

# ACTIVE SAMPLING FOR AIR CONTAMINANTS AS GAS, VAPOR, DUST, FUME, MIST



# **FIRST STEP:**

## **SELECT THE SAMPLING & ANALYTICAL METHOD**

U.S. GOVERNMENT METHOD MANUALS  
for sampling in the workplace:

- National Institute for Occupational Safety and Health (NIOSH)
- Occupational Safety and Health Administration (OSHA)



# WEBSITES

## FOR GOV'T AGENCY METHODS

- **NIOSH Methods**

<http://www.cdc.gov/niosh/nmam/>

- **OSHA Methods**

<http://www.osha.gov/dts/sltc/methods/toc.html>



# NIOSH METHOD

## SILICA, CRYSTALLINE, by XRD (filter redeposition)

7500

SiO<sub>2</sub> MW: 60.08 CAS: 14808-60-7 (quartz) RTECS: VV7330000 (quartz)  
 14464-46-1 (cristobalite) VV7325000 (cristobalite)  
 15468-32-3 (tridymite) VV7335000 (tridymite)

METHOD: 7500, Issue 4

EVALUATION: FULL

Issue 1: 15 August 1990

Issue 4: 15 March 2003

OSHA: quartz (respirable) 10 mg/m<sup>3</sup> / (% SiO<sub>2</sub>+2);  
 cristobalite and tridymite (respirable) 1/2 the above

NIOSH: 0.05 mg/m<sup>3</sup>; carcinogen

ACGIH: quartz (respirable) 0.1 mg/m<sup>3</sup>  
 cristobalite (respirable) 0.05 mg/m<sup>3</sup>  
 tridymite (respirable) 0.05 mg/m<sup>3</sup>

PROPERTIES: solid; d 2.65 g/cm<sup>3</sup> @ 0 °C; crystalline  
 transformations: quartz to tridymite  
 @ 867 °C; tridymite to cristobalite  
 @ 1470 °C; α-quartz to β-quartz  
 @ 573 °C

SYNONYMS: free crystalline silica; silicon dioxide

SAMPLING		MEASUREMENT	
<b>SAMPLER:</b>	CYCLONE + FILTER (10-mm nylon cyclone, Higgins-Dewell (HD) cyclone, or aluminum cyclone + 5-µm PVC membrane) *see sampling section	<b>TECHNIQUE:</b>	X-RAY POWDER DIFFRACTION
<b>FLOW RATE:</b>	Nylon cyclone: 1.7 L/min; HD cyclone: 2.2 L/min; aluminum cyclone: 2.5 L/min	<b>ANALYTE:</b>	Crystalline SiO <sub>2</sub>
<b>VOL-MIN:</b>	400 L	<b>ASH:</b>	Muffle furnace or RF plasma asher or dissolve in tetrahydrofuran
<b>-MAX:</b>	1000 L	<b>REDEPOSIT:</b>	On 0.45-µm Ag membrane filter
<b>SHIPMENT:</b>	Routine	<b>XRD:</b>	Cu target X-ray tube, graphite monochromator Optimize for intensity; 1° slit Slow step scan, 0.02°/10 sec Integrated intensity with background subtraction
<b>SAMPLE STABILITY:</b>	Stable	<b>CALIBRATION:</b>	NIST SRM 1878a quartz, NIST SRM 1879a cristobalite, USGS 210-75-0043 tridymite suspensions in 2-propanol.
<b>BLANKS:</b>	2 to 10 per set (see step 13.g)	<b>RANGE:</b>	0.02 to 2 mg SiO <sub>2</sub> per sample [2]
<b>BULK SAMPLE:</b>	High-volume or settled dust to identify interferences	<b>ESTIMATED LOD:</b>	0.005 mg SiO <sub>2</sub> per sample [2]
<b>ACCURACY</b>		<b>PRECISION (S<sub>x</sub>):</b>	0.08 @ 0.05 to 0.2 mg per sample [1]
<b>RANGE STUDIED:</b>	25 to 2500 µg/m <sup>3</sup> [1] (800-L sample)		
<b>BIAS:</b>	None known		
<b>OVERALL PRECISION (S<sub>x</sub>):</b>	0.09 (50 to 200 µg) [1]		
<b>ACCURACY:</b>	± 18%		

**APPLICABILITY:** The working range is 0.025 to 2.5 mg/m<sup>3</sup> for an 800-L air sample.

**INTERFERENCES:** Micas, potash, feldspars, zircon, graphite, and aluminosilicates. See APPENDIX.

**REAGENTS:**

1. Silica Standards.
  - a. Quartz\* (SRMs 1878a, 2950, 2951, 2958) and Cristobalite\* (SRMs 1879a, 2960, 2957), available from Standard Reference Materials Program, Rm. 204, Bldg. 202, National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899; [www.nist.gov](http://www.nist.gov).
  - b. Tridymite\* (210-75-0043) available from U.S. Geological Survey, Box 25046, MS 973, Denver, CO 80225.
2. 2-Propanol\*, reagent grade.
3. Desiccant.
4. Glue or tape for securing Ag filters to XRD holders.
5. Optional: tetrahydrofuran (THF)\* (if LTA or muffle furnace are unavailable).
6. 1.5 % parlodion solution. (Dissolve 1.5 g of parlodion\* in isopentyl acetate\* and dilute to 100 mL with isopentyl acetate.)
7. Optional (if calcite present): 25% v/v concentrated hydrochloric acid\* (ACS reagent grade) in distilled water and 25-mm filters of PVC or cellulose ester with pore size of 1  $\mu\text{m}$  or less.

\* See SPECIAL PRECAUTIONS.

**EQUIPMENT:**

1. Sampler:
  - a. Filter: Polyvinyl chloride (PVC) filter, 37-mm, 5.0- $\mu\text{m}$  pore size supported with backup pad in a two-piece, 37-mm cassette filter holder (preferably, conductive) held together by tape or cellulose shrink band.  
NOTE: Check each new lot of PVC filters by analyzing one or more by this method. For example, Gelman VM-1 filters (all lots) were found to be unacceptable because of high ash and background. If THF is used, check for complete dissolution by dissolving a blank PVC filter and following steps 5c through 8.
  - b. Cyclone: 10-mm nylon, Higgins-Dewell (HD), Aluminum (Al), or equivalent [11].
2. Area air sampler: PVC membrane filter, 37-mm diameter, 5- $\mu\text{m}$  pore size; three-piece filter cassette.
3. Sampling pumps with flexible connecting tubing, capable of the following flow rates: nylon cyclone, 1.7 L/min; HD cyclone, 2.2 L/min; Al cyclone, 2.5 L/min; and bulk sampler, 3 L/min.
4. Silver membrane filters, 25-mm diameter, 0.45- $\mu\text{m}$  pore size, available from Sterlitech Corp., 22027 70<sup>th</sup> Ave S, Kent, WA 98032-1911; [www.sterlitech.com](http://www.sterlitech.com).
5. X-ray powder diffractometer (XRD) equipped with copper target X-ray tube, graphite monochromator, and scintillation detector.
6. Reference specimen (mica, Arkansas stone, or other stable standard) for data normalization.
7. Low-temperature radio-frequency plasma asher (LTA) or muffle furnace, or ultrasonic





# OSHA Method

Crystalline Silica  
Quartz and Cristobalite



Method number:	ID-142
Version:	4.0
Target concentration: (for quartz and cristobalite)	50 $\mu\text{g}/\text{m}^3$
OSHA PEL:	50 $\mu\text{g}/\text{m}^3$ (combined polymorphs) 25 $\mu\text{g}/\text{m}^3$ action level

ACGIH TLV:  
(respirable fraction)  
( $\alpha$ -quartz and cristobalite)

0.025  $\text{mg}/\text{m}^3$

Procedure:



Samples are collected by drawing workplace air through pre-weighed 5- $\mu\text{m}$  pore size, 37-mm diameter low ash polyvinyl chloride (PVC) filters preceded by 10-mm nylon Dorr Oliver cyclones. The weight of the respirable dust is determined by gravimetric analysis. The PVC filters are dissolved and the samples are suspended in tetrahydrofuran (THF). The samples are then deposited on silver membranes and analyzed by X-ray diffraction (XRD).

Recommended sampling time  
and sampling rate:

480 min at 1.7 L/min (816 L)

Reliable quantitation limit:

9.76  $\mu\text{g}/\text{sample}$  (12.0  $\mu\text{g}/\text{m}^3$ ) quartz  
20.6  $\mu\text{g}/\text{sample}$  (25.2  $\mu\text{g}/\text{m}^3$ ) cristobalite

Standard error of estimate  
at the target concentration:

8.2% quartz  
9.6% cristobalite

Status of method:

Fully validated method.

# HELP FROM SKC

## SAMPLING GUIDE

- Lists all regulated chemicals
- Includes:
  - Agency Method Number
  - Sampling Details such as Flow Rate, Time, Volume
  - Analytical method
  - Sample collection media and SKC catalog number



**Located in the back of the catalog and on the SKC website.**



# **FOR EXAMPLE:**

## **LET'S LOOK AT BENZENE SAMPLING**

- Find benzene in the SKC Sampling Guide
- Look at the options for OSHA methods along with the sampling details.
- OSHA Method 1005 has a passive sampler (PS) option and two sorbent tube (ST) options with the sampling details given including flowrate, air volume, and SKC collecting equipment.





# ARE YOU REQUIRED TO USE NIOSH OR OSHA METHODS?

- U.S. OSHA does NOT mandate the sampling method that employers must use.
- The employer has the obligation of using a method that meets the requirements:  $\pm 25\%$  of the true value at a 95% confidence level.
- But OSHA and NIOSH methods are normally used for compliance.



# ACTIVE SAMPLING DEFINED

The collection of airborne contaminants using a mechanical device such as a pump to draw the air/contaminant mixture *into or through* the sampling device such as a sorbent tube, filter, impinger, or sample bag.



