United States Environmental Protection Agency

INTERNAL OAQPS PROCEDURE

Standard Operating Procedure (SOP) for the Verification and Re-Verification of EPA's Ozone Standard Reference Photometers

Office of Air Quality Planning and Standards (OAQPS)
Office of Research and Development (ORD)
National Risk Management and Research Laboratories (NRMRL)

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1.0 Scope and Applicability

In ambient air monitoring applications, gas concentration standards are required for the calibration and auditing of various ambient gas monitors. Because of the instability of Ozone (O_3), the certification of O_3 concentrations by Standard Reference Materials (SRMs) is impossible. Therefore a Standard Reference Photometer (SRP) was developed as a primary standard to validate the linearity of other photometers when challenged with various concentrations of locally generated O_3 gas. This document has been prepared to assist the United States Environmental Protection Agency (EPA) Regional Operators to operate the National Institute of Standards and Technology (NIST) Standard Reference Photometer (SRP) located and used in their respective Regions. Two very important related US government documents are the NIST SRP Operations Manual, and the EPA Technical Assistance Document (TAD), Transfer Standards for Calibration of Air Monitoring Analyzers for Ozone. The NIST document addresses how NIST uses the SRP at NIST, but does not address the requirements for EPA, state, local, tribal (and CASTNET) air monitoring networks, regarding requirements for ozone traceability, which the EPA SRP network supports. The first step in assuring that an ozone device (SRP or commercial) will do what it is needed to do, is called qualification. That process is addressed in the TAD, and is not addressed in this SOP. The scope of this Standard Operating Procedure (SOP) will include program background, shipping, receiving, operation, repairs, Verification and Re-Verification of SRPs. This document will explain:

1. The Re-Verification of the Regional SRP (as the Guest instrument) against the Traveling SRP (as the host instrument). The host is considered the standard of higher authority. This will be the preferred setup.
2. The Re-Verification of the Regional SRP (as a host instrument) against the Traveling SRP-07 (as the guest instrument). This is the optional.
3. The Verification or Re-Verification of all Level 2 standards from monitoring organization (as a Guest instrument) to the Regional SRP as the host instrument.

Important Note: If you have any technical questions about the procedure covered in this US EPA SOP, contact the EPA Technical Lead, Scott Moore at 919-541-5104, or email moore.scott@epa.gov. For other EPA SRP program questions, contact Mark Shanis, at 919-541-1323, or email shanis.mark@epa.gov.

1.2 O_3 Photometer Traceability

O_3 photometers (used as a standard) can be classified into two basic groups: stationary and traveling standards. A stationary O_3 standard is a photometer (with an internal or external O_3 generator) that would be dedicated exclusively to use as a calibration standard. The stationary O_3 standard should always be used with clean dry air (preferably zero air) and never used for ambient air sampling. Consideration should be given to locating the calibration photometer in a clean laboratory where the photometer can be stationary, protected from physical shock, operated by a responsible analyst and used as a common standard for all field calibrations via transfer standards. See Title 40 of the Code of Federal Regulations, Part 50, Appendix D (40 CFR Part 50) Section 5.1. The calibration procedure is based on the photometric assay of O_3 concentrations (six concentration points as per Section 5.5.5) in a dynamic flow system. The concentration of O_3 in an absorption cell is determined from a measurement of the amount of 254 nm light absorbed by the sample. An O_3 traveling standard is a transportable device capable of producing O_3 concentrations and accurately analyzing those O_3 concentrations that has been verified by another O_3 photometer of a higher or equal level of authority. In ambient air monitoring applications, precise O_3 photometers called transfer standards are required for the calibration of O_3 analyzers. Therefore, an O_3 standard must be used to measure ozone that has to be generated and its concentration measured by the standard and the station analyzer. That is, the concentrations measured by the analyzer have to be verified, or calibrated, on site. When the monitor to be calibrated is located at a remote monitoring site, it is necessary to use a transfer standard that is traceable to a more authoritative standard. “Traceability can be defined as an unbroken record of documentation ("documentation traceability") or an unbroken chain of measurements and associated uncertainties ("metrological traceability"). As used here, the word "traceability" always means "metrological traceability."

The National Institute of Standards and Technology (NIST) maintains the U.S. national standards for ozone. In other countries, similar national standards laboratories perform the same function.

To establish and maintain traceability, the readings of an ozone analyzer are compared to a NIST made Ozone Standard Reference Photometer (SRP).

This process of using NIST traceable standards to verify the concentrations of an analyzer used to monitor ambient air for ozone is implemented for all the US EPA, state, local, and tribal, and CASTNET network ozone analyzers used to make comparisons against the ozone National Ambient Air Quality Standards. The testing process is called verification. Once the accuracy of a standard is authenticated, that standard can serve as a reference to establish traceability for other ozone standards or analyzers. This process can be continued, providing an unbroken chain of measurements from the final ozone analyzer all the way back to the NIST standards. The final measurement will have traceability to NIST standards if the following conditions are met:

1. An unbroken chain of measurements back to NIST standards is maintained.
2. Each step of the chain has known and documented uncertainties.
3. There is a quality system to ensure that the ozone standards and associated measurement equipment maintain their measurement uncertainty (accuracy)." Quoted from: http://www.nist.gov/pml/mercury_traceability.cfm

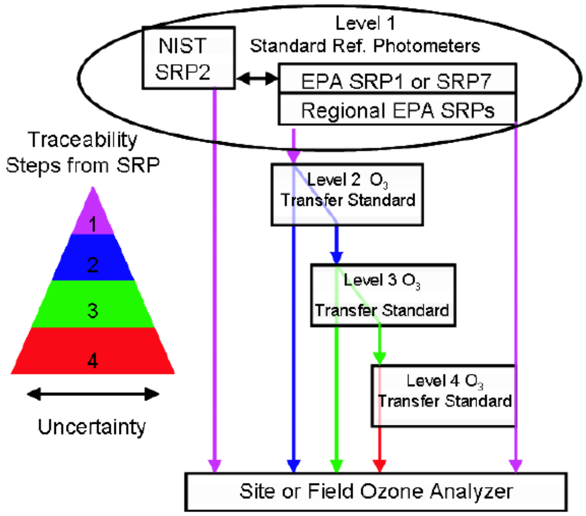


Figure 1.2.1 (Taken from TAD)

Measurement uncertainty describes a region about an observed value of a physical quantity that is likely to enclose the true value of that quantity. Measurement uncertainty is related to both the systematic and random error of a measurement and depends on both the bias and precision of the measurement instrument. At each measurement phase errors can occur. In most cases, the errors are additive.

Figure 1.2.1 represents the scheme that will be employed to ensure that the use of O_3 transfer standards is applied in a manner that will ensure a specified level of measurement uncertainty and traceability. As detailed within the Technical Assistance Document (TAD) for Transfer Standards for Calibration of Air Monitoring Analyzers for O_3 dated November 2010.
http://www.epa.gov/ttn/amtic/files/ambient/qaqc/OzoneTransferStandardGuidance.pdf

1.3 EPA's Standard Reference Photometer Network

The Network of EPA's Standard Reference Photometers (SRPs) is structured to provide traceability for O_3 measurements from NIST to each of the various EPA Regions. In turn, each of the regional SRPs provides traceability to various State, County, City, Tribal and other local authorities for auditing and ambient monitoring of O_3. There are a total of 11 SRPs within the EPA network (see Fig 1.3.1 and Table 1.3-1). Each year SRP Serial Number 01 (SRP-01) will travel to NIST to be Re-Verified against the NIST SRP Serial Number 02 (SRP-02) and when available, NIST's SRP Serial Number 00 (SRP-00). During the one year re-verification period, SRP Serial Number 07 (SRP-07) will be Re-Verified against SRP-01 and then SRP-07 (also referred to as the traveling SRP) is shipped to the next EPA region for Re-Verification. Once the Re-Verification is completed, the data are sent to the program coordinator, who verifies that the procedures have been followed and completes a report for the site operator. The traveling SRP is then shipped back to the program coordinator, re-verified against SRP-01 and then prepared to ship to the next region. The annual SRP verification schedule is posted on AMTIC http://www.epa.gov/ttn/amtic/srpqa.html

