MOSES SOPs for Exposure Chambers

UNIVERSITY OF ROCHESTER MEDICAL CENTER

MOSES Standard Operating Procedure (SOP)

SOP **C18 MOSES Exposure Chamber and Ozone Analyzer Calibration (UofR)**

TITLE: Ozone Exposure Chamber and Ozone Analyzer Calibration

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Signature	**Date**

SOP UPDATED TO VERSION [d]

Explanations: [a] Incorporate the version of SOP into the file name, e.g., Ind_sputum_v10_sop.

[b] If the revision is minor the version number is increased by 0.1, i.e., to 1.1. <u>If the revision is major, do not overwrite the old document but create a new one with the updated file name and a new cover sheet with the new version number, i.e., 2.0. You must keep the old version on the server in SOPS directory and in your files!</u>

[c] Annual review does not change the version of the SOP.

[d] Change the version # as necessary.

1. SCOPE and APPLICATION

The MOSES study utilizes three separate 3-hour exposures to clean air, 0.07 ppm ozone, and 0.12 ppm ozone. The purpose of this SOP is to provide descriptions and operation instructions for the generation and monitoring of ozone in the MOSES study at the University of Rochester's Human Exposure Facility.

2. PREREQUISITES

This SOP assumes familiarity with the installation and operation of the instrument(s) where applicable. For detailed instructions in the operation of the instrument(s) refer to the respective operation and service manuals.

2.1 Equipment and Supplies

1) 03V5-0 Ozone Generator manufactured by Ozone Research and Equipment Corporation (OREC, a division of CCSi Inc., Akron, OH).
2) Ozone Analyzer Model 49i manufactured by Thermo Environmental Instruments, Inc. (TECO), Franklin, MA.
3) Ozone Analyzer Model 1008-PC manufactured by Dasibi Environmental Corp., Glendale, CA.
4) Gas Calibrator Model 8850 manufactured by Monitor Labs Inc., Boulder, CO.
5) Ozone Calibrator (Either Columbia Scientific Industries (CSI), Austin, TX Model 1700 or Teledyne-Advanced Pollution Instrumentation (API), San Diego, CA Model 703E supplied by NYSDEC).
6) Refrigerated circulating water bath. Endocal Model RTE-5 manufactured by Thermo Neslab, Newington, NH.
7) USP Oxygen tank with CGA 540 pressure regulator, Airgas East, Rochester, NY.
8) Flow Controller manufactured by Aalborg, Orangeburg, NY
9) Acquisition computer.
10) Data acquisition software, Labview Signal Express 2010 from National Instruments, Austin, TX.

2.2 Training Requirements

The operator conducting the tests should be trained in operating the instruments and performing the tests in a safe and standardized manner.

3. PROCEDURES

It is assumed that the instruments are set up according to manufacturer's instructions.