# TYPES OF SAMPLES FOR VARIOUS EXPOSURE PERIODS

- **Integrated samples**- Contaminants are collected and **concentrated** over a period of time to obtain average exposure levels during the entire sampling period
- **Grab samples**- Contaminants are collected into a device over a short interval of a few seconds or minutes to represent exposures **at a given point in time.**



# THREE KEY COMPONENTS FOR ALL ACTIVE SAMPLING

- Sampling pump

To pull or push air

- Sample Collection Media

Something to pull or push the air through or into for analysis

- Calibrator (flowmeter)

Something to indicate how much air has been pulled or pushed through the collection media.



# FIRST KEY COMPONENT

## SAMPLING PUMP



# CHOOSING A PUMP

## FLOWRATE OPTIONS

- **GASES AND VAPORS** are sampled at low flow rates (ml/min) to allow effective adsorption to occur onto the sorbent material.
- **PARTICULATES** are sampled at higher flow rates (L/min) so that airborne particles can be effectively captured from the air and drawn onto the filter material.





# LOW FLOW PUMP



**Pocket Pump Touch:**  
Flow range of  
20-500 ml/min

- Smaller power requirements allow for a smaller battery (and smaller pump).
- Primary application is sorbent tube sampling for gases/vapors.

# HIGHER FLOW PUMP WITH LOW FLOW CAPABILITIES



Flow range  
from 5 L/min  
down to  
5 ml/min using  
the low flow  
tube holder.

**AirChek TOUCH**



# SAMPLING PUMPS: FEATURES TO CONSIDER

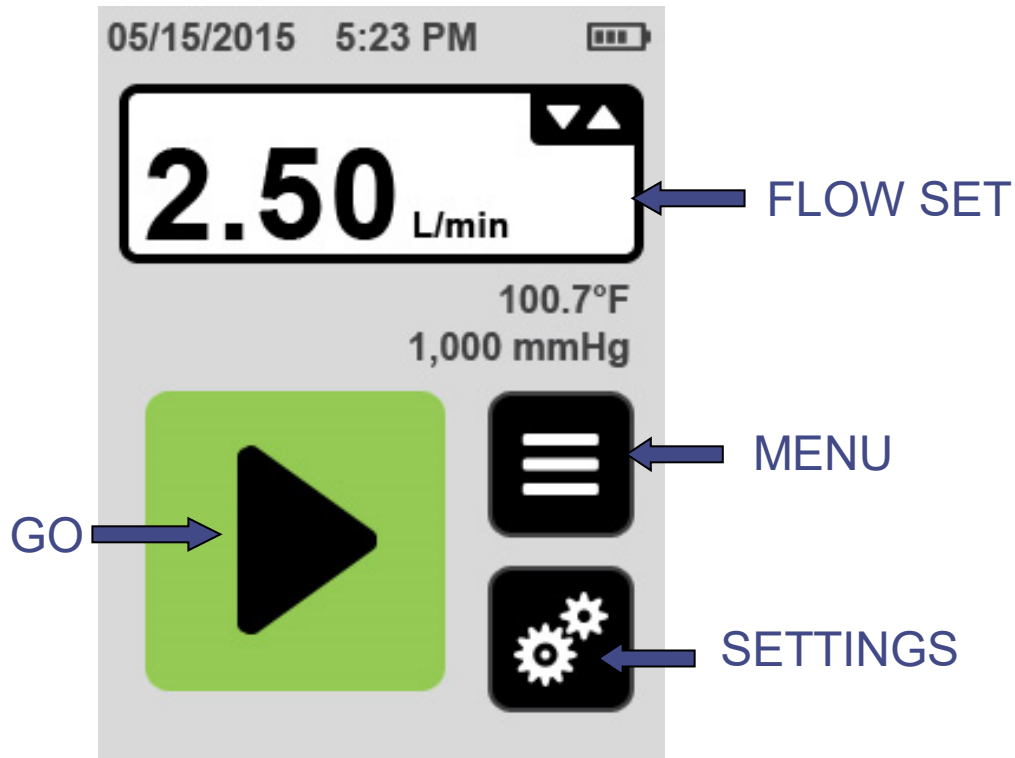
- **Constant Flow**- Is there an internal mechanism to compensate for restrictions to flow?
- **Electromagnetic Susceptibility**- Is there shielding from RFI/EMI?
- **Backpressure**- What is the maximum pressure drop that the pump can handle?



# NEW OPTION FROM SKC

## TOUCHSCREEN NAVIGATION

### HOME SCREEN



**AirChek TOUCH**



# PUMP PROGRAMMABILITY USING PC AND PUMP SOFTWARE

ADVANTAGE: SKC  
Datatrac<sup>®</sup> software  
provides enhanced  
capabilities for pump  
scheduling  
and record keeping.



# SKC DATATRAC SOFTWARE

## BASIC FUNCTIONALITY

### **Document:**

- Sample ID number
- Method/Media
- Calibration Data
- Environmental Conditions
- Analysis Results

### **Program:**

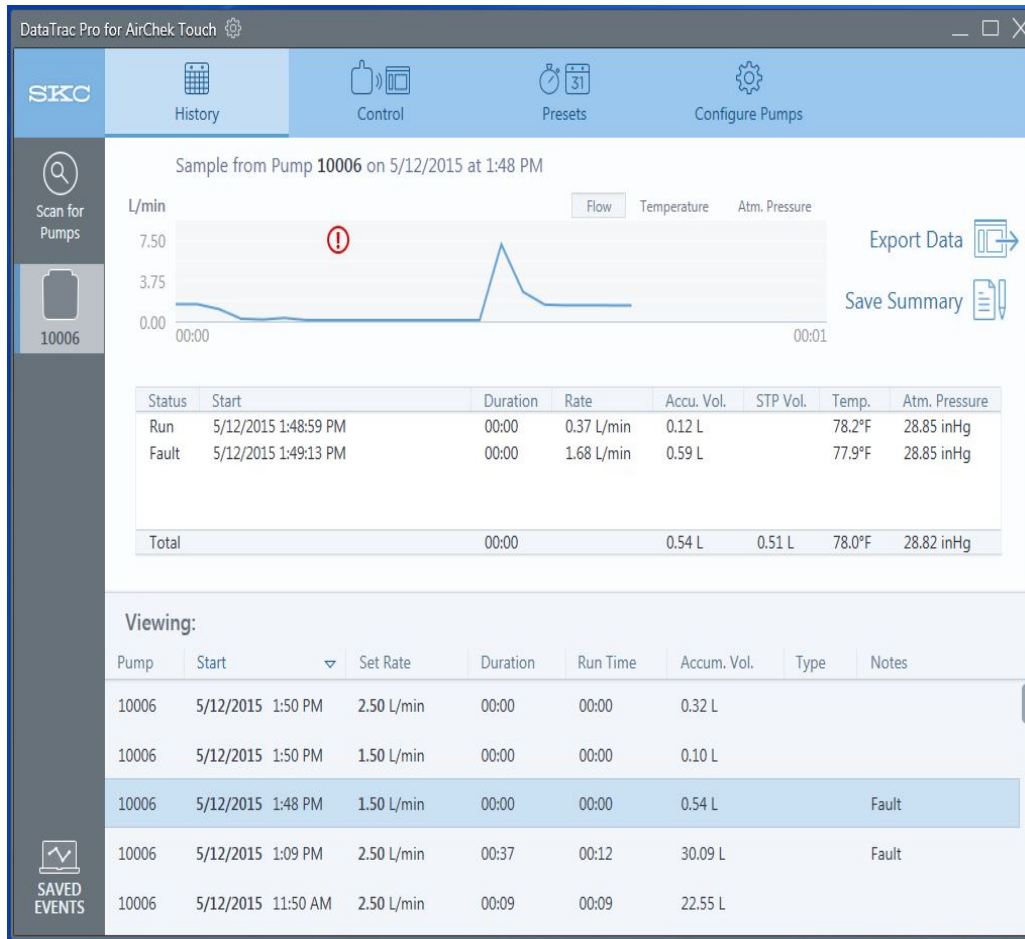
- Start/stop date, time, flowrates
- Complex sampling schedule with multiple start/stop times





# SKC DATATRAC SOFTWARE

## SAMPLE HISTORY WINDOW



Record, view, save, or print:

- Run Time
- Flow Rate
- Air Volume
- Changes in operational status



# LATEST PUMP DEVELOPMENT

## BLUETOOTH APPS



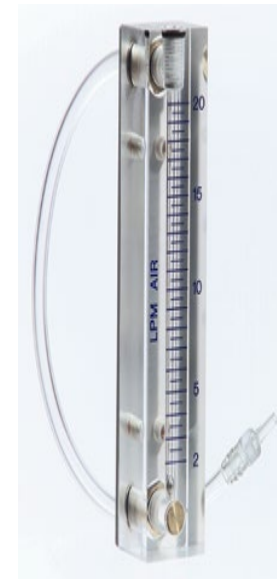
SKC SMART WAVE FOR POCKET  
PUMP TOUCH

allows users to:

- Check pump status
- Adjust flow
- Start/stop the pump

USING A MOBILE DEVICE.

# SECOND KEY COMPONENT CALIBRATOR (FLOWMETER)



# PUMP CALIBRATION

## WHAT DOES IT MEAN?



- Pump calibration does **not** typically refer to an annual service by the factory.
- Calibration is done by the user to set and verify the flowrate of the pump to that required in the sampling method.
- Should be done before and after every sample.
- The average of the pre- and post- sample flowrates is used to calculate the air volume.



# **PUMP CALIBRATION**

## **USE AND APPLICATION**

Accurate pump calibration is essential:

- SO YOU can calculate total air volume for the sample and report this to the laboratory when you submit your samples to them.
- SO THE LABORATORY can use the total air volume provided to calculate airborne concentration levels.



