRUMENTS				
FILTERS				
CHAMBERS				
ANIMALS LOADED				
EXPOSURE START				
EXPOSURE STOP				

NOTES:

Reviewed by: _____ Date: _____

Z:\UCAP\System info\ON Daily Exp. Status Cl List 08108.doc

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HUCAPs Boiler Service

Project/Order#071121veg222 Page 1 of 1

UCAPs/UCAPS Operations Check List (HUCAPs Boiler)

Date: _____ Test ID: _____

Operator: _____ Reviewed by: _____

HUCAPs - Boiler Check box or record value

CI Water System Flow gauge reading
 Open floor drain valve (set timer to >12 min)
 Flush drain tubing
 Close Boiler Feed Water valve (BFW) (DI supply loop)
 Open Boiler Drain Valve (BDV)
 Silence Boiler Low Water Alarm (Red Flt)
 Close BDV once drainage stops
 Disconnect and drain tubing
 Reconnect and close drain tubing
 Open BFW about 1/2oz (hearing water sound)
 Check CI Water Loop press. (if not pre-vented flow)
 Reset Boiler Low Water Alarm (Red Flt, F1)
 CI Water System Flow gauge reading

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Filter Samples

Project: Dashed071110aegggg Page 1 of 1

UCAP/CAFs Operations Check List (Filters)

Date: _____ Test ID: _____

Operator: _____ Reviewed by: _____

Filter Samples Check box or list value

Prior to System Startup

- Prepare Filter Data Sheets
- Preweigh 37mm Tuffon Filters
- Coat Containers with Toluene Carboxylic Methylol
- Load Filters into holders
- Mount holders and connect vacuum lines
- Record all initial OGM readings
- Verify all filter values are correct

After System Startup

- Start Filter Vacuum Pumps (# 4, 5)
- Open filter ON/OFF valves for active samples
- Adjust Filter Flows (Chambers = 10 Lpm, Inlet = 10 Lpm)
- Adjust Chamber Flows
 - UCAP's Ch = -02 Lpm total
 - CAFA's Ch = -02 Lpm total
 - CF Ch = -04 Lpm total
- Record initial Rotometer values on filter data sheet
- Readjust Chamber Flows
 - UCAP's Ch = -02 Lpm total
 - CAFA's Ch = -02 Lpm total
 - CF Ch = -04 Lpm total

Prior to Exposure Stop

- Record final Rotometer values on filter data sheets
- Close all Filter ON/OFF valves
- Adjust Chamber Flows
 - UCAP's Ch = -02 Lpm total
 - CAFA's Ch = -02 Lpm total
 - CF Ch = -04 Lpm total

Post Exposure

- Stop Filter vacuum pumps (# 4, 5)
- Record all OGM final readings
- Unmount filter holders
- Unmount filter from holders to petri dishes
- Store Mass Filters (37mm), desiccator in A-003
- Store Quartz Fibers (47 mm), Resistor
- Store Nitrate II (47 mm Tuffon, 47 mm nylonwork) Resistor
- Calculate sample volume on filter data sheet
- Update Filter Master Data file
- Update Study Master data file

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Instrument Operations

Project: Dashed071110aegggg Page 1 of 2

UCAP/CAFs Operations Check List (Instruments)

Date: _____ Test ID: _____

Operator: _____ Reviewed by: _____

Monitoring Instruments Check box or list value

Prior to System Startup

DustTrak

- DustTrak - verified time & date
- DustTrak - check flow (1.7 Lpm +/- 0.2)
- DustTrak - check zero (0.0)
- DustTrak - compare all using room air (offlocate) (<1.0 ug/m³)
- DustTrak - switch to Log Mode 1 and verify Log Interval = 3:00
- DustTrak - open sample line valve
- DustTrak - connect sample line

I-Trak

- I-Trak - install "charger" alcohol cell
- I-Trak - install monitor cell storage unit
- I-Trak - verified time & date
- I-Trak - check zero (0.0)
- I-Trak - compare all using room air (offlocate)
- I-Trak - switch to Log Mode 1 and verify Log Interval = 3:00
- I-Trak - open sample line valve
- I-Trak - connect sample line

DASTLab system monitor (LabAN PC)

- DASTLab software On, system monitoring workstation selected
- DASTLab software latest system monitoring worksheet selected
- DASTLab system monitoring started >25 min pre Exposure start
- DASTLab data WRITE started (> 25 min pre Exposure start)

SMPS

- SMPS - check balanced level in CPC (30 hrs), 48 if mg/L
- SMPS - vacuum pump exhaust line run to hood port
- Complete laptop PC on
- SMPS - Classifier and CPC USB lines mounted to PC
- SMPS - install resistor (checked, remove if break lead)
- SMPS - sample port open to room air
- SMPS - power stop ON (starts pump and CPC)
- SMPS - Classifier power ON
- SMPS - (breath air flow set to 0.3 lpm)
- SMPS - sample air flow adjusted to 0.3 Lpm (breathair bleed valve)
- SMPS - MM SMPS program started
- Flashline frame as VMADa
- Preprocessor (000, 001, 010, 02, 070 min, 0.0 g per 0.1)
- Schedule set for minimum of 9 hrs (180 samples)
- SMPS - start sample
- SMPS - stop sample, FileSave
- SMPS - sample room

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Exposure Operations

Project: Deter071110aegggg Page 1 of 4

UCAP/UCAP Operations Check List (Exposure Operations)