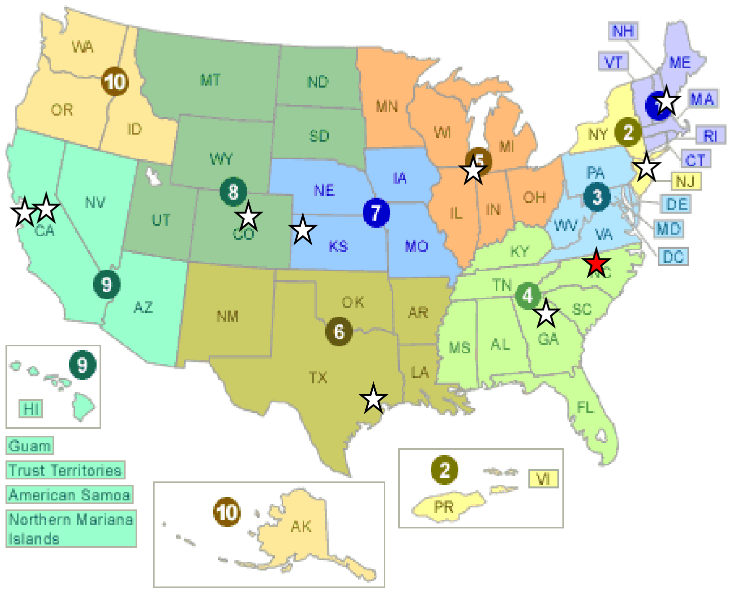


Figure 1.3.1 US O_3 Program Locations

Some of the regional operators have selected the option to ship their SRP to the program coordinator to perform annual maintenance, repairs (if needed) and re-verification. This Standard Operation Procedure (SOP) will be focusing on the re-verification process for the SRP program. The following re-verification steps will be discussed in this SOP: receiving and unpacking SRP-07 from the cases, setting up SRP-07 to run the re-verification, running the Quality Assurance/Quality Control (QA/QC) checks, setting the re-verification parameters, running the re-verification, breaking down SRP-07, packing SRP-07 and shipping SRP-07. All the steps required for setting up and comparing SRP 7 (as the Host) to a Regional SRP (as the Guest) start in Subsection 9.2.1 and continue through subsection 9.4.2. Following the re-verification process there will be a section on repairs and annual maintenance.

Table 1.3-1 Locations of SRPs

SRP	Region	Location
SRP-09	Region 1	N. Chelmsford, MA
SRP-03	Region 2	Edison, NJ
SRP-10	Region 4	Athens, GA
SRP-06	Region 5	Chicago, IL
SRP-05	Region 6	Houston, TX
SRP-13	Region 7	Kansas City, KS
SRP-08	Region 8	Golden, CO
SRP-36	Region 9	Richmond, CA
SRP-04	ARB	Sacramento, CA
SRP-01	RTP	RTP, NC
SRP-07	RTP	SRP7 to NIST

2.0 Summary of Method -NIST Standard Reference Photometer (SRP) History

2.1 Development of the SRP

A collaborative effort between NIST and EPA in the development of the original SRPs has become the basis for O_3 measurements globally. The SRP Program began in the early 1980s as a collaborative effort between NIST and the EPA to design, construct, certify and deploy a network of identical O_3 reference instruments. The design specifications called for an instrument with a standard uncertainty of ±2 nmol/mol (ppbv) in the range of 0 nmol/mol to 1000 nmol/mol. Since the SRPs have been deployed, beginning in 1983, the performance of all SRPs has exceeded the design specifications. In the US, two (2) SRPs are maintained by NIST, one serving as the NIST standard and the other as a backup/traveling instrument. Eleven additional SRPs are maintained by the EPA at various EPA Regional laboratories across the United States to facilitate requests for local access to an authoritative (i.e., NIST) reference standard for O_3. The current international network of SRPs totals nearly fifty (50) worldwide that now includes instruments maintained in at least fifteen countries. The international network is coordinated by the Bureau Internationale des Poids et Mésures (BIPM) in France, which maintains the international responsibility for the comparison of national O_3 standards as the NIST does here in the United States.

Over the past several years, the network of NIST SRPs has undergone significant upgrades in its electronic systems, sampling configuration, and control software. Each SRP consists of a separate optical bench and two instrumentation modules (electronics and pneumatics). The Optical Bench consists of a single mercury ultraviolet (UV) discharge lamp, UV filter, UV beam splitter, two absorption cells, and signal-processing electronics.

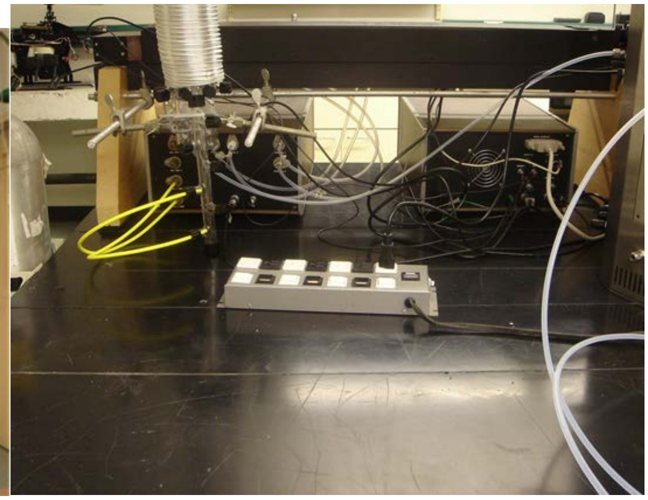


Figure 2.1.1 Front View of SRP-01 *Figure 2.1.2 Rear view of SRP-01*

In May 2003, a new electronics module was designed as a plug-compatible replacement for the original unit to simplify upgrading of existing systems. Several improvements were made in the overall electronics module design to provide enhanced stability and to simplify operation. The electronics upgrade also involved a complete redesign of the detector module. The new detector voltage to frequency (V/f) converters are more stable and three times more sensitive than the previous converters. This additional stability and sensitivity provides increased measurement sensitivity through higher resolution scalar counts, a longer source lamp life, a lower noise level and a smoother output signal. The most recent upgrade to the SRPs solved a previously existing O_3 measurement bias and improved the accuracy of the pressure measurements by using equal length sample and reference lines, effectively balancing the pressure drop through each sample pathway. The new dual manifold sampling configuration is now considered standard on the SRP. New SRPs are produced and delivered using this sampling configuration.

2.2 Hardware Upgrades

SRPs made by NIST and used by EPA have been upgraded over time. From 1989 through 2004, a series of 11 individual upgrades were made, including redesign" of the detector module (see reference, "Upgrade and Intercomparison…," June , 2004). The following material in quotes are derived from the two references listed in section 12.4.

The first upgrade involved "replacing 4 independent function circuit boards, and a separate relay circuit board, with one main circuit board, called the Digital Interface and Timing Generation (DI/TG) Board. Board connections and mounting bracket were improved, and a 24 DC volt power supply added. Critical parameter adjustment controls were installed on the front panel, which allowed the unit to remain thermally stable during adjustment, which the old design had not allowed. The main board is a multi-layer board providing more stringent handling of transmitted signals. Particular attention was paid to separation of the digital and analog grounds to avoid interference, and various test points are available for convenience during testing. The specifications of the signal amplifiers used on the temperature and pressure input signals are 50% more stable than those used in the original design. Electric contact relays used in the original design for powering the lamp shutter and solenoid valves were replaced by solid state relays, thus minimizing power spikes throughout the system. The front panel displays for the two scaler channels, temperature and pressure, are brighter and easier to view".

"The electronics upgrade also involved a complete redesign of the detector module. The original housing was costly and impractical. The new design utilizes an extruded aluminum box with internal mounting brackets. The new detector/ preamp board provides 2 independent, stable voltages corresponding to the light intensity in the absorption cells and utilizes a signal amplifier with a factor of 5 improvement in stability compared to its predecessor. The new detector V/f converters are more stable, and three times faster than the previous ones. These features provide increased measurement sensitivity through higher resolution scalar counts, and longer source lamp life. The open-collector devices used in the original design for transmitting the V/f output signals to the electronics module were replaced with TTL line drivers. Further design improvements were made such that these signals are now transmitted over independent coaxial cables for improved signal fidelity. Summary: Overall, the new detector modules provide faster V/f conversion, a lower noise level, and a smoother output signal."

"Use of the Industry Standard Architecture (ISA) bus in Personal Computers (PCs) has vanished, and it has become impossible to purchase a PC with the ISA expansion slots necessary for the operation of the version 3 software. A fourth generation control program for the SRP has been developed by NIST. New software was required due to the unavailability of new computers with the ISA bus. New computers then became available with the Peripheral Component Interface (PCI) bus only, which means that the SRPs had to use these PCI control cards. A direct replacement of the ISA multi-function card previously used was not available in a PCI version. The PCI replacement uses a different connector, so an additional signal distribution module was designed and produced to handle the new connector used on the PCI card. This modification allowed continued use of existing SRP control cables. A direct replacement for the 24-bit Digital Input/Output (DIO) card for controlling a guest SRP became available for PCI bus operation. Several of the EPA operators had their SRP computer connected to the Agency network as a convenient way to store data to a server and work on data from a remote location. Newer computers may have at least one PCI slot and several e-PCIs."