# HERE'S HOW THE PUMP CALIBRATION DATA IS USED

- The pump flow rate measured by your pump calibrator is multiplied by the total sampling time.  
**Flow rate (L/min) X Time (min)=Air Volume (L)**
- This air volume is provided by you to the laboratory with your sample collection media.
- The lab will then measure the amount of contaminant in mg or  $\mu\text{g}$  on the sample collection media and will use the air volume supplied to calculate concentration of the contaminant in air.





# CALIBRATOR CLASSIFICATIONS

## PRIMARY STANDARDS

Historically, the IH profession has defined calibrators as PRIMARY STANDARDS if they involve the movement of a bubble or piston in a chamber of a known fixed volume.

### **Examples include:**

- Soap bubble meters/Film flowmeters
- Electronic bubble meters (Buck, Gilibrator)
- Electronic piston meters (Defender)

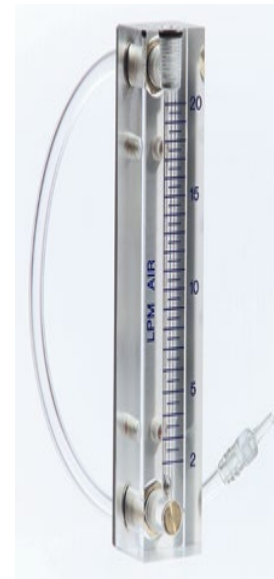


# CALIBRATOR CLASSIFICATIONS

## SECONDARY STANDARDS

- Calibrators that must be recalibrated by a certified calibrator on a frequent basis are called secondary standards.

Rotameters are an example of a secondary standard.



# CALIBRATOR CLASSIFICATIONS

## INTERMEDIATE STANDARDS

- Calibrators that have the accuracy of a primary standard, but are not based on the movement of a bubble/piston in a chamber of a known fixed volume.
- The SKC ChekMate is an intermediate standard.



# A SIGNIFICANT DEVELOPMENT FOR FLOWMETERS

- It is now best practice to have flowmeters recalibrated by an ISO 17025 calibration lab annually.
- Flowmeters are calibrated to a NIST-traceable standard to ensure the stated accuracy.



# SOAP FILM FLOWMETERS: OLD TECHNOLOGY

- Pump pulls air through a glass tube with volumetric indications.
- A liquid film-*a soap bubble*-is placed into the flow path from the squeeze bulb or other container.
- The air flow causes the bubble to move from one volume mark to another.
- The travel time is measured with a stopwatch
- By knowing the travel time and the tube volume, the flow rate can be calculated.



# FLOWRATE CALCULATION

Question: If it took 128 seconds for the bubble to travel a 500 ml volume, what is the flowrate?

$$\frac{500 \text{ ml}}{128 \text{ sec}} \times \frac{60 \text{ sec}}{\text{min}} = 234.4 \text{ ml/min}$$





# ELECTRONIC BUBBLE METERS

- Work on the same principle as a manual soap film flowmeter, but infrared sensors electronically time the bubble
- A microprocessor instantly calculates flow rate and displays it digitally.
- Examples include Buck Calibrator and Gilibrator.



# NEAR-FRICTIONLESS PISTON METERS

- The flow of the pump causes a piston inside a chamber to rise and fall.
- Photo-optic sensors sense the rise and fall of the piston.
- An electronic timer combine with a microprocessor to automatically measure travel time and calculate the flowrate.
- Examples include Dry-Cal flowmeters such as the Defender.



# NEW CALIBRATOR OPTIONS

## SMALL, FIELD FRIENDLY, ACCURATE

- SKC CHEKMATE



Accuracy is +/- 1% of reading.

- TSI 4046/4146



Accuracy is +/- 2% of reading.



# SKC CHEKMATE CALIBRATOR



- Uses a differential pressure sensor
- Adjusts for changes in temperature and atmospheric pressure
- Displays a moving average of the flowrates
- Certified to NIST national standard in U.S.; UKAS in U.K.

# DIRECT COMMUNICATION TO PUMP CALIBRATORS



SKC CalChek® feature allows CalChek enabled SKC pumps to communicate directly with the SKC Chek-mate Calibrator for hands-free adjustment and verification of flow rate.



# TECH TIPS ON CALIBRATION

- It is a best practice to let your pumps run 5 minutes before calibration after removing them from the battery charger to let the flow stabilize.
- The pump must be calibrated with representative sample media in line. Use a clean set of media to collect the sample in the field after calibration.



# TECH TIPS ON CALIBRATION

- It is not necessary for the flowrate to be the exact flow specified in the method. Typically, the flowrate should be within 5% of that specified. Just be sure you know exactly what it is.
- Use the running average or take at least 3 flow measurements that agree within 5% and use the average of the readings as your flow rate measurement.
- If pre-and post-averages differ by more than 5%, your sample is called into question.





# TECH TIP IN CALIBRATION SET-UP



- Set up the pump and sample media like its ready to place on the worker.
- Then attach the calibrator to the open end (inlet) of the sample media.

# TECH TIP IN CALIBRATION SET-UP

- If the inlet of the sampler can not be attached directly to the calibrator using connecting tubing, **calibration adapters** may be available for some devices.



# TECH TIP IN CALIBRATION SET-UP

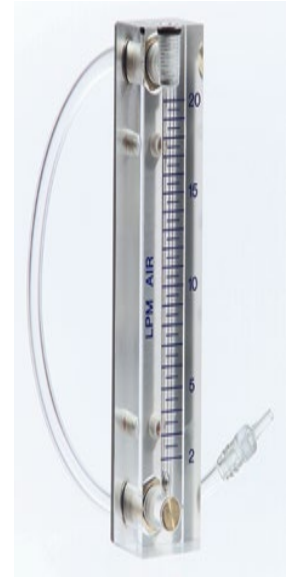
- If the sampler can not be attached directly and it has no calibration adapter, use a **calibration jar** of a size to fit the sampler.

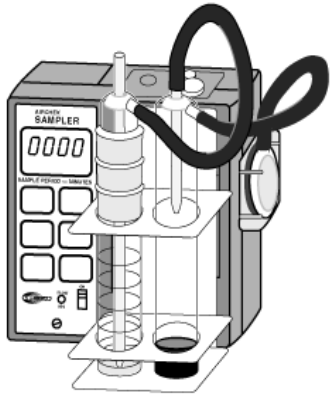


# ROTAMETERS

## HANDY IN THE FIELD

- Air passes through a vertical tube with a ball or float inside
- The flow of air upward through the tube causes the ball to stabilize at a certain point
- Affected by temperature and atm pressure
- So calibrate the rotameter using a certified calibrator under conditions of use.





# **SAMPLING MEDIA:** **ACTIVE SAMPLING OF** **GASES AND VAPORS**



# DEFINING GASES AND VAPORS

- A substance is considered a **GAS** if this is its normal physical state at room temp (25° C) and one atm. (760 mm Hg) pressure (Example: Carbon monoxide)
- If the substance is a liquid at normal temp and pressure, then the gaseous component in equilibrium with its liquid (or solid) state is called a **VAPOR**. (Example: Benzene)





# SOLID SORBENTS

## INTRODUCTION

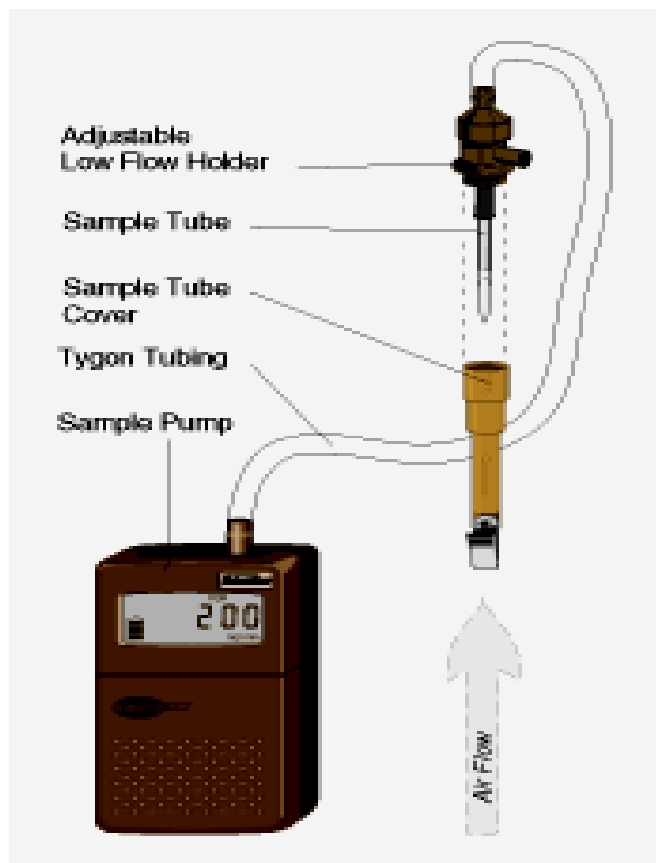
- Most widely used sample media for gases/vapors
- Consist of small granules or beads
- Adsorb the contaminant onto the surface
- Packed into tubes to collect various amounts and types of chemicals for lab analysis



Not the same as colorimetric detector tubes.



# SAMPLE COLLECTION WITH SORBENT TUBES



Most tubes have 2 sorbent layers:

- Larger section serves as the primary collection layer
- Smaller section is the back-up layer

The arrow on the tube indicates the direction of air flow.

# MORE ON SORBENT LAYERS

- Both sorbent layers are analyzed by the lab and used to calculate exposure levels.
- Breakthrough (sample loss) is indicated when contaminant levels on the backup layer are  $\geq 25\%$  of the levels found on front sorbent layer.
- Samples are not valid when breakthrough occurs.