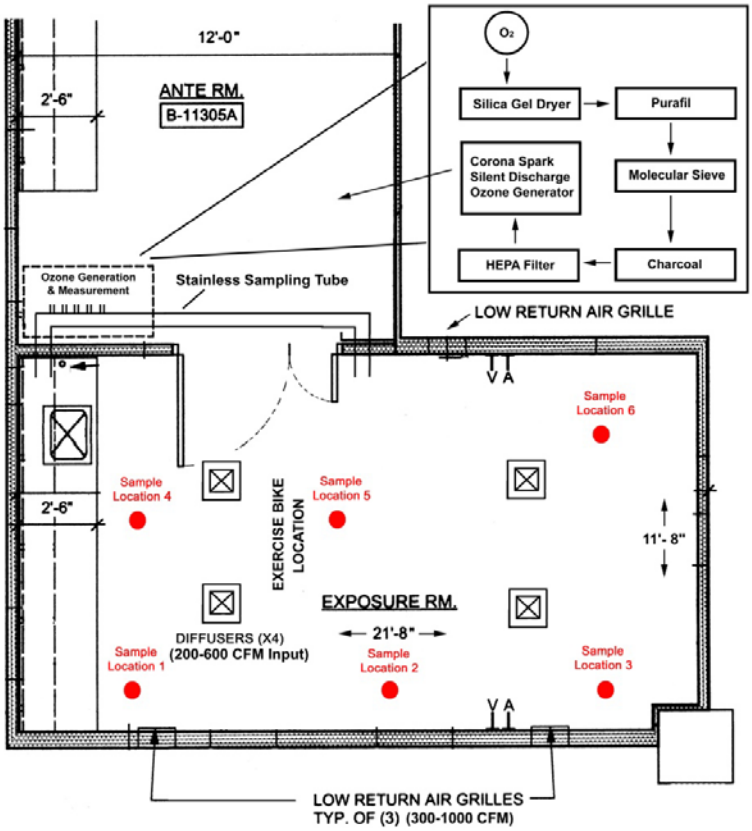
3.1 Exposure Chamber Specifications

A description of the exposure room is described in detail in SOP C13 Human Facility. In short, the room (MRBX B.11305B) is approximately 20' x 12' with four supply diffusers and four lower exhaust ducts configured to ensure even mixing and turnover of the room air. The supply air flow is approximately 500 CFM with approximately 15 air changes per hour. The outside supply air is temperature and humidity controlled, HEPA filtered, and passed through a 12-inch bed of SP Media Blend, which is a mix of 50% Purafil (potassium permanganate) and 50% Purakol (activated carbon). The heavy duty media pak of 12.6 lbs of SP Blend has been rated by the

Purafil supplier to last at least one year with continuous operation. Actual NOx and ozone scrubbing effectiveness will be evaluated on a weekly basis. During O_3 exposures, nitric oxide (NO) and nitrogen dioxide (NO_2) concentrations are typically <0.005 ppm. Ultrafine particles are typically less than 500 p/cm^3. Other monitored variables are chamber temperature, relative humidity, CO and CO_2.

Temperature is monitored with a calibrated (±0.1 °C), wide-range temperature platinum Resistance Temperature Detection (RTD, 100 Ω) probe (Vernier, Beaverton, OR). Humidity is monitored with Relative Humidity Sensor Probe (Vernier, Beaverton, OR). The probe includes a Hy-Cal Engineering IH-3602 Integrated Circuit humidity Sensor (±2% RH with saturated salt calibration). Both probes are attached to Labview data acquisition hardware and software. Control of the temperature and humidity is done by U of R facilities through the chamber thermostat (Siemens Controls). Figure 1 provides a schematic of the chamber.

Figure 1: Chamber Schematic in MRBX B.11305B.



3.2 Ozone Generation Instructions

A practical method of generating ozone is by silent arc discharge. We use a water-cooled, 0-1 Amp AC ozonator with filtered tank oxygen feed gas. The oxygen feed gas is filtered through

Hydro-Purge (Alltech Associates, Deerfield, IL) drying agent, PURAFIL (Potassium permanganate and AL, Thermo Environmental, Franklin, MA), and Activated Carbon. Preliminary test studies established the optimum parameters for setting up the ozone generator. The different ozone concentrations, based on chamber dilution of 500 CFM, were established by different settings of ion current and/or different volumes of oxygen delivered to the reaction chamber. i.e. 0.6 Amps current, oxygen flow of 4 L/min, reaction cell pressure of 5 psi yields 120 ppb of ozone, 0.5 Amps current, oxygen flow of 5 L/min, reaction cell pressure of 5.5 psi yields 70 ppb of ozone. The following are the specific operation instructions.