A "dual external manifold was designed using borosilicate glass and the same fittings as on the original SRP nmanifolds.TO incorporate the new manifolds into existing SRPs, the original manifolds were removed from the front panel of the pneumatics module, and an adaptor plate installed with 2 Teflon bulkhead unions. The new arrangement has allowed easy connections to manifolds in commercial ozone instruments."

By April of 2013, the redesign and testing of the source /optics lamp block with different mounting components had been made and tested. During the same period, an issue of optical path length bias had been addressed, and then tested, using a modified design for the optical cell, incorporating 3°-tilted windows. The SRPs upgraded with these changes have shown improved overall agreement.

Research is currently in process to adapt the SRP to newer technologies. More information will become available as solutions are documented.

2.3 Software Upgrades

Since the beginning of the SRP program, control of the instrument was handled in part by computer. Originally, the SRP was controlled by using a Hewlett Packard HP85B computer with a General (Control) Program Input/ Output GPIO interface card. This version 1 control software was written using HP Series 80 software. While certain functions of this operation were automated, an operator was required to start each concentration measurement manually and change O_3 generator settings manually. Additionally, an independent computer was required for the operation of each SRP while verifying one SRP against another SRP. In 1992, NIST developed an SRP control system based on the personal computer (PC). An Industry Standard Architecture (ISA) 24-bit digital input/output (DIO) control card was used to interface the SRP to the PC. Version 2 control software was written using QuickBASIC™ version 4.5. At the request of EPA, the software was written to emulate the original software. While some improvements were made, the basic functionality of the SRP control remained the same.

In 1995, NIST developed an automated control system allowing multiple SRPs to be compared automatically against each other using one PC and without the presence of an operator. Additionally, analog output signals from commercial O_3 instruments could be read simultaneously by the same PC to provide automated verifications. Version 3 control software was written in the "C" programming language using a front-end graphical interface similar to Windows™. While Windows™ version 3.1 was available during this time, the new version 3 control software was Disk Operating System (DOS)-based. When Windows™ 95 began to be used on SRP control computers, SRP operators began to notice problems running the version 3 control software in Windows™ from a DOS shell. Most of the problems were related to reading the analog output signal from a commercial O_3 instrument and seemed to be PC-dependent. Increasing processor speeds of the newer PCs may have contributed to the problems. For these reasons, in 2000 NIST began developing a new software/hardware update for the SRP using Peripheral Component Interconnect (PCI) control cards and Windows-based VisualBasic™ software. Use of the version 4.4.1 SRP control system began at NIST in November 2001 and became available to all other SRP users in April 2002.

The software called "SRP Control" was written in VisualBasic™ version 6.0 and operates under the Windows™ NT, 2000 or XP environment. The program works in conjunction with Excel™ 2003 (version-xls) where all data collected from the program are automatically reported to templates, which can be customized to suit the user's needs. The templates can have additional macros performing specific calculations or operations available in Excel™. The control system is operated using scripts, which call lower-level functions embedded in the program. The user cannot modify the lower-level functions, but the default scripts can be modified or new scripts written to obtain customized system operations. The ability to customize system operations allows the user to create custom program control without the possibility of corruption to the basic functions of the SRP. Comparisons to an SRP can be done for up to three guest instruments (Level 2 transfer standards), including multiple guest SRPs. The guest system data input can be via SRP digital interface, serial communication, analog signal or manual input of O_3 concentrations from the guest instrument display. The internal O_3 generator available on some guest O_3 instrumentation can be operated using serial communication. Calibration methods created for specific instrumentation can be saved and recalled for repeated use or linked together in series to provide consecutive calibrations with different formats on the same instrument. Network access to the control system providing some control functions is available, but can be difficult to implement with network security systems. The current version is 4.4.1 from the NIST web site http://www.nist.gov.

NIST has worked on the next upgrade that will enhance some of the graphic display. It will be possible to connect SRP to SRP via Universal Serial Bus (USB) interface. However, additional hardware would need to be purchased, and there is a known O_3 lamp voltage issue that limits the control of the O_3 lamp from zero-100% to zero-50%. The new software was written in Lab View, and then compiled into an executable program to run on the computer (Windows 7 operating system). The free driver will need to be downloaded, in order to run the program.

As indicated, Lab View will replace Visual Basic. The version of the software in Lab View is currently being Beta tested, by NIST, EPA and Canada. It is not yet ready for distribution. When it becomes available, it will be obtained through the assistance of the EPA ORD SRP coordinator. For the time being, until the new software is installed, for those operators with Windows XP, since XP is no longer supported, workarounds may be needed. At least one SRP operator has to use a USB drive to get the data off the SRP computer.

Proposed Future Upgrades: Low voltage temperature cards, made by the company STOLAB**,** to minimize a 0.2 °C observed temperature bias; four temperature sensors, one each on the inlet and outlet of each of the cells. Possibly two pressure transducers to measure the pressure from each cell and the LabView Control Software.

3. Definitions and Acronyms:

3.1. Definitions

Qualification: The process of demonstrating that a candidate device is sufficiently stable (repeatable) to be used as a transfer standard. Repeatability is necessary over a range of variables, such as temperature, line voltage, barometric pressure, elapsed time, operator adjustments, or other conditions that might be encountered during intended use of the device. Tests and possible compensation techniques for common variables may be provided.

Verification: confirmation by examination and provision of objective evidence that specified requirements have been fulfilled.

NOTES:

1. Comparison is made without making any adjustments to make the analyzer performance (concentration) match the concentration of the higher standard that is closer to NIST in comparison traceability.
2. Verification should not be confused with calibration of a measuring system, or *vice versa*. Verification of the accuracy of a transfer standard is established by (1) relating the output to a O3 standard of higher authority (level) and (2) demonstrating that the repeatability of the transfer standard is within the performance acceptance limits. Calibration involves the possibility of adjustment, verification does not.

Re-verification: While the principle accuracy of a transfer standard is established during verification, the confidence in that accuracy is maintained by continual re-verification to demonstrate stability. The objective is to show, to the greatest extent possible, that the transfer standard did not change significantly between verification and use.(TAD, 2013)

Traceability: is the "property of a measurement result whereby the result can be related to a stated reference through a documented unbroken chain of calibrations**,** each contributing to the measurement uncertainty" (ISO). (TAD, 2013)

3.2 Acronyms and Abbreviations

AC-	Alternating Current
BIPM-	Bureau International des Poids et Méasures
CFR -	Code of Federal Regulations
CHGMI-	Czech Hydro-meteorological Institute, Prague, Czech Republic
DC	Direct Current
DIO	Digital Input /Output
DOS	Disk Operating System

e-CFR-	electronic version of the Code of Federal Regulations
EPA-	United States Environmental Protection Agency
Mb-	millibar
mV-	millivolt(s 1000 mV = 1.0 Volts Direct Current (DC)
MFC-	Mass Flow Controller
NIST-	National Institute of Standards and Technology
NRMRL-	National Risk Management and Research Laboratories
O_2-	Oxygen
O_3 -	Ozone
OAQPS-	Office of Air Quality Planning and Standards
OD-	Outer Diameter
ORD-	Office of Research and Development
PC-	personal computer
PCI-	Peripheral Component Interface
PID	Proportional-Integral-Derivative (Controller)
PPM-	parts per million
QA/QC-	Quality Assurance/Quality Control
RTD-	Resistance Temperature Detectors
RTP -	Research Triangle Park, NC
SLPM-	Standard Liters per minute (?)
SOP-	Standard Operation Procedure
SRM-	Standard Reference Material
SRP-	Standard Reference Photometer
STOLAB-	Stow Laboratories, 7 Kane Industrial Drive, Hudson, MA 01749
TAD-	Technical Assistance Document
USB-	Universal Serial Bus
UV-	UltraViolet
VAC-	Volts Alternating Current
V/f -	Voltage to Frequency (Converter)

4. Health and Safety Warnings:

- Because ozone is regulated because it can cause health effects, the ozone generated in the pursuit of this procedure must be vented in a way that does not jeopardize anyone's health.
- Since the SRP has a large glass cell, care must be taken in handling and, especially when packing and unpacking the cell for the traveling SRP.
- Since UV light is the basis of detection, and must be generated (by a UV lamp) for the method to work, care should be taken to protect the user from any negative effects of UV light.
- There may be, at times, areas of significant voltage in the electronics module, so no water should be used near that module when it is in use. The power should be turned off if any repairs are attempted. And any such efforts should proceed with caution, in case any static charge has built up.