# POSSIBLE CAUSES OF SAMPLE BREAKTHROUGH

- High concentrations of target compound
- High concentrations of similar compounds
- High humidity
- Tube is reversed in the sampling train so the smaller section is exposed to the air first

NOTE: When a high concentration is found on the backup layer, there is a concern that some chemicals escaped completely from the tube.



# HOW DO YOU KNOW WHICH SORBENT TUBE TO USE?

NIOSH or OSHA methods specify the tube to use.

- SKC captures this information and provides the catalog number in the SKC sampling guide.
- The laboratory can also advise on the sorbent tube to use.



# SORBENT TUBES

## FOR PPM LEVEL MEASUREMENTS

Sorbent tubes specified for workplace compliance sampling in the ppm range are designed for solvent extraction by the lab. Simply, break off the tips of the glass tube and attach to the pump for sample collection.



Make sure  
to use a  
tube  
breaker.



# **SORBENT TUBES**

## **FOR PPM LEVEL MEASUREMENTS**

- After sampling, seal the glass tubes with caps provided and ship to the lab.
- The lab will break open the tube, pour the sorbent into vials, add a liquid solvent, and shake for a while so that the solvent extracts the contaminant from the solid adsorbent and into the liquid for GC analysis.



# SORBENT TUBES

## FOR PPB LEVEL MEASUREMENTS

- Sorbent tubes specified for low level sampling in the ppb range are designed for thermal desorption by the lab.
- These specialty tubes are available in glass or stainless steel.





# **SORBENT TUBES**

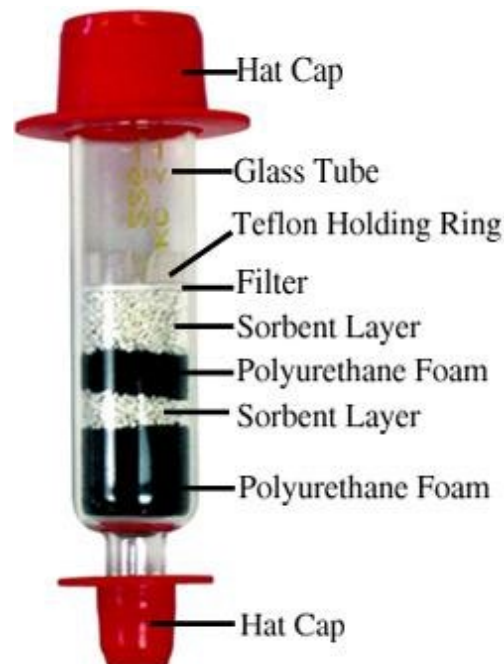
## **FOR PPB LEVEL MEASUREMENTS**

- The lab will place the entire tube into the thermal desorber and apply heat and an inert gas to drive contaminant molecules from the adsorbent material to the detector of the GC.
- Provides lower detection limits for environmental or indoor air sampling.



# SPECIALTY SORBENT TUBES

- Both U.S. NIOSH and OSHA have developed methods using the OSHA Versatile Sampler (OVS).
- Contains both sorbent and an internal filter
- Agency methods available for pesticides, explosives, glycols, and more.



**226-30-16** with  
XAD-2  
and glass fiber  
filter

**226-58**  
with Tenax  
and quartz filter

**226-57**  
with XAD-7  
and glass fiber  
filter

# SPECIALTY SORBENT TUBES



## PUF Cartridges

Used for semi-volatiles  
including PCBs and pesticides  
by ASTM or EPA methods.

Not specified in any  
NIOSH/OSHA methods.



# TYPES OF SORBENT MATERIAL

## CARBON BASED SORBENTS

- **Activated charcoal**- Most widely used solid sorbent suitable for collection of *non-polar* organic compounds including benzene, toluene, and xylene.
- **Anasorb<sup>®</sup> 747**- Beaded carbon material that can collect a variety of both *nonpolar and polar* organic compounds.
- **Carbotrap<sup>®</sup> and Carbosieve<sup>®</sup>**- High surface area; useful for very volatile compounds.



# TYPES OF SORBENT MATERIAL

## INORGANIC SORBENTS

**Silica gel**- Used to collect *polar* organic compounds such as alcohols, amines, and phenols.

Side Note: Electronic products are often packaged with a silica gel pouch as a desiccant since it readily adsorbs water vapor.



# TYPES OF SORBENT MATERIAL

## ORGANIC POLYMERS

Poropaks<sup>®</sup>, Chromosorbs<sup>®</sup>, XAD resins, Tenax<sup>®</sup>

- These sorbents are used to collect a variety of specialty compounds. Chromosorbs and XAD-2 are specified in NIOSH and OSHA methods for pesticides.
- Tenax is specified in EPA methods for (sub) ppb level VOC sampling with specialized analysis.



# TYPES OF SORBENT MATERIAL

## SPECIAL PREPARATION

Some chemicals are not effectively trapped by any solid sorbent without special preparation:

- **Extensive washing** is done of silica gel to trap inorganic acids such as hydrochloric or hydrofluoric.
- **Chemical coating** is done on various sorbents to trap aldehydes, nitrogen dioxide, amines, and many more compounds.





# **SORBENT TUBE**

## **SAMPLING ADVANTAGES**

- Sample is integrated over the entire period of exposure.
- Published methods exist with extensive testing and documentation of reliability.
- Workplace compliance officers typically use this sampling method.
- Air flow is calibrated and measured.
- Backup layer indicates sample breakthrough.



# **SORBENT TUBE**

## **SAMPLING ADVANTAGES**

- Known contaminants can be quantified easily and economically using tubes with a single type of a specific sorbent.
- Unknown contaminant mixtures can be identified and quantified using specialty thermal desorption tubes.
- Multi-phase contaminants can be collected using sorbent/filter combination tubes.



# IMPINGERS

## INTRODUCTION

Impingers are specially designed glass bottles that are filled with a collection liquid specified in the sampling and analytical method.



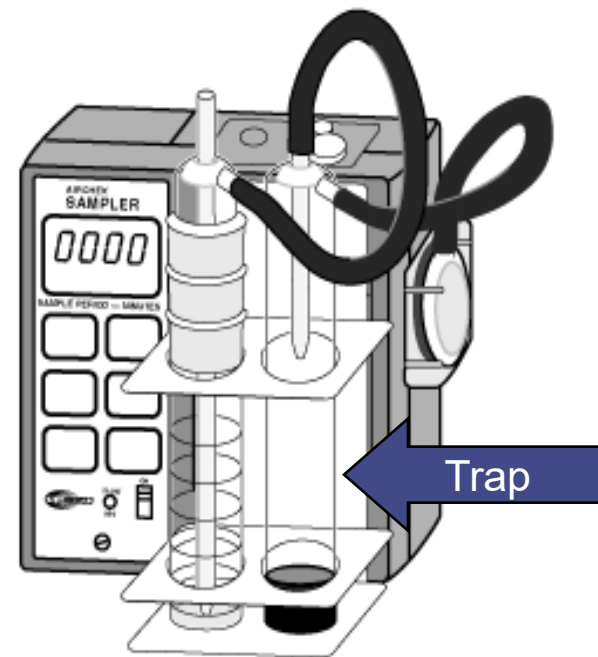
# NOTES ABOUT IMPINGERS

In some cases, the impinger nozzle is **fritted** or modified with thousands of small holes. This disperses the air and allows for better contact between the air sample and the impinger liquid.



# SAMPLE COLLECTION WITH IMPINGERS

A sample pump is used to bubble air through the impinger which contains a liquid reagent that has been specified in the method. The liquid will physically dissolve or chemically react with the chemical of interest.



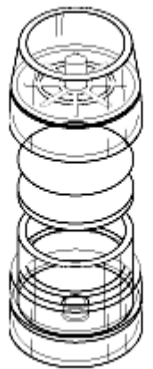
Better for area sampling



# CHEMICALLY TREATED FILTERS

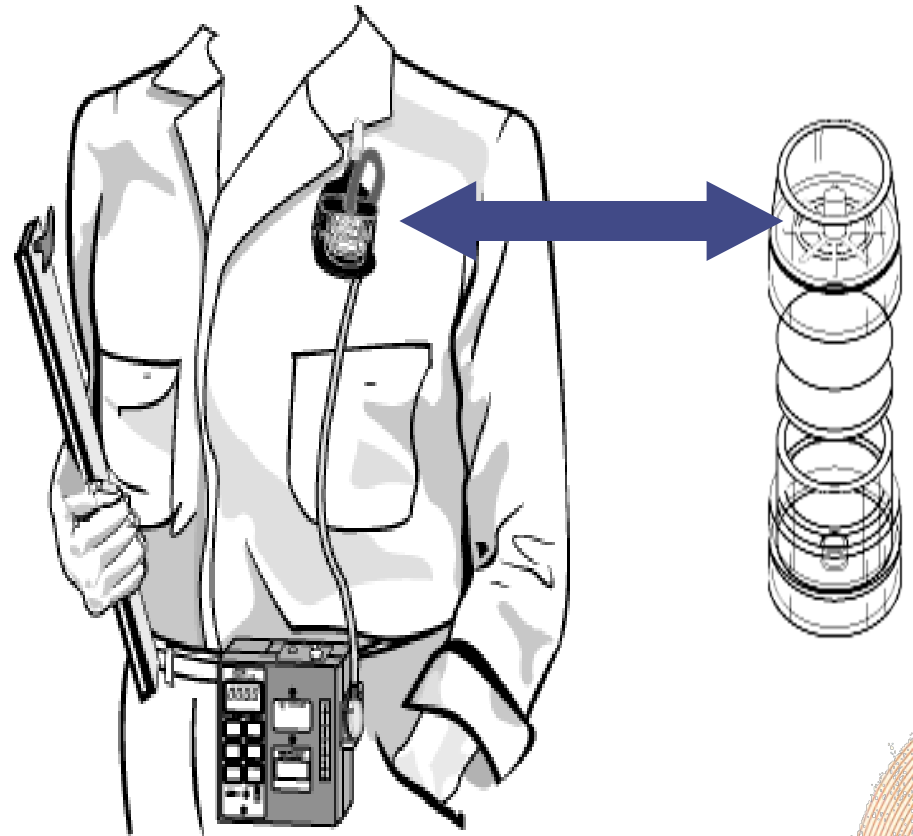
## INTRODUCTION

- Specified in some newer methods as an alternative to older impinger methods
- Filters are used as a substrate for liquid media that can trap contaminants.
- Liquid media will chemically react with the contaminant of interest.
- Result is a stable compound for storage and analysis.