1) Turn on the ozone analyzer (Monitor Labs) at least 30 minutes prior to the start of exposure.
2) Close door to exposure room. Ensure circulating fan is on to the stainless steel gas sampling tube and room supply air is approximately 500 cfm and slightly negative to the ante room. (Supply and exhaust pneumatic room controls are to the right of the door)
3) Turn on the Ozone generator.
4) Open the Oxygen tank (Ensure the back pressure is set to 30 psi on the regulator). Each three hour exposure uses about 500 psi of tank oxygen, so a new tank is required for every four exposures.
5) Turn on the circulating water bath (preset to 20 ^{o}C at a flow of approximately 1 L/min).
6) Start Labview Signal Express Flow Controller application. Set control to 1 volt or 5.2 L/min. (flow controller calibration: **flow(L/min)=4.09*voltage+1.09**) Ensure oxygen pressure at generator gauge rises to approximately 5-8 psi. This can be adjusted from there as necessary.
7) Hold the 'raise' switch on the generator until a current is started. (approx. 5-10 seconds). Then adjust current to desired range of 0.1-0.2 amps using the raise/lower switch.
    a. In "Manual" mode, generator power may be varied manually using "raise/lower" switch. Increasing current increases ozone production.
    b. In "Automatic" mode, the generator power is varied via the analog signal and "control" potentiometer dial. Once placed in automatic mode, current is adjusted by feedback loop to provide agreement between the set point (concentration desired) and the chamber concentration measured by the ozone analyzer.
    c. Ozone generation for the MOSES study is run in 'Manual' mode due to the delay in response in generation of ozone in such a large room and the response of the analyzer. Control of the concentration is done with a manual control valve that varies the amount of Ozone introduced into supply air stream. The overflow of Ozone is directed to an exhaust hood separate from the chamber exhaust system.
    d. The desired set-point of ozone concentration is usually achieved within 10 minutes of initiating generation.

3.3 CONTROL OF CHAMBER CONCENTRATIONS, INCLUDING CONCENTRATION STABILITY CRITERIA

The ozone concentration in the chamber will be maintained to allow an exposure of 0, 70, and 120 ppb over 3 hours at 22°C $\pm$2°C and 40% RH $\pm$7% RH. The exposure concentrations are to be $\pm$10% of the target. Adjustment is made by the operator upon review of the real time signal to

determine the appropriate response ± of the ozone generation. Six points within the chamber itself were checked for distribution and uniformity during ozone generation. Each half hour the sample points were checked by moving the Dasibi Analyzer to the desired locations shown in Figure 1. Location 1 was started at 12:00 and sampled for a half hour, the analyzer was then moved to the next location while running for the next half hour and so on for the duration of the 3-hour test. Ozone concentrations are summarized in Table 1 and Figure 2. The ML8810 and Dasibi Analyzers were calibrated prior to sampling and showed agreement within 4%. The ML8810 analyzer sampled at the fixed chamber stainless steel sampling loop. Sampling with the Dasibi was done at a nominal height of four feet above the floor at each location. There were two minor adjustments made manually to the generator output flow at approximately 12:15 and 13:10 for the entire 3-hour test.

Figure 2: Example of an Ozone Generation in MRBX B.11305B Chamber in 2012.