5. Cautions:

Since there are important functions carried out by the pneumatic and electronic modules of the SRP, care should be taken to minimize any over pressure in the system, when reassembling the traveling SRP for use, and then turning on; and to minimize stress to the circuit board connections. Be sure to pack the glass cell(s) of the traveling SRP carefully.

6. Interferences:

There can be interferences from other molecules of gasses that also absorb UV light in the wave lengths that ozone absorbs at. This, in particular, includes certain Volatile Organic Carbon molecules (VOCs), and, to some extent, NO2. However, the SRPs are provided zero air, or zero air with ozone only and so these other gasses are not a source of error for SRP vs SRP, or SRP vs commercial ozone analyzer comparisons for which this SOP is used, unless the zero air is not adequately scrubbed.

7. Personnel Qualifications:

SRP operation to perform the CFR required verification of State local, tribal, and CASTNET primary ozone standards will be accomplished by an SRP operator that has been through the step by step hands-on SRP Training provided by EPA RTP SRP technical lead. Personnel should have experience calibrating ozone analyzers, including providing of zero air, and independent generation of ozone.

8. Equipment and Supplies

Each of the 8 Regions with an SRP:

SRP: 3 modules (Glass Cell, Cell Stand, Pressure Module, Electronic Module); Glass Manifold, and connecting cables, and dedicated desktop computer, with latest SRP software; Shipping cases (1for each module; and one for miscellaneous items).

Zero Air Supply: Either is a commercial unit, or assembled by SRP lab operator, or is a combination of both. Normally contains a pump, air storage/ballast tank, pressure regulator and gauge, adsorbent scrubbers, particulate removing filter (as needed). The scrubbers, or equivalent, must remove constituents that might absorb UV in the wavelength(s) that ozone absorbs; in particular, some Volatile Organic compounds (VOCs), N2 and O2. Typical scrubber materials include activated (heat-treated) charcoal, Mole sieve, etc. Scrubbing materials may need to be replaced, or, in the case of the heat treated (activated) charcoal, the materials may need to periodically be re-heat-treated. The performance of the zero air supply can be checked periodically, or if a problem is suspected, by comparing the “zero” air it provides against the output of an ultra pure air cylinder from a trusted and respected vendor who provides a certificate of analysis that shows any impurities that independent analyses found were still in the cylinder, and at what amount.

Power Supply: NIST recommends a power line buffer, so that it provides stable voltage frequency, so that there are no unwanted effects on the performance of the electronics module.

SRP Support Lab Equipment: NIST traceable Temperature, Pressure, STOLab calibrator and Voltage sensor used to independently check, and, as needed, recalibrate the pressure and temperature sensors that are in each of the NIST-made EPA SRPs; as well as to check the many different voltages that have to be correct throughout the electrical circuitry of the electronics module. This equipment needs to be equal to or greater than the acceptance criteria in section 9 and verified against the NIST traceable standards annually.

9.0 Ozone SRP to SRP or Standard Comparison Procedure

9.1 Pre-SRP Comparison Considerations

9.1.1 Qualification

Qualifications are rarely ever run any more. Years ago, new O_3 analyzers were put out in the market with no type of qualification being run on them. These days the manufacturer will run any new instrument against a Lab Standard that is maintained at the factory, and this may be considered a Qualification. However, when a request for a Qualification is made, here are some operational parameters that should be considered:

SRP vs. a Candidate to be used as Level 2 Transfer Standard (TAD Page 2-5 ¶1)

Qualification: Once, new or after major repair (TAD Page 3-2, ¶1, Figure 3-1)

Range: The Qualification should be testing the full range of the instrument. However, this range is not fully defined. In the TAD (Page 1-4 ¶1), there is a reference in the CFR-50 that refer to the full range of the instrument. **Pass/Fail criteria for a Qualification: Slope 1±.04 and Intercept 0 ppb ±4 ppb; see TAD Page 1-4 ¶1)**

- Instrument Stability Factor: 0.7
- Data Quality Factor: 0.7
- Number of Concentration Points (not including Zero): minimum of 6
- Number of Concentration Points (including Zero at start and end): minimum of 8
- Points per Concentration: Average of at least 7 points.
- Number of Cycles: Minimum of one cycle per day for 6 days under various conditions (see TAD Appendix B) Temp/Pressure/Line Voltage. It may be noted that running a Qualification under varius Room Temperatures, Pressures and various Line Voltages may

be very difficult or even impractical in today's laboratories. It should also be noted that after passing the Qualification, the instrument should now have a Verification performed on it.

9.1.2 Verification

The Verification is defined in the TAD 1-6 ¶4, 3-2 ¶2. This definition is often referred to as a 6 by 6. For any new Level 2 Transfer Standard, this Verification is mandatory.

- Level 2 SRP vs. Qualified Instrument to be used as Level 2 Transfer Standard (TAD Page 2-5 ¶1)
- Verification (6x6): Once after qualification, new or after major repair (TAD Page 3-2, ¶1)
- Range: (90 % ± 5) % of the upper range limit (TAD Section 4.2) RSD of six slopes 3.7 % Std. Dev. of six intercepts 1.5, TAD Fig. 3-1) and a Slope of 1 ± 3.0 % and an intercept of 0 ± 3 ppb. RSD = (Standard Deviation of six slopes)/(Average of six slopes)*100
- Setup Parameters:
- Instrument Stability Factor: 0.7
- Data Quality Factor: 0.7
- Number of Concentration Points (not including Zero): 6 minimum
- Number of Concentration Points (including Zero at start and end): 8 minimum
- Points per Concentration: 7 minimum
- Number of Cycles: 6 (1 (minimum) each on 6 different days TAD Section 4.2)

When running a Verification, the automated feature of the SRP Program can be very useful. If a Verification is started on a Thursday and allowed to run over the weekend, then the Verification could be completed by Tuesday morning. A couple of tricks could be used to extend the run time of each cycle. Even though only one per day is required, more can be run. The cycle can be set up to run more than 6 concentration points. If the cycle is increased to 12 or 14 concentration points with 12 points per concentration, each cycle may take up to three hours. The other trick that can be used to extend the cycle time is in the calibration file. When a calibration is set up, you are prompted to save it. Save the calibration (as an example) as A.met, and save it again as B.met and save it again as C.met. Now open B.met and in the lower right hand corner there is a box that is labeled "Link Method". Click on the down arrow and find the C.met and save the method (or calibration). Now do the same for the A.met, except under "Link

Method" find and select the B.met. What will happen is that the first calibration (cycle) will run until it has completed and then the second (B.met) will run until it is completed and then finally the third calibration will run (C.met) and when it is completed, it will go into "Standby".

9.1.3. Re-Verification

The Re-Verification is the procedure used most often by all the SRP Operators.

A Re-Verification can be run on a Level 2 Standard as long as there have been no major repairs to the instrument. Major repairs would include replacement of the cell or cells, a mother board, a pressure transducer or a temperature probe. Any annual maintenance, cleaning, replacement of a pump or of a lamp would not be considered a "Major Repair" if the instrument readings return to normal.

The Re-Verification can also be an SRP Level 1 vs. an SRP Level 1. The NIST comparison against an EPA SRP is essentially Level 1 as there is no higher authority currently available for Ozone Analysis. NIST will refer to the Re-Verification as a "Report of Calibration". See NIST Verification 2014.pdf. Our goal is to run our Re-Verifications of SRP to SRP in as close a pattern as NIST would run. Typically, NIST will run 40 to 50 cycles over a three- to four-day period. NIST will run concentrations from zero to 1000 ppb and may include a set of zero to 500 ppb. BIPM typically will run zero to 500 ppb. Running comparisons from zero to 500 ppb, as NIST and BIPM do, will aid us in being comparable to the worldwide network of SRPs.

The following is a list of the recommended parameters that a Re-Verification should be set for the Guest Level 2 Standard along with the location of the reference in the TAD.
http://www.epa.gov/ttn/amtic/files/ambient/qaqc/OzoneTransferStandardGuidance.pdf

SRP vs. Level 2 Transfer/Lab Standard

- Re-Verification: Annual (TAD Page 1-4, Paragraph 1)
- Range: Full Range of Instrument (Slope 1 ±. 03 and Intercept 0 ppb ± 3 ppb; see TAD Page 1-4 ¶1)
- Instrument Stability Factor: 0.7
- Data Quality Factor: 0.7

- Number of Concentration Points (not including Zero): 6
- Number of Concentration Points (including Zero at start and end): 8
- Points per Concentration: 7
- Number of Cycles: 3 (all can be completed on the same day)

All instruments should be started at an Instrument Stability Factor and a Data Quality Factor of 0.7 each. If it is necessary to bump up these factors due to instability in the Guest Standard, the factors can be increased, but a note of why this increase was needed must be entered into the "Comment" section of the calibration file. NOTE: If the SRP cannot achieve these factors of 0.7, the SRP then is in need of repair and should not be used until corrected.