# SAMPLE COLLECTION WITH COATED FILTERS

SKC provides chemically coated filters for NIOSH/OSHA methods assembled in cassettes and ready for use.





# COATED FILTERS

## COMMONLY USED TYPES

### Chemically Coated Glass Fiber Filters

- 2,4 DNPH
- 1-(2-pyridyl)  
piperazine
- Sulfuric Acid
- Mercuric Acetate



### To Collect Target Compounds

- Glutaraldehyde
- Diisocyanates  
(HDI,MDI,TDI)
- Aromatic Amines
- Mercaptans



# COATED FILTERS

## ADVANTAGES

- Sample is integrated over the entire period of exposure.
- Published methods exist with extensive testing and documentation.
- Workplace compliance officers use these methods for some compounds.
- Air flow is calibrated and measured.
- Front and back filter can be used in one cassette to determine breakthrough.



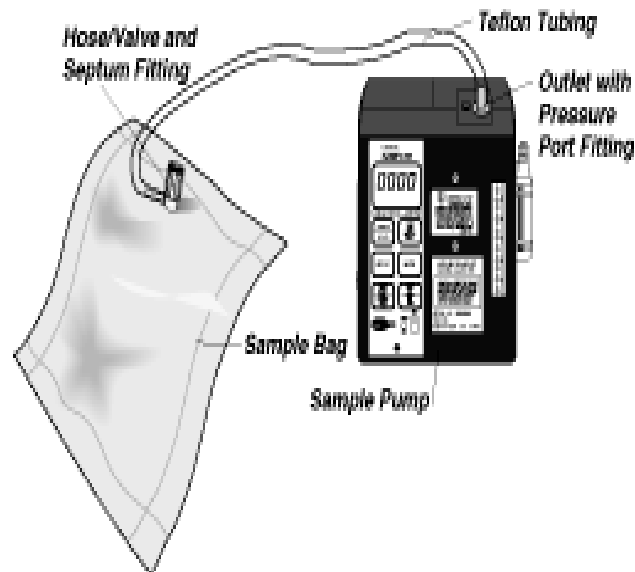
# SAMPLE BAGS

## INTRODUCTION

- Used since the 1950's to collect a fixed volume of an air-contaminant mixture into a flexible container for subsequent analysis.
- Called *grab* samples in industrial hygiene and *whole air* samples in environmental applications.



# SAMPLE COLLECTION WITH SAMPLE BAGS



**Positive Pressure**



**Negative Pressure**



# SAMPLE BAGS

## SPECIFIED IN SAMPLING METHODS

- Benzene in NIOSH 3700
- Carbon Dioxide in OSHA ID 172
- Carbon Monoxide in OSHA ID 210
- Ethylene Oxide in NIOSH 3702
- Nitrous Oxide by NIOSH 6000
- Sulfur hexafluoride in NIOSH 6602
- Trichloroethylene in NIOSH 3701
- Various hydrocarbons in EPA 0040 and EPA SOPs

See Sample Bag Stability Data at [www.skccinc.com](http://www.skccinc.com) to help choose the best bag for the target compound.



# TYPES OF SAMPLE BAGS

## Tedlar®

- Most commonly used sample bag
- Classic bag for 1-2 day storage of organic vapors
- Available from SKC with polypropylene or stainless steel fittings in a wide range of sizes



# TYPES OF SAMPLE BAGS

## Standard FlexFoil

Suitable for gases and compounds not stable in Tedlar such as CO, CO<sub>2</sub>, H<sub>2</sub>, methane, hydrogen sulfide or sulfur hexafluoride.



SKC FlexFoil **PLUS** bags are suitable for both gases and organic vapors.





# **SAMPLE BAGS**

## **APPLICATIONS**

- Leaks, spills, emergency situations requiring rapid collection and analysis
- Peak concentrations from specific plant processes or worker tasks
- Field applications using on site portable, direct-reading instruments like PIDs
- Gases or highly volatile compounds for which sorbent tubes are not suitable



# **SAMPLE BAGS**

## **ADVANTAGES**

- Rapid screening can be done in the field using direct-reading instruments such as PIDs.
- Bag samples can also be analyzed by a laboratory for further characterization.
- Instantaneous/peak concentrations can be determined.



# STAINLESS STEEL CANISTERS

## LOW LEVEL VOC DETECTION

The interior of the canister is treated using SUMMA® or other processes so it does not react with collected compounds .



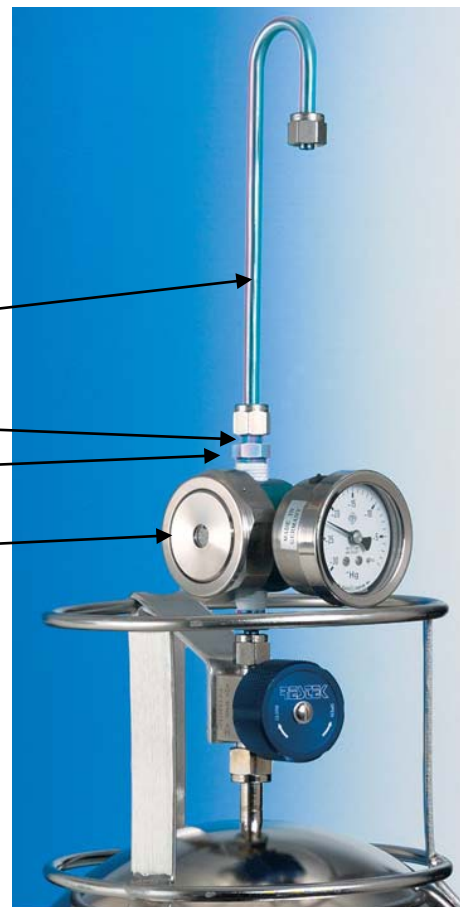
# STAINLESS STEEL CANISTERS

## KEY COMPONENTS

The canister sampling train includes:

- Stainless steel sampling inlet
- Particle filter
- Critical orifice
- Flow controller

A vacuum gauge is used to visually monitor canister status during sampling.



Source: [www.restekcorp.com](http://www.restekcorp.com)



# CANISTER SAMPLING

## THE BASICS

- Most canister sampling is done *passively* using a canister that has been evacuated to a specified vacuum level.
- Prior to sample collection, a qualified laboratory will clean and certify the canister, evacuate the canister to the appropriate level, and provide sample identification.
- At the sampling site, the valve is opened and air flows from the environment into the canister without the need for a pump.



# COLLECTION MEDIA AND SAMPLERS: FOR PARTICULATES



# DEFINING PARTICULATES

## Solid and liquid matter such as:

- Dusts-particles rendered airborne during crushing or grinding of rock-like material
- Fumes-airborne solid particles formed above molten metal
- Mists-droplets rendered airborne by bubbling, boiling, spraying or splashing
- Smokes-particles resulting from incomplete combustion of organic matter





# NOTE ON FIBERS SUCH AS ASBESTOS

- Fibers are particles that are 5  $\mu\text{m}$  or longer with a length-to width ratio (aspect ratio) of 3 to 1 or longer.
- Asbestos and other fibers are typically sampled using 25-mm MCE filters in black conductive cassettes following NIOSH Method 7400.
- Size-selective sampling (discussed next) is NOT done for asbestos.



# FILTERS

## SAMPLE MEDIA FOR AIRBORNE PARTICLES

- Published sampling and analytical methods provide specifications on the type of filter to be used for target compounds along with the pore size, diameter, and type of filter cassette.



# TYPES OF FILTER MEDIA

## MIXED CELLULOSE ESTER

### APPLICATIONS

- Asbestos, Fibers  
25-mm with Conductive Cassette
- Metals  
37-mm with Clear Styrene Cassette
- Oil Mist (Mineral)  
37-mm with Clear Styrene Cassette



# TYPES OF FILTER MEDIA

## POLYVINYL CHLORIDE (PVC)

### APPLICATIONS

- Particulates, Not Otherwise Classified
- Chromic Acid and Hexavalent Chromium
- NOW also being used by some labs for metals
- Silica using a respirable dust sampler



# TYPES OF FILTER MEDIA

## GLASS FIBER/QUARTZ

### APPLICATIONS

- Pesticides
- Coal Tar Pitch Volatiles, Selected PAHs
- PCB's
- Mercaptans, Isocyanates and more *when chemically coated*
- Diesel Particulate Matter (DPM)



# TYPES OF FILTER MEDIA

## PTFE

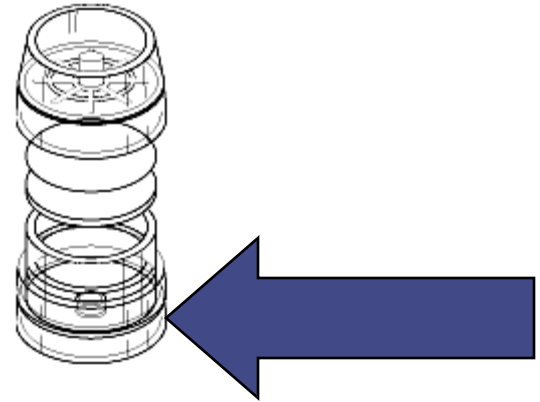
### APPLICATIONS

- Specified in EPA methods for environmental sampling of particulates in indoor or ambient air particulates.
- Specified in NIOSH methods for polyaromatic hydrocarbons (PAHs).