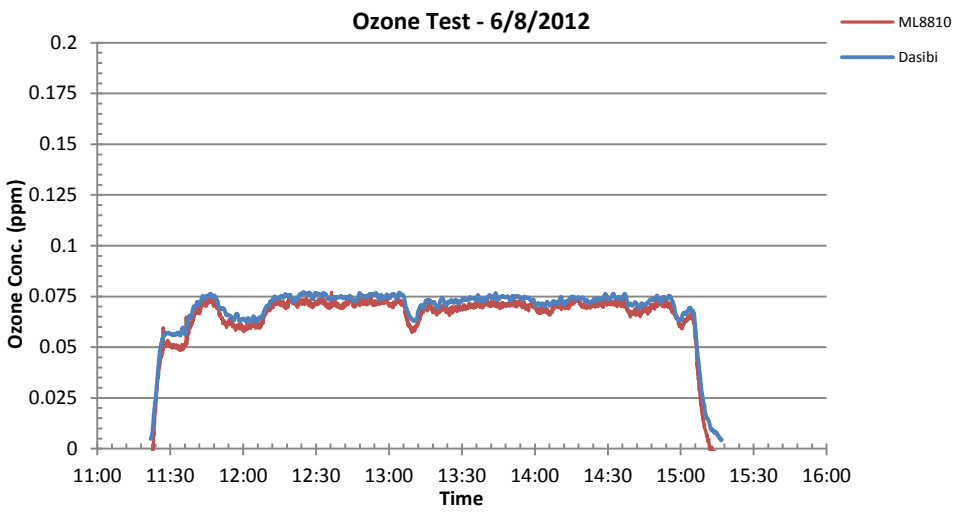


Table 1: Measurement of Chamber Ozone over 3 hours, 6/8/2012:

Sample Location	Dasibi 1008 Conc. (ppm) Mean±Stdev.	ML8810 Conc. (ppm) Mean±Stdev.
1	0.071±0.005	0.068±0.005
2	0.075±0.001	0.072±0.001
3	0.071±0.003	0.068±0.004
4	0.072±0.005	0.069±0.005
5	0.073±0.001	0.070±0.002
6	0.072±0.003	0.069±0.003
Avg.	0.073±0.003	0.069±0.003

3.4 OZONE MEASUREMENTS

The concentration of ozone is monitored and verified using two calibrated UV photometric analyzers (TECO Model 49i and Dasibi Model 1008-PC, respectively). Samples are extracted from the exposure chamber using an integral 3" stainless steel sampling manifold. The entrance to each manifold is located approximately at the center of the exposure chamber. A fan pulls chamber air at a speed of 3 m/sec through each exhaust manifold and analyzers are connected to the manifolds by ¼" Teflon tubing. The UV photometer determines the ozone concentration by measuring the attenuation of light due to ozone in the absorption cell (with a path length of 37.84 cm for Thermo and 71 cm for Dasibi) at a wavelength of 254 nm.

These UV photometric analyzer models have been designated by the US EPA as an equivalent method for the measurement of ambient concentrations of ozone pursuant with the requirements defined in 40 CFR Part 53. The designated equivalence method is # EQOA-0383-056, and the EPA designation date is March 10, 1983 for approved ranges of either 0-0.5 or 0-1.0 ppm full range. Our analyzer is operated 0-1 ppm full range.

The ozone measurement operating procedure is as follows:

1) Run the Labview Signal Express software to collect the ozone concentration data. Data are collected every five(5) seconds. Analyzer should be set to 1v = 1ppm on front panel.
2) Check a zero reading by attaching the sample inlet of the analyzer to the UZA port of the calibrator (model 8550). UZA (Unpolluted Zero Air) is made using laboratory house input air through a pressure regulator at 6 psi with an in-line oxidizer, charcoal scrubber, and filter. The analyzer is connected with the Teflon sample line to an overflow of the UZA to sample at atmospheric pressure. 0-2 ppb should be achieved within 5 minutes of sampling. If zero is not achieved the analyzer needs to be adjusted via set screw inside the case (see manual for further instruction).
3) Return the analyzer teflon sample tube to the stainless steel room sample to measure the real time exposure room concentration. The concentration is held ±10% of the desired concentration.
4) Data are collected until exposure is completed, the subject leaves the chamber, and the ozone is evacuated from the chamber. The data are then uploaded from Signal Express to an Excel spreadsheet. Hourly averages and standard deviations can then be calculated. An example is shown in Figure 2.

4. CALIBRATION PROCEDURES

The ozone analyzers are calibrated using a multipoint calibration that includes a zero and four upscale points. The highest point is higher than the highest concentration for active protocols. The operator uses the following procedure.