A summary report if the level 2 verification will be provided to the monitoring organizations.

9.1.4. Audit Level Comparisons

Taken from 40 CFR Part 58, Appendix A

Audit level	Concentration range, ppm			
	O_3	SO_2	NO_2	CO
1	0.02-0.05	0.0003-0.005	0.0002-0.002	0.08-0.10
2	0.06-0.10	0.006-0.01	0.003-0.005	0.50-1.00
3	0.11-0.20	0.02-0.10	0.006-0.10	1.50-4.00
4	0.21-0.30	0.11-0.40	0.11-0.30	5-15
5	0.31-0.90	0.41-0.90	0.31-0.60	20-50

Some of our EPA auditors have voiced concern about these Audit Level Comparisons and especially concerns over the impending new ten Point Audit Level Comparisons on the horizon. These auditors want to use these points in the chart when they run a Re-Verification against an SRP. The O_3 Generation Lamp in the SRP will "fire" at a certain level of Lamp Power, usually between 10 % and 20 %. The SRP Operator should be aware of where that point is and then adjust the flow to achieve the 20 ppb to 50 ppb point. The SRP Operator can adjust the Lamp Power up to find the 900 ppb point and use the high and low settings for the calibration. Each calibration point can be determined in that manner, or the number of concentration points can be increased so that the SRP will automatically hit on or near the points in the chart.

9.1.5 Calibrations for Scientific Research

On very rare occasions, the SRP Operator may receive requests to perform an O_3 calibration for scientific research. It is entirely up to the operator to decide to perform this calibration. The SRP Operator should never allow a commercial instrument to feed O_3 to the SRP to avoid any type of cross contamination. Any of the Pass/Fail criteria would be based upon the QA Project Plan provided to the SRP Operator.

An organization may request that the SRP operator calibrate their commercial analyzer. If a research organization here at RTP requests the calibration of a commercial analyzer, the RTP SRP operator may do the calibration. If asked for a recommendation, a copy of this SOP may be provided.

9.1.6 Zero – Span Check

Calibration for scientific research is actually a subject that needs to be approached very carefully. It is not the intent of this program to fix or "tweak" Guest instruments to work better. The intent is a verification and that is all. Again, it is entirely up the SRP operator to run a Zero and Span. If an Operator should decide to do this, it would be best if the owner of the Guest instrument be present and let the owner do the adjustment to their own instrument. If the owner wants you to make the adjustment and you are willing to do so, you should get their permission in writing first. In either case, you should record what the Slope and Intercept were before and after the Zero-Span Check.

Additionally, sometimes a Guest Instrument will arrive at the Region, and it is not working. The SRP Operator is not responsible for any repairs to any of these instruments. However, some of the SRP Operators are very sensitive to the time and travel that may be involved in getting a Guest to the Region. Again, it is entirely up the SRP Operator to decide if the repair can be done easily and quickly in house. If the SRP Operator is willing to do the repair, you should get the permission of the owner in writing first. Extreme caution should also be used in making repairs: the more the Operator does, the more individuals will come to rely on the Operator doing these things. The Guest owner should be reminded that, before they bring in the Guest, it would be a good time to do the annual maintenance on it. They should also run the Guest and make sure it is fully operational before it leaves their laboratory.

9.1.7 Running Multiple Guests at One Time

Each of the Regional SRPs has the capability of running up to three different Guests at a time. Most of the computers have only two serial ports to communicate to three serial devices. A USB to Serial adapter can be purchased and installed on the computer, and this adapter works very well. Analog input may be an option that will not be supported in the future, but an analog-to-digital volt meter can be purchased, and a driver has been written for the SRP to communicate with it. For further details, please contact your program coordinator.

9.2 Procedure: SRP-07 INITIAL SETUP

Note: All the steps required for setting up and comparing SRP 7 (as the Host) to a Regional SRP (as the Guest) start in Subsection 9.2.1 and continue through subsection 9.4.2.

9.2.1 Receiving and Inspection

When the SRP-07 arrives from EPA in Research Triangle Park (RTP) the following items will in the shipping containers:

Contents:

- Optical Bench
- Pneumatics Module
- Electronics Module
- DB37 pin male to female cable
- Detector Cable and Scalar 1 and 2 cables.
- J1 Cable
- Power Cable
- Sample Teflon line
- Reference Teflon line

Inspect each of the shipping containers carefully for any obvious damage during shipping. If there is significant damage to any of the cases, please take a picture of the damage and contact the program coordinator as soon as possible. Four different types of cases are now being used. Some of the Regions received a set of two Silver A&J cases. These cases are old but some of them are still in use. There is a set of Blue A&J cases that hold the Optical Bench, Pneumatics Module and the Electronics Modules separately. For shipping a Regional SRP to the Program Coordinator for Re-Verification or repairs, either of these cases would be preferred. SRP-07 will usually be shipped in a set of three Starlite cases that were purchased and cut specifically for SRP-07. A newer set of cases has been purchased that consist of one Starlite case for the Optical Bench and two Pelican cases for the two modules. The program is not endorsing any one case over the other but just documenting what is being used for shipping the SRPs around the country.

9.2.2 Unpacking the Electronics Module

Carefully remove the Electronics Module from the shipping container. Place the Electronics Module on the bench top 25” to 30” away from the Glass Manifold that the Regional SRP is using. Please note which container the Electronics Module is in because the foam is cut specifically for this module. Open the top of the Electronics Module and check for anything that may have broken loose. Check the power supplies inside the Module to make sure nothing came loose during shipping. See also section 9.5.2.5 for the STOLAB Card Temperature Board verification.

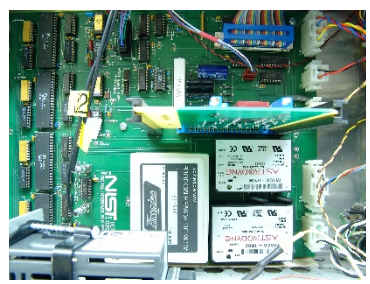
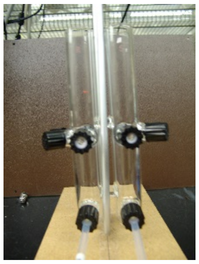


Figure 9.2.2.1 Electronics Module front ***Figure 9.2.2.2 Electronics Module back*** ***Figure 9.2.2.3 Electronics Module inside***

9.2.3 Unpacking the Pneumatics Module

Carefully remove the Pneumatics Module from the shipping container and place it on the bench top, leaving approximately 14 to 16 inches between the two modules. The Pneumatics Module should be placed between the Electronics Module and the Glass Manifold. Please note which container the Pneumatics Module is in because the foam is cut specifically for this module. Place the Module as close to the Regional SRP manifold as possible. Open the top of the

Pneumatics Module and inspect the inside for any power supplies that may have come loose. Report any damage to the program coordinator immediately.



14” to 16”


or

Figure 9.2.3.1 Orientation of Manifold (on left), Pneumatics and Electronics Module

14” to 16”

Figure 9.2.3.2 Orientation of Pneumatics and Electronics Module and Manifold (on right),

9.2.4 Orientation of the Electronics and the Pneumatics Modules

Orientation of the two modules will not be critical. However, the two Pneumatics Modules (SRP-07 and the Regional SRP) will need to be as close to the Regional Manifold as possible to avoid any crimping in the Teflon tubing. The most common setup for a Regional SRP is with the Pneumatics Module on the left and the Electronics Module on the right with the Optical Bench sitting over them. If this is the case, then place the SRP-07 Pneumatics Module to the left of the Regional Manifold as shown in Figure 9.2.4.1 and Figure 9.2.4.2, below.

Figure 9.2.4.1 Regional Pneumatics Module

Figure 9.2.4.2 Regional Glass Manifold

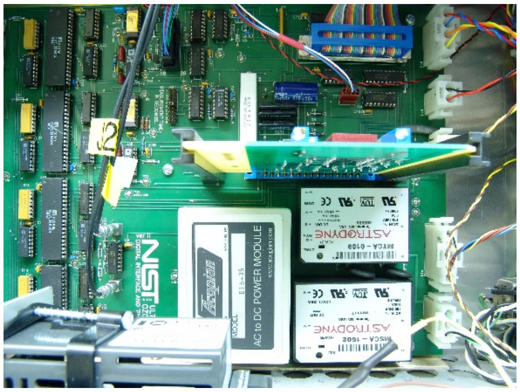
Figure 9.2.4.3 Regional Glass Manifold

Figure 9.2.4.4 SRP-07

Orientation is not limited to just the right and the left. Some Regions have enough counter space to place the SRPs back to back. Some Regions have multilevel counter space to work with. Any orientation is acceptable but the key element is to get the Pneumatics Module within one meter of the Regional Manifold.