# CLOSED-FACE VS OPEN-FACE SAMPLING WITH CASSETTES

- Closed-face sampling refers to sample collection with the inlet piece of the filter cassette in place (and only the small plug removed).
- Open-face sampling refers to sample collection with the entire inlet removed. This approach is used for asbestos using 25-mm conductive cassettes.





# MATCHED-WEIGHT VS PRE-WEIGHED FILTER CASSETTES

- Matched-weight filter cassettes contain (2) filters of the same type matched in weight within 50-100  $\mu\text{g}$ .
- One filter is exposed to the dust; one is not.
- The difference in the weight is the weight of the collected sample.
- Advantage: Eliminate pre-weighing.
- Pre-weighed filter cassettes contain (1) filter that has been pre-weighed by the supplier.
- NOTE: For highest accuracy, the filter should be pre-and post-weighed by the lab on the same analytical balance.



# **FILTER FRONT AND BACK?**

- Filters do NOT have a designated front and back.
- As a result of the manufacturing process, filters may have a rough side and a shiny side.
- The shiny side is the preferred collection side for microscopic analysis; rough side is preferred for gravimetric analysis.

NOTE: Filters are WHITE.

The blue/yellow items in the box are wax paper spacers.



# THE HAZARD POTENTIAL OF AIRBORNE PARTICULATES



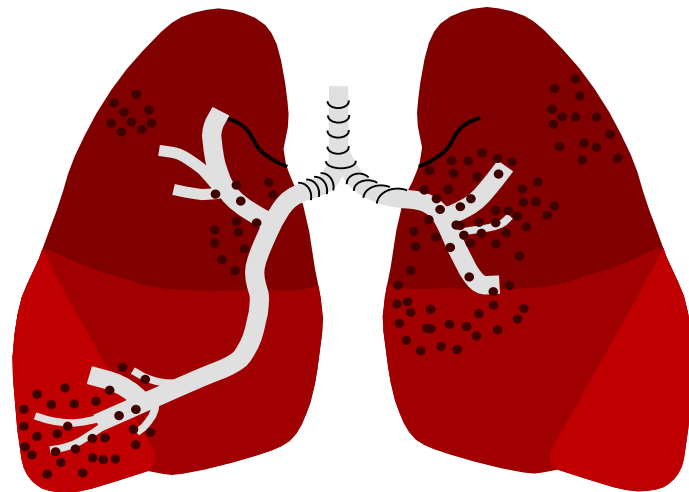
Determined by:

- Chemical composition
- Mass concentration
- Size characteristics



# SIZE CHARACTERISTICS OF AIRBORNE PARTICULATES

Determine the deposition site in the respiratory tract. Smaller particles will tend to deposit deep into the gas exchange region of the lung.



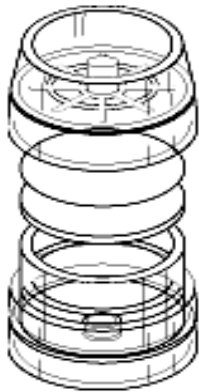
# **SIZE-SELECTIVE EXPOSURE GUIDELINES**

Because the size of the particle ultimately affects the health outcome, exposure guidelines have been issued for different sizes of particles.

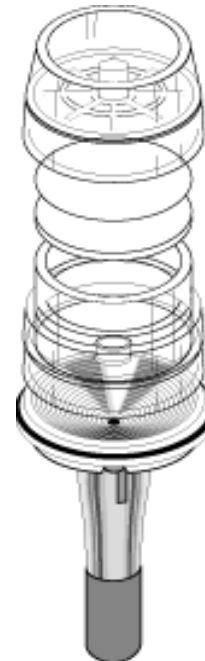


# TRADITIONAL PARTICULATE SIZE-SELECTIVE CRITERIA

Total Dust



Respirable Dust

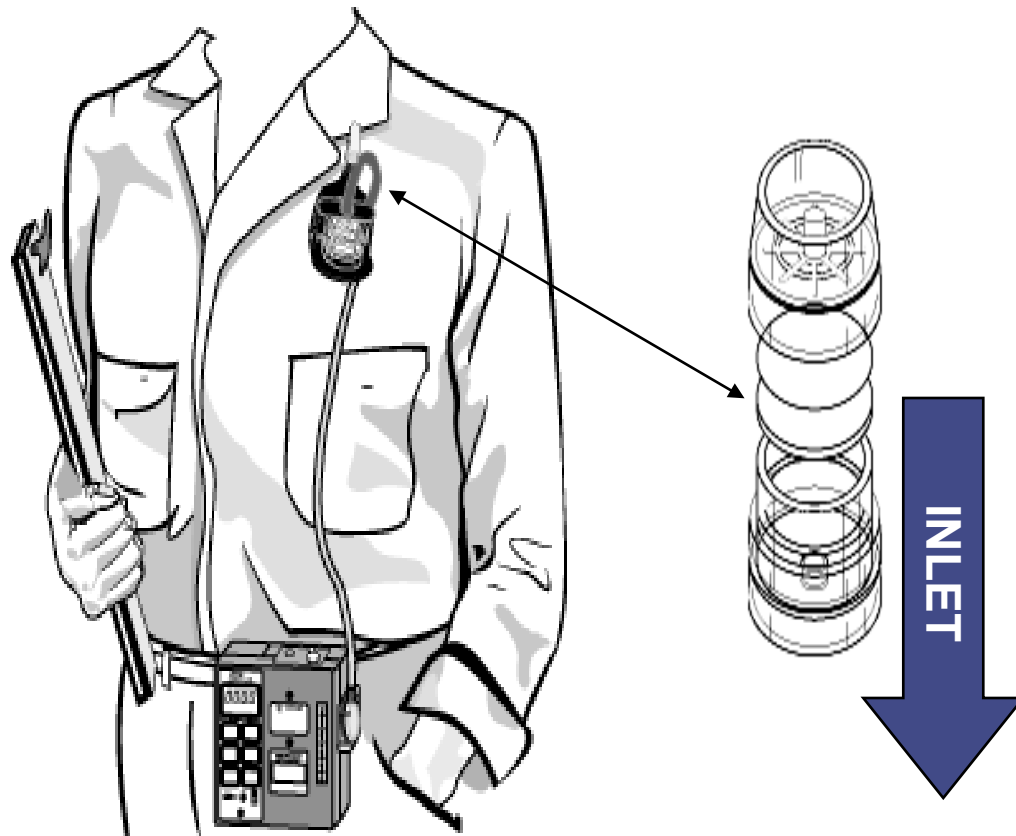


# TOTAL DUST DEFINED

- Dust that is captured onto a 37-mm filter loaded into a cassette and connected to a sampling pump calibrated to a flow of at least 1 L/min.
- The filter should be of a type and pore size appropriate to the particulate being sampled.
- Samples are collected in an area or in the breathing zone of workers.



# TOTAL DUST SAMPLING IN THE BREATHING ZONE





# RESPIRABLE DUST

## DEFINED

- Respirable dust particles are those small enough to reach the gas exchange alveolar region.
- Like total dust, respirable dust is normally collected onto a PVC filter.
- Preceding the filter, however, is a particle size-selective device that will separate the smaller respirable dust particles from the larger non-respirable particles.



# WHY USE A RESPIRABLE DUST SAMPLER?

- Because that's the way the chemical is regulated.
- Respirable dust samplers separate the smaller respirable particles from the larger particles in the dust cloud and will collect the particles onto the filter for analysis when connected to a sampling pump.
- Collecting larger, non-respirable particulates would inflate results of the sample, overestimating exposure.



# A HIGH PROFILE RESPIRABLE DUST CRYSTALLINE SILICA

Let's now discuss  
the 2016 U.S. OSHA  
Final Rule.



# **FOR ANY REGULATED CHEMICAL EXPOSURE LIMIT IS THE DRIVING FORCE**

## **NEW OSHA PERMISSIBLE EXPOSURE LIMIT (PEL)**

- 50  $\mu\text{g}/\text{m}^3$  as an 8-hr TWA for all forms of silica including quartz, cristobalite, and tridymite for all industry sectors covered by the rule.
- 25  $\mu\text{g}/\text{m}^3$  action level

**SO HOW CAN YOU SAMPLE SILICA TO MEET  
THE OSHA FINAL RULE?**



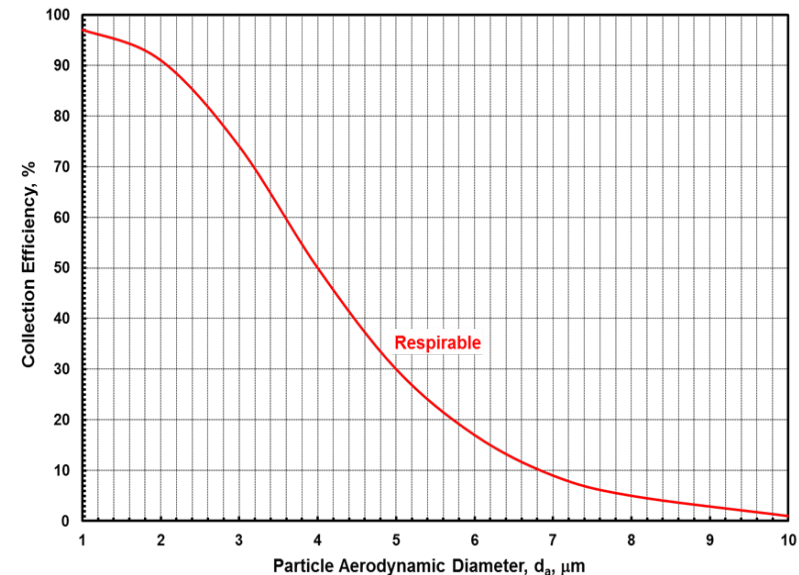
# OSHA ADOPTED NEW CRITERIA FOR SILICA SAMPLERS



- Respirable crystalline silica samplers must meet the performance criteria specified in ISO 7708.
- ISO 7708 is also referred to in the OSHA silica rule as the ISO/CEN convention. ISO=International Standards Organization.  
CEN=Committee for European Normalization.