Date: _____ Test ID: _____

Operator: _____ Reviewed by: _____

Chamber Systems

Prior to Exposure Startup

System

Chambers (2) sanitized for animal exposures

Draw valves (2) closed or vented

LabLAN PC On (User: LAL/Watch; PW: check w/LR)

LabLAN PC Printer Log On (User: lal/Watch; PW: che w/LR)

HUGA/PC On

APC PC On

Draw and set HUGA/PC boiler

Prepare Filter Data Sheets (12)

Preweigh 37mm Filter Filters (6)

Coat Desiccant (5) with Sodium Carbonate (Monthly)

Load Filter into holders (18/12)

Mount holders (12) and connect vacuum lines (12)

Record all initial DOM readings (12)

Verify all filter valves (12) are closed

Plug any unused filter mounting ports

Chamber TRM sensors mounted (2)

Record Boiler Water Low alarm on HUGA/PC panel

Filters mounted/unused ports sealed

DAT/ILab systems monitoring started (25 min pre Exposure start)

DAT/ILab data WRITE started (2) + 25 min pre Exposure start

Chamber TRM sensors in place

Chambers sealed (2)

Power On to Vacuum Pump 1 (AP1)

UCAP/PC Flow -50 Lpm (50-65 Lpm, each flow + all samples)

UCAP/PC Press -14" H2O (130-117) (if +17" service SSC)

CA/PC Flow -50 Lpm (48-62 Lpm, each flow + all samples)

CA/PC Press -17" H2O (150-147) (if +17" service SSC)

CE/PC Flow -54 Lpm (48-60 Lpm, each flow + all samples)

CE/PC Press -12" H2O (110-117)

Complete Instrument Monitoring Checklist

Open instrument sample port valves

Connect instrument sample lines

Close valves for any unused ports

Record Chamber setup and planned operation in Run Notes

Filters

Complete Filter Sample Checklist

Mount Filter holders in Chamber

Close Filter sample valves

Plug any unused filter mounting ports

Record Dry Gas Meter (DGM) initial readings on Filter Data Sheets

Once chambers are On record initial filter flows on Data sheets

Post exposure, record final DOM readings on Filter Data Sheets

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Chamber Systems

Project: Deter071110aegggg Page 1 of 3

UCAP/UCAP Operations Check List (Chamber Systems)

Date: _____ Test ID: _____

Operator: _____ Reviewed by: _____

Chamber Systems

Prior to Exposure Startup

System

LabLAN PC On (User: LAL/Watch; PW: check w/LR)

LabLAN PC Printer Log On (User: lal/Watch; PW: che w/LR)

DAT/ILab software On, system monitoring unhooked/selected

DAT/ILab software, safety system monitoring unhooked/selected

DAT/ILab systems monitoring started (25 min pre Exposure start)

DAT/ILab data WRITE started (2) + 25 min pre Exposure start

Filters

Complete Filter Sample Checklist

Mount Filter holders in Chamber

Close Filter sample valves

Plug any unused filter mounting ports

Record Dry Gas Meter (DGM) initial readings on Filter Data Sheets

Once chambers are On record initial filter flows on Data sheets

Post exposure, record final DOM readings on Filter Data Sheets

Instruments

Complete Instrument Monitoring Checklist

Open instrument sample port valves

Connect instrument sample lines

Close valves for any unused ports

Mount Chamber TRM sensor

Verify that HUGA/PC is monitoring Chamber TRM Press

Verify that DAT/ILab is monitoring/reading data

Prior to exposure start initiate Data Logging on all instruments

Post exposure, shut down, ball Data Logging on all instruments

Post exposure, halt DAT/ILab data recording (25 min post Exp)

Post exposure, copy DAT/ILab Data to WORD document

Down load/verify all instrument data files, transfer to LabLAN PC

Upload all run files to LabLAN share drive

Down load run files to Office PC for analysis and storage

Chambers

UCAP/PC Chamber

Chamber sanitized for animal exposures

Draw valve closed or vented

Filters mounted/unused ports sealed

Chamber TRM sensors in place

Power On to Vacuum Pump 1 (AP1)

Exhaust Flow set to -50 Lpm (50-65 Lpm, each flow + all samples)

Chamber Press -14" H2O (130-117) (if +17" service SSC)

Record Chamber setup and planned operation in Run Notes

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Filter Sample Data Form

Project: dsh0701112cap83cap Page: _____

UCAP/CAPs Filter Data Form

Date: _____ Exposure # _____ Project: dsh0701112cap83cap

Investigator: Dubois/Gilman Operator: JW

Filter No.: _____ Sample Location: INLET MASS

Flow Meter On: _____

Flow Meter On/Off: 2000

Start Time: _____

End Time: _____

Total Sample Time: _____ min. Name: _____

Volume Sampled: _____ L

Inlet Flow: _____ L/min Yr _____ "Hg

Outlet Flow: _____ L/min Yr _____ "Hg

Filter Weight:

	Pre-Weight	Asy (nd)	Post-Weight	Asy (nd)	Sample Mass (mg)
Date					
Time					
Disrupted					
Y/N					
Weight (mg)					
Vol. (L)					
Flow					
Inlet (L/min)					
Outlet (L/min)					
Yr					
Yr					

Name: _____

Read and Understood: _____ Date: _____

© UCAP/UCAPs CAPs Filter Data Form 12/08/07/08/09/10