9.2.5 Electronics Module Internal Inspection

Open the top of the Electronics Module and inspect to see if there was any damage during shipping. One of the most common problems with shipping the SRP is that the Temperature card will become loose and will need to be re-set (see 9.2). There have been occasions where the power supplies on the mother board have come loose and bounced around inside the modules during shipping. SPR-07 will now be sent with protective packing inside the module, and this protective packing will need to be removed before power is supplied to the instrument. This extra packing will be for the protection of the instrument, and there will be a note attached to the cover indicating that extra packing will need to be removed. The Watlow Temperature Controller should be firmly mounted to the side of the module.



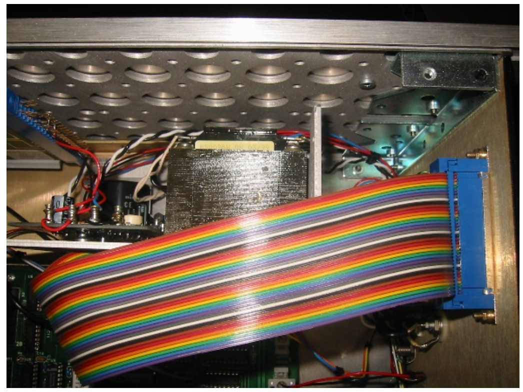


Figure 9.2.5.1 Inside the Electronics Module ***Figure 9.2.5.2 Ozone Boards power supply***

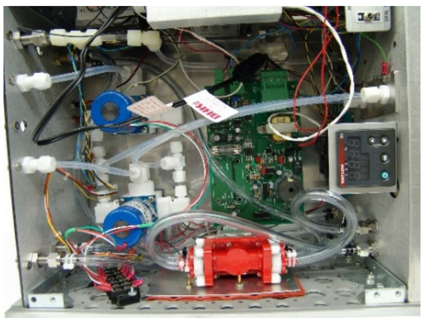
9.2.6 STOLAB Temperature Board Inspection

The STOLAB Temperature Board needs to be checked to ensure that it is seated properly into the mother board. This board has a tendency to pop loose during shipping, and care needs to be taken to check that it is properly seated into the edge connector (see Figure 9.2.6.2) Gently push down on the top of the STOLAB Card and check that it clicks back into place. If unsure, the card can be pulled out and re-inserted.

Figure 9.2.6.1 STOLAB Card/ Figure 9.2.6.2 STOLAB Location on MB/ Figure 9.2.6.3 STOLAB inserted

9.2.7 Pneumatics Module Inspection

Open the top of the pneumatics module and inspect to see if there was any damage during shipping. Also remove any packing materials that may have been put inside the module. Please ensure that any power supplies and the Watlow temperature controller are well secured inside the module. EPA is in the process of upgrading the Pneumatics Module. The new solenoid valves have a larger internal orifice. The Mass Flow Controller (MFC) has been separated from the heated block and the O_3 generator has been moved to allow easy access to the lamp.



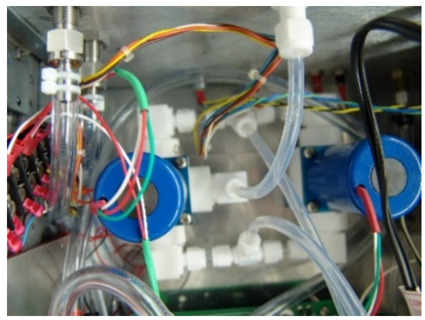


Figure 9.2.7.1 Inside the Pneumatics Module /Figure 9.2.7.2 New style of solenoid valves

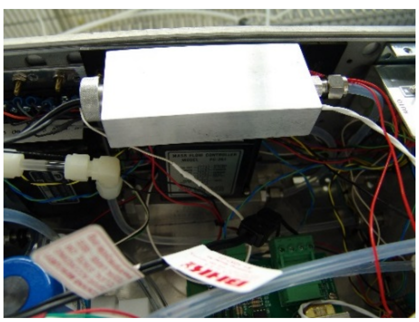


Figure 9.2.7.3 heated block and MFC

9.2.8 Unpack Miscellaneous Items

Remove all miscellaneous items from the shipping containers and lay out on a table so specific items can be found easily. The following items should have been shipped with SRP-07:

1) 1 Detector Cable
2) 2 Scalar Cables
3) 2 J1 Cable
4) 1 Power Cord
5) 1 DB-37 pin communication cable.
6) Two ¼” Outer Diameter (OD) Teflon tubing (1.0 meters in length) {not shown below}

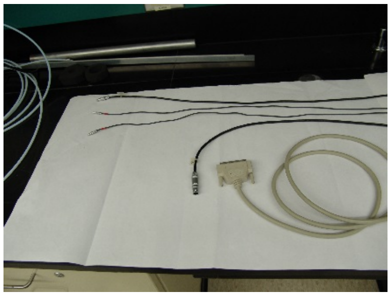
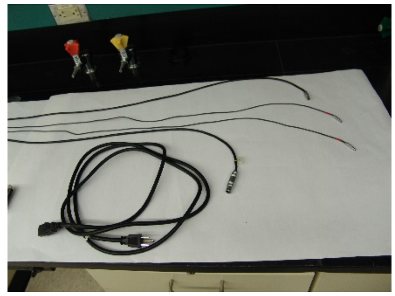



Figure 9.2.8.1 DB-37 Pin Cable / Figure 9.2.8.2 Power Cord

9.2.9 Unpack Optical Bench

Carefully remove the Optical Bench from the shipping container and place upside down (use the NIST nameplate for orientation) across the Electronics and Pneumatics Modules. Inspect the bottom of the Optical Bench (as seen in Figure 9.2.9.1 below) for any loose fittings and any crimped tubing and repair as needed. The biggest concern would be the glass cells inside the Optical Bench. If the glass cells have been broken during shipping please notify the Program Coordinator as soon as possible

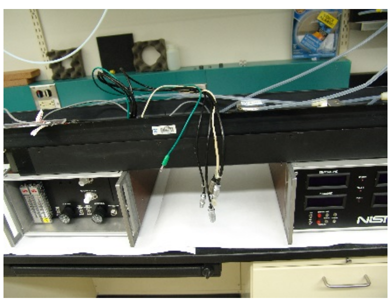


Figure 9.2.9.1

The Optical Bench will arrive with a “Spacer” (see Figures 9.2.9.2- 9.2.9.4) that has been placed between the Heated Block and the body of the bench. This “Spacer” has been placed there to help minimize any stress to the block during shipping. On a few occasions, the block has cracked and the screws that hold it all together have been bent during shipping. Please remove the “Spacer” before powering up the SRP.

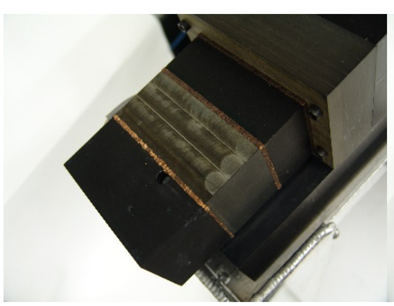
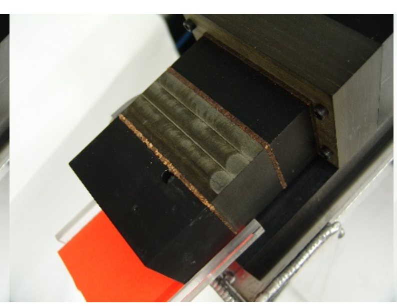
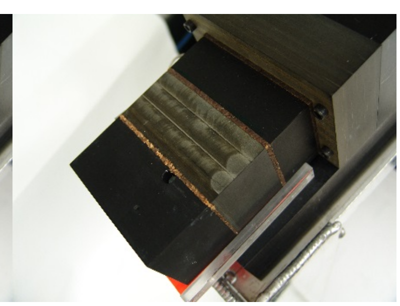




Figure 9.2.9.2 No Spacer /Figure 9.2.9.3 Spacer half inserted / Figure 9.2.9.4 Spacer Fully Inserted

9.2.10 Orientation of the Optical Bench

Normally the Optical Bench will be placed on top (or over) the two Modules with the Heated Block (Figure 9.2.10.1) located to the left and the Detector (Figure 9.2.10.2) to the right.