# ISO 7708 CRITERIA FOR RESPIRABLE DUST SAMPLERS

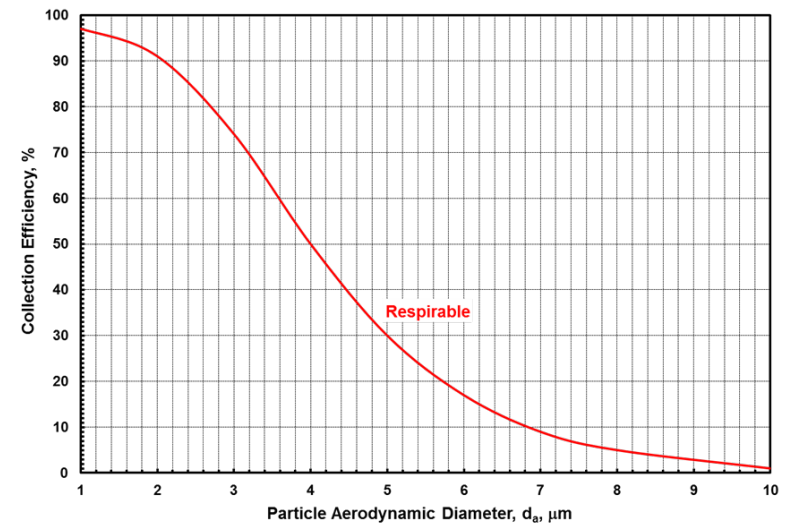
- The performance criteria is essentially a collection efficiency curve that specifies the efficiency of the sampler for particles of designated sizes.



# OF PARTICULAR NOTE

## THE 50% CUT-POINT

- This is the particle size that the sampler collects with 50% efficiency.
- ISO 7708 specifies a 50% (median) cut-point of 4  $\mu\text{m}$ .





# **RESPIRABLE DUST SAMPLERS:** **TO MEET OSHA CRITERIA**

## **OPTION #1** **CYCLONES**





# CYCLONE OPERATION

- Cyclones are inserted into filter cassettes containing PVC filters and are attached to sample pumps.
- Air enters through a slit on the side of the cyclone which creates cyclonic action.
- Through centrifugal force, large particles fall into the cap at the bottom (called a grit pot) and are discarded. Small particles are thrown onto the filter for analysis.



Cap must be  
in place  
during  
sampling!



# MORE ON CYCLONE OPERATION

- After sampling, the cyclone is removed and the filter is capped and sent to the lab.
- **DON'T TURN THE CYCLONE UPSIDE DOWN UNTIL YOU HAVE REMOVED THE FILTER!** If the large particles in the cap at the bottom get dumped onto your filter, your sample is invalid (and the results will look very high).
- Clean the cyclone with soap and water between every use or the dust stuck to the interior walls of the cyclone will affect your results.



# OLDER CYCLONE SAMPLERS TO MEET OSHA CRITERIA



Used at 1.7  
L/min

## 10-mm NYLON DORR- OLIVER CYCLONE

- U.S. OSHA inspectors have been using this cyclone since OSHA began in the 1970s.
- But it suffers from electrostatic effects causing particles to stick to the cyclone or cassette and not the filter.



# OTHER CYCLONE SAMPLERS TO MEET OSHA CRITERIA

## SKC ALUMINUM CYCLONE

- Listed in OSHA Final Rule on page 16439 of 29CFR 1910
- Used at 2.5 L/min to meet OSHA criteria



# SKC ALUMINUM CYCLONE

## ADVANTAGES

- Metal construction eliminates static electricity concerns.
- Larger collection area. The cyclone is inserted into the middle ring of a 3-piece filter cassette.
- Calibration adapter offers user convenience.



# OTHER CYCLONE SAMPLERS TO MEET OSHA CRITERIA

## GS-3 CYCLONE

- Originally designed for coal mines since aluminum can not be taken into an underground mine
- Conductive plastic construction eliminates static electricity concerns
- Not a spark hazard for coal mines
- Designated flow rate is 2.75 L/min to meet OSHA criteria



# **AVOID COMMON MISTAKES IN CYCLONE SAMPLING**

1. Make sure that you use the correct flowrate designated for your brand of cyclone. All cyclones are not created equal and don't use the same flowrate.
2. Make sure that you keep the "grit pot" (cap on the bottom) in place during sampling.
3. Load SKC cyclones into the MIDDLE RING of a THREE piece cassette.
4. Don't tip over the cyclone when its attached to the filter.





# **RESPIRABLE DUST SAMPLERS TO MEET ISO 7708 & OSHA CRITERIA**

## **NEW OPTIONS**





## **IMPORTANT NOTE** **IN THE OSHA FINAL RULE**

The new silica rule states on page 16439 that employers can use ANY sampling device that conforms to the ISO/CEN convention.



# NEW SAMPLERS

## FOR ISO 7708 & OSHA CRITERIA

Page 16439 goes on to say that in addition to traditional cyclones:

- “There are also personal impactors available for use at flowrates from 2 to 8 L/min that have been shown to conform closely with the ISO/CEN convention”.
- This data was supplied by SKC to the OSHA docket on new PPI samplers.



# DISPOSABLE PPI SAMPLERS



# HOW PPI SAMPLERS WORK



Same filter and same analysis as with cyclones.

Instead of a cyclone, PPI impactors have 4 internal, pre-oiled plates that scrub out larger particles. The impactor plates are sonically welded into place by SKC and require no assembly.



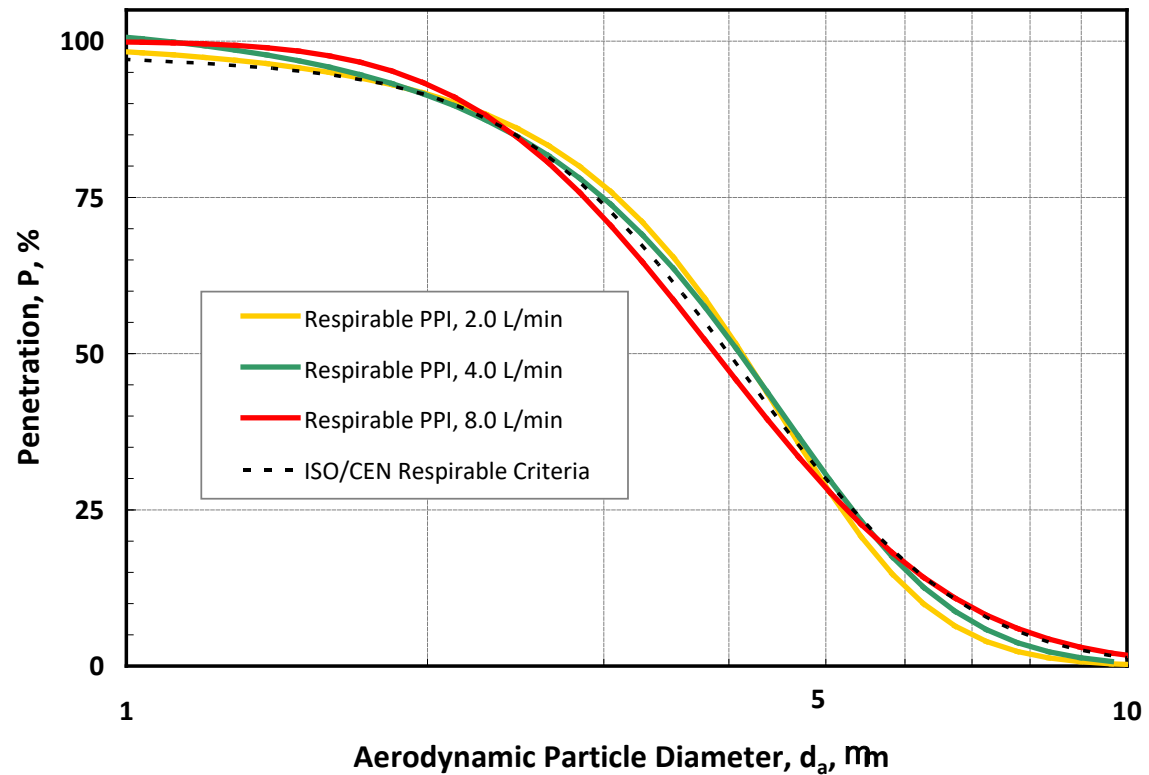
# MORE ON PPI OPERATION

The pump draws the air into the PPI. Larger particles are scrubbed out onto the plates and the smaller respirable dust is collected onto the PVC filter for analysis as usual.



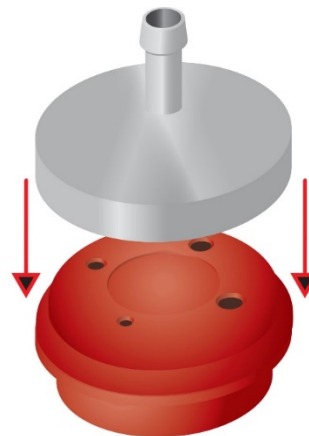


# PPI SAMPLER PERFORMANCE COMPARED TO ISO/OSHA CRITERIA



# MORE PPI ADVANTAGES

- A handy calibration adapter is available to attach the PPI to the calibrator (flowmeter).
- No tipping hazard. You can invert the sampler without causing large particles to land on the filter invalidating the sample.