1) The operator connects the analyzers to a zero air source (Monitor Labs Calibrator Model 8550) and purges the analyzers for 10 minutes. The zero air source consists of medical grade wall air passed through a silica gel dryer, molecular sieve, activated carbon charcoal, purafil, and a HEPA filter. The zero air output is regulated with the ML Calibrator with an overflow to supply ambient pressure zero air to the analyzers.

2) The analyzers are then attached to a manifold with the ozone produced by the reference UV source (CSI Model 1700 or Teledyne-API Model 703E).
3) The operator then uses at least four fixed set points of ozone up to 425 ppb. The operator waits until both analyzers have settled including the reference photometer and records each of the analyzer's reading for that point.
4) When all of the points have been completed the analyzers are again connected to the zero air source calibrator to check for a zero reading.
5) The calibration constants are verified by the operator. If accepted, operator replaces the old calibration constants with the new ones in the TECO 49i.

The ozone analyzers are verified by at least a four point audit within 1 working day before each exposure. The calibration standard is NYSDEC supplied calibrator with the same zero air used by the calibration system.

5. CERTIFICATION OF STANDARD AND TRACEABILITY TO AN EPA STANDARD REFERENCE PHOTOMETER

The Thermo Environmental Model 49i is certified as a transfer standard by verifying it against a certified primary standard reference photometer by the New York Department of Environmental Conservation (NYSDEC) in Albany, NY. The Albany photometer is traceable to primary EPA photometer in Edison, NJ at least every six months. The verification process is done by verifying the accuracy by relating the analyzer to the output of the higher authority (level) photometer in Albany, and demonstrating that the repeatability of the transfer standard is within the specified limits. Verification requires the averaging of 6 comparisons between the transfer standard and the higher level UV O_3 standard. Each comparison must cover the full range of O_3 concentrations and is to be carried out on a different day to the primary standard for six days. This procedure is commonly referred to as a 6x6 certification and is performed for this protocol at least every three years. This procedure follows the EPA Ozone Transfer Standard Guidance Document 11/2010. The full range of 400 ppb will be checked with each point to be within 4% accuracy after correction for atmospheric pressure.

The TECO Model 49i is subsequently verified for accuracy <u>quarterly</u> using a reference photometer supplied by the NYSDEC. (Typically a CSI Model 1700 or Teledyne-API Model 703E) This reference photometer is also a certified transfer standard that is verified in Albany at least every six months by the NYSDEC.

6. PREVENTIVE MAINTENANCE, INCLUDING ADDRESSING MOISTURE ISSUES

During each calibration prior to each exposure the operator checks the cell lamp intensities, cell flows, instrument pressure, and instrument temperatures. The scrubbers on the ozone analyzers are routinely changed every three years to prevent moisture problems. We will also periodically (monthly) check changes in the analyzer outputs when going from dry air in the calibration manifold to humid air in the chamber.

The chamber's hard surfaces are wiped down between subject exposures (at least 12 hours prior to the next exposure session), and at least weekly, with a disinfectant cleaner and deodorant (**Virex II 256, Johnson Diversity Inc.**: it is bactericidal, virucidal, fungicidal, mildewcidal, deodorizer, mildewstatic, and meets OSHA Bloodborne Pathogen Standard for HBV & HIV). A concentrate of this disinfectant is mixed as needed according to directions, at least 1:256 dilution or ½ oz. per gallon of water for each cleaning. The chamber room is continuously

(24 hrs./day, seven days/week) being circulated with charcoal, purafil-filtered air. The room turnover rate is approximately 13 times/hr. The filters are changed as per our chamber SOP, manufacturer's recommendation, or if a higher than normal pollutant measurement occurs. (O_3, NO_2, NO, CO, CO_2).

7. REFERENCES

1) Thermo Scientific Model 49i Ozone Analyzer Operation Manual (2010)
2) Dasibi Series 1008 UV Photometric Ozone Analyzer Model 1008-PC Operating and Maintenance Manual (1990)
3) OREC Model 03V5-0 Ozonator Operating Manual (1988)