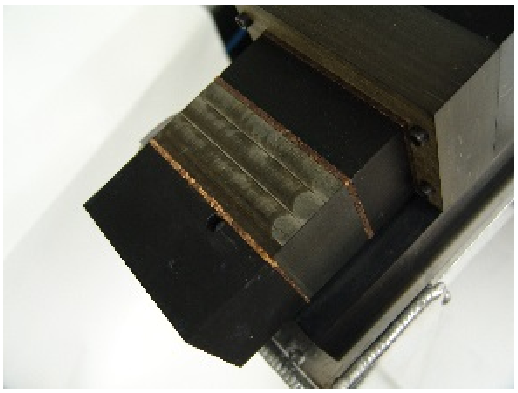
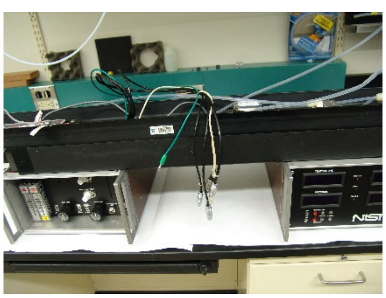


Figure 9.2.10.1 Heated Block and UV Source

Figure 9.2.10.2 Detector

Initially the Optical Bench can be placed upside down on top of the Modules (see Figure 9.2.10.3) so that the cables and the ¼” tubing can be easily inspected.



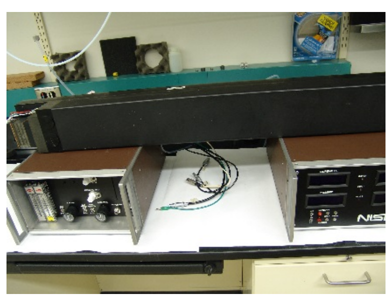


Figure 9.2.10.3 Bench Upside Down/ Figure 9.2.10.4 Rolling Over Bench / Figure 9.2.10.5 Correct Position

Carefully roll the Optical Bench over (see Figure 9.2.10.4) and then to the right side up (see Figure 9.2.10.5) position, allowing the cables and lines to fall between the electronics and pneumatics modules. It is possible, but not recommended, to run the cell lying down on its side (see Figure 9.2.10.4). Care needs to be taken not to bend the tubing too much and to place some form of vibration dampening between the

Optical Bench and the two modules. Some of the regions have a special stand for SRP-07 to use during the Re-Verification.

Optical Bench Stand: Several types of Bench Stands are in use. EPA Las Vegas had built and distributed several Bench Stands made of light aluminum. These Bench Stands are light weight and can be broken down for shipping, but new Bench Stands are no longer available. EPA RTP had built a few stands that are very solid but cannot be broken down. NIST has designed a very nice stand that can be built by any machine shop. The NIST stand is light weight, strong and can be broken down easily and can also be purchased from NIST. However the Optical Bench will be set up, care should be taken to buffer any vibration between the Optical Bench and the two Modules.

9.3 Connecting the Electronic Signal Cables to SRP-07

9.3.1 Connecting the J1 Cable to the Electronics Module

No power should be supplied to SRP-07 at this time.

All of the following steps are for setting up SRP-07 as the "Host" SRP. Initially, J1, J2, J3, Scalar Cables, Temperature and Pressure are interconnected between the SRP-07 Optical Bench, Electronics Module, Pneumatics Module and the Regional SRP Computer. The following plugs are "Lemo Connectors ©" The J1 is Part Number ERA 2S 308 CLL.

Figure 9.3.1.1 No Connections

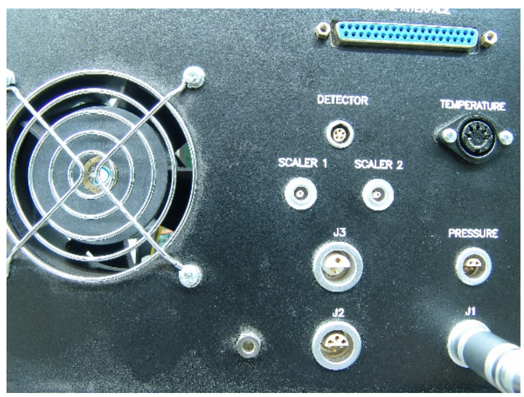


Figure 9.3.1.2 J1 Connected

The J1 is an eight-pin connector and will fit in only one direction. Please note that on the Module, the top half of the receptacle is flush with the surface of the body, and there are female connectors and the bottom half is sunk below the surface and there are male connectors. The J1 plug will need to be aligned opposite from the plug so that it will be male to female on top and female to male on the bottom. Do not try to force the plug into the socket. If the plug does not go directly into place, try to rotate the plug to the left or right until it falls into place. The plug will then snap into place, and there will be a secure connection.

9.3.2 Connect the J2 Cable to SRP-07 Electronics Module

The following plugs are "Lemo Connectors ©" The J2 is Part Number ERA 2S 306 CLL.

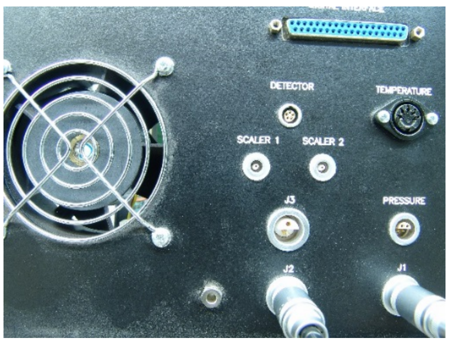


Figure 9.3.2.1 J2 Cable plugged in.

The J2 is a six-pin connector and will fit in only one direction. Please note that on the Module the top half of the receptacle is flush with the surface of the body and there are female connectors, and the bottom half is sunk below the surface and there are male connectors. The J2 plug will need to be aligned opposite from the plug so that it will be male to female on top and female to male on the bottom. Do not try to force the plug into the socket. If the plug does not go directly into place, try to rotate the plug to the left or right until it falls into place. The plug will then snap into place, and there will be a secure connection.

9.3.3 Connect the J3 Cable to SRP-07 Electronics Module

The following plugs are "Lemo Connectors ©" The J3 is Part Number ERA 2S 302 CLL.

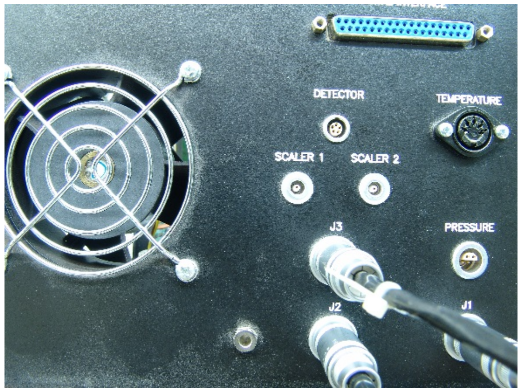


Figure 9.3.3.1 The J3 Plug

The J3 is a two-pin connector and will fit in only one direction. Please note that on the Module, the top half of the receptacle is flush with the surface of the body and there are female connectors, and the bottom half is sunk below the surface and there are male connectors. The J3 plug will need to be aligned opposite from the plug so that it will be male to female on top and female to male on the bottom. Do not try to force the plug into the socket. If the plug does not go directly into place try to rotate the plug to the left or right until it falls into place. The plug will then snap into place, and there will be a secure connection. The other end of the J3 will plug into the Pneumatics Module as it supplies power to that module.

9.3.4 The J4 Cable from the Regional SRP

Normally the Regional SRP that is getting Re-Verified should generate O_3 to help the Program Coordinator to understand how well the Regional SRP is operating over the full range of O_3. The Regional SRP will have a Blue Analog Input Box (this box is not shipped with the SRP-07) and a J4 cable (also not shipped with SRP-07). This J4 Cable will be plugged into Regional's Blue Analog Input Box and will remain plugged into the Regional SRP. There is no need for the Region to request the Blue Analog Input Box or the J4 cable from the Program Coordinator. The controls for the MFC and the O_3 Generator Lamp are carried over the J4 cable. During the Re-Verification, SRP-07 will be in control of the SRP Software as the Host, but the Region SRP will be generating O_3 for the Re-Verification. The LEMO Part number for the J4 connector is ERA 1S 304 CLL.

9.3.5 Connect Detector Cables to SRP-07 Electronics Module

From the items that were set to the side while unpacking SRP-07, find the Detector Cable, the Scalar 1 and Scalar 2 cables. Connect, the "Scalar 1" (Fig 9.3.5.1) and the "Scalar 2" (Fig 9.3.5.2) and the "Detector" cable (Fig 9.3.5.3) into the corresponding plugs on the rear of the electronics module. Then plug the other ends of these cables into the corresponding sockets on the SRP-07 Detector Module mounted on the right hand side of the Optical Bench.