# SKC RESPIRABLE PPI

## FLOWRATE OPTIONS



- Single-use, disposable PPI models are available for use at either 2, 4, or 8 L/min.
- This allows some flexibility for various sample times and airborne silica concentrations.





# CHOICE OF FLOWRATES

## USING THE PPI

- By far, the number one most popular PPI model is the 2 L/min model.
- If you want to sample a shorter duration, you could consider the option of using the 4 L/min PPI (SKC 225-3871) or 8 L/min PPI (SKC 225-3841).
- Keep in mind, you will need sampling pumps that can achieve these flowrates.

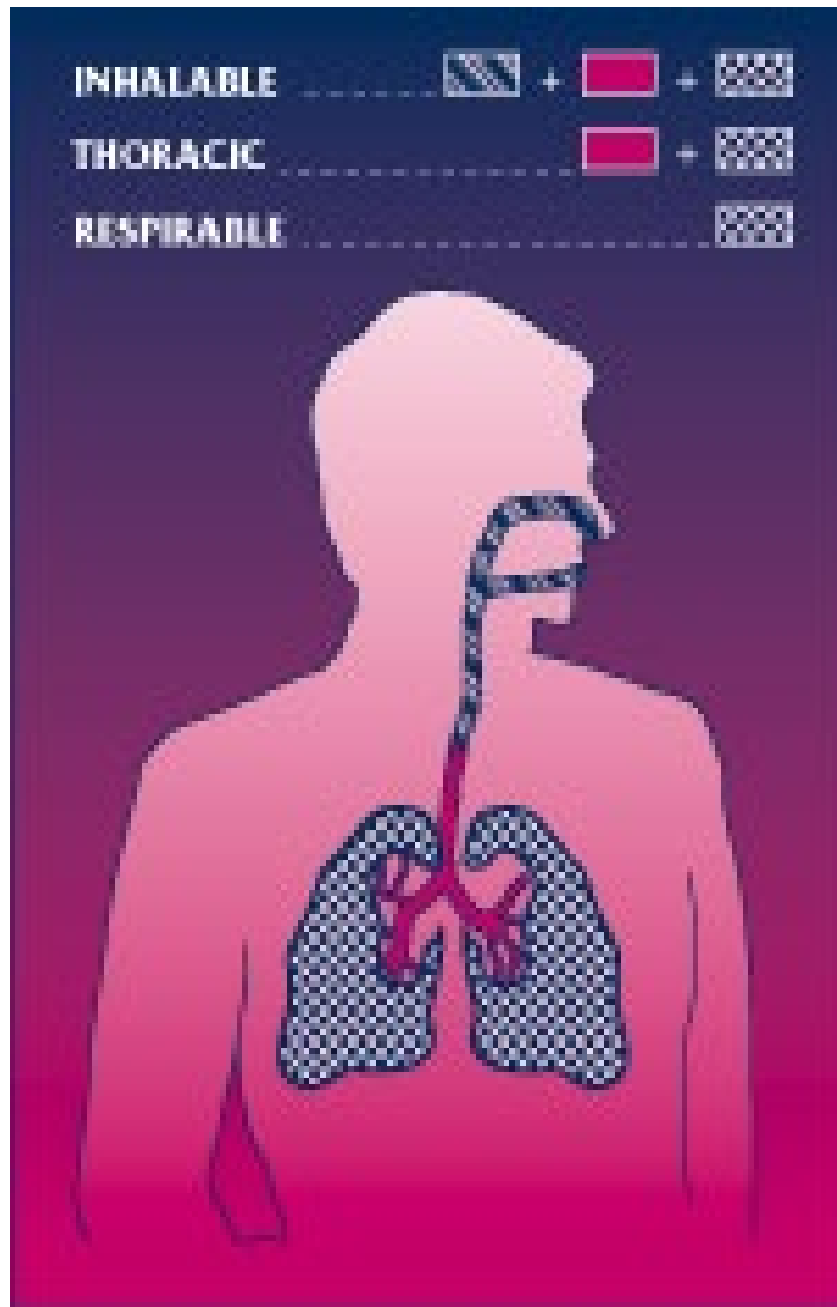


# OTHER PARTICULATE SIZE-FRACTIONS LISTED IN ISO 7708

In addition to respirable dust, ISO 7708 provides specifications for two other size fractions of particulate matter. These are also described in the TLV handbook:

- Inhalable Particulate
- Thoracic Particulate





Visual representation of the three particulate size-fractions described in ISO 7708 and the TLV handbook.

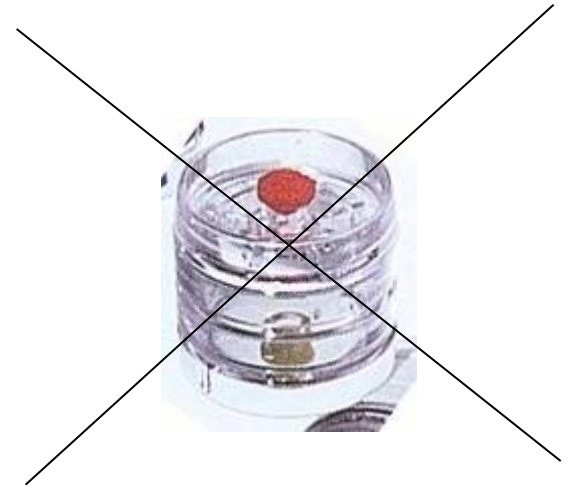
# INHALABLE PARTICULATE DEFINED

- Inhalable is the replacement for the old “total dust” term. Defined as particulate matter that is hazardous when deposited anywhere in the respiratory system.
- This includes larger particulates that enter the nose and mouth.
- Also includes materials that can produce systemic toxicity from deposition anywhere in the respiratory system.



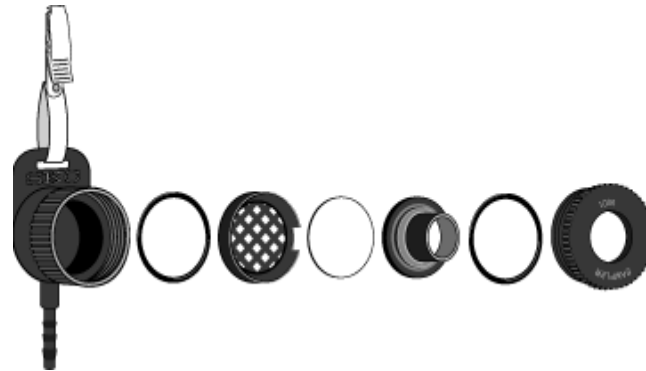
# INHALABLE SAMPLERS

- Defined as having a 50% cut-point of 100 microns.
- Traditional filter cassettes do not effectively capture ***inhalable*** particulate matter.
- The collection efficiency of a traditional 37-mm filter cassette drops to near zero at 30 microns.



# INHALABLE SAMPLERS

## IOM SAMPLER



Exploded View



# **SAMPLE COLLECTION**

## **WITH IOM SAMPLER**

- A 25-mm filter is loaded into the cassette using forceps and wearing gloves.
- The filter and cassette are equilibrated to standard laboratory conditions then weighed as a unit.
- The sample is typically collected at 2 L/min.
- The sample is weighed again following the procedures described above.



# INHALABLE SAMPLERS

## BUTTON SAMPLER

- Alternative to the IOM sampler for inhalable particulates.
- Inlet has a screen to keep large particles from landing onto the filter by blasting, splashing, or other work activities.





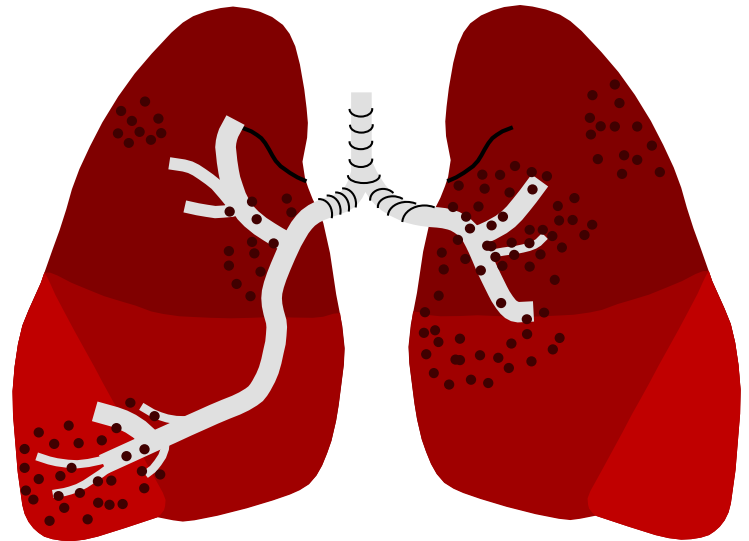
# **SAMPLE COLLECTION WITH BUTTON SAMPLER**

- A 25-mm filter is pre-weighed and loaded onto the stainless steel screen.
- The sample is collected at 4 L/min.
- The sample is weighed again following the procedures described above.



# THORACIC PARTICULATE DEFINED

- Materials that are hazardous when deposited anywhere within the lung airways and the gas-exchange region.
- Thoracic samplers have a 50% cut-point of 10  $\mu\text{m}$ .



# THORACIC TLVs

## As of 2018

Sulfuric acid

- TLV of 0.2 mg/m<sup>3</sup>

Cotton Dust

- TLV of 0.1 mg/m<sup>3</sup>

Hard Metals containing Cobalt and Tungsten carbide as Co

- TLV of 0.005 mg/m<sup>3</sup>



# THORACIC SAMPLING OPTIONS



THORACIC  
CYCLONE



SKC THORACIC  
PPI

# THANK YOU FOR YOUR INTEREST IN SKC TRAINING

- Email [skctech@skcinc.com](mailto:skctech@skcinc.com) with any questions
- Contact your local SKC representative.