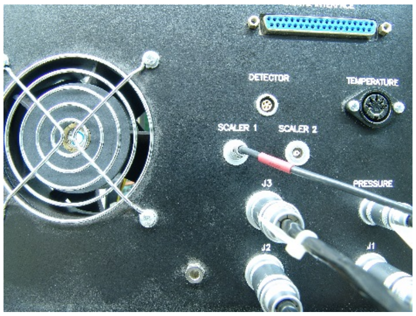


Figure 9.3.5.1

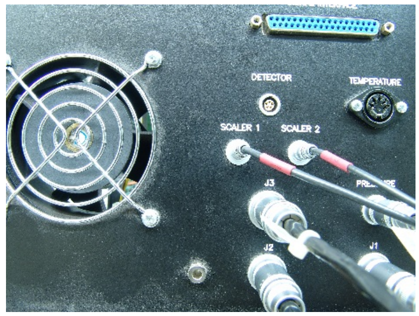


Figure 9.3.5.2

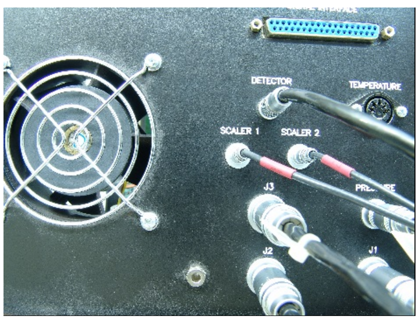


Figure 9.3.5.3

9.3.6 Setting up SRP-07 as the Host SRP

Setting up SRP-07 as the Host SRP is the preferred method for conducting a Re-Verification on a Regional SRP. In this setup, the Regional SRP will be compared to SRP-07, and in the spreadsheet that is generated from the software, the Dependent and Independent Ranges for calculating the Slope and Intercept will be in harmony with the rest of the program. From a statistical point of view, setting up SRP -07 as the Host and the Regional SRP as the Guest will make it easier to follow the traceability throughout the entire SRP program.

All power to the computer, SRP-07 and the Regional SRP should be off during this step. Trying to hot swap the communication cables can cause severe damage to all of the equipment. Remove the DB-37 Pin cable from the back of the Regional SRP Electronics Module and connect it to the corresponding connector on the back of the SRP-07 Electronic Module. The DP-37 (Fig 9.3.6.1 upper right hand corner) pin cable that was shipped with SRP-07 should now be plugged into the back of the Regional SRP, and then the other end will be connected to the DB-37 pin connection on the back of the Regional Computer.

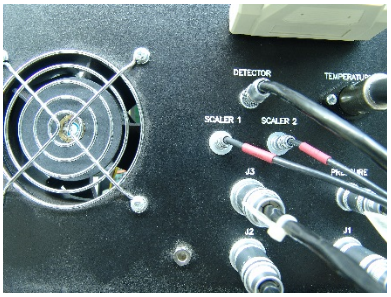
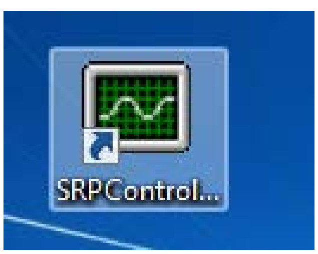


Figure 9.3.6.1 DB-37 pin (upper right)

Connect Power Cable to rear of Electronics Module (100-120 VAC required) to a power strip that has a breaker in it. Use the power strip to power the SRP on and off. Now power up the two SRPs and the computer.

Once the computer has booted up, start the SRP Software by double-clicking the SRPControl icon (Fig 9.3.6.2) on the desktop or single click the SRPControl icon on the windows menu bar. The SRPControl.exe file can be found in C:\Program Files\SRP2002. On newer computer models, the SRPControl.exe file may be found at C:\Program Files (x89)\SRP2002.



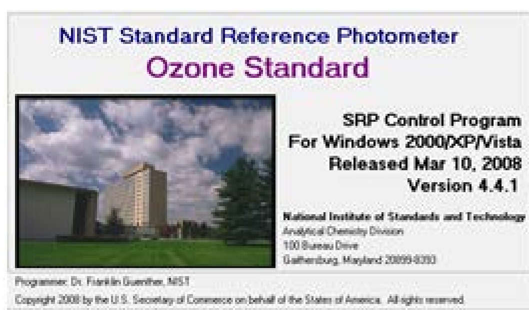


Figure 9.3.6.2 Desktop Icon ***Figure 9.3.6.3 SRP Software Opening Screen***

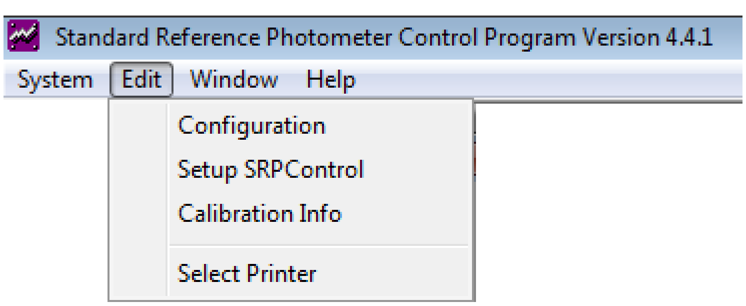


Figure 9.3.6.4. SRP Software Opening Screen (click on configuration)

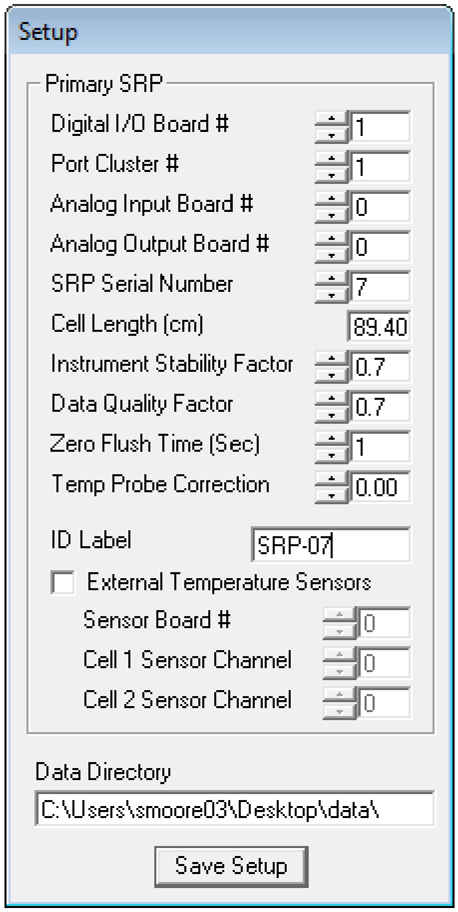


Figure 9.3.6.5 Configuration Setting for Host SRP

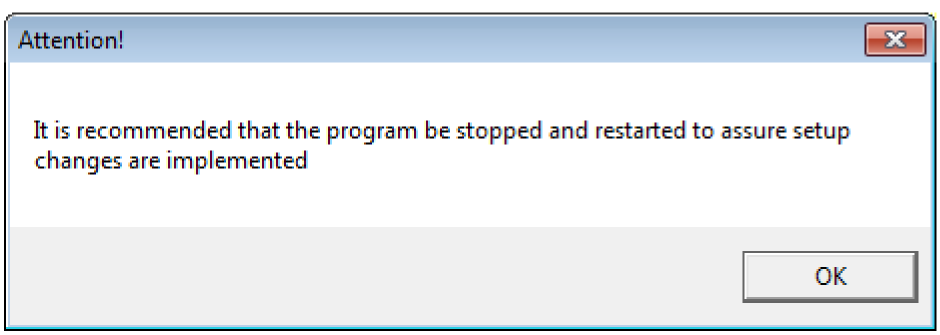


Figure 9.3.6.6

9.3.7 Setting up SRP-07 as a Guest

Setting up SRP-07 as the Guest SRP is strongly discouraged for conducting a Re-Verification on a Regional SRP. In this setup, the Regional SRP will be compared to SRP-07 and in the spreadsheet that is generated from the software the Dependent and Independent Ranges for calculating the Slope and Intercept will be in reverse to the rest of the program. From a statistical point of view, setting up SRP-07 as the "Host" and the Regional SRP as the "Guest" will make it easier to follow the traceability throughout the entire SRP program.

All power to the computer, SRP-07 and the Regional SRP should be off during this step. Trying to hot swap the communication cables can cause severe damage to all of the equipment. Remove the DB-37 Pin cable from the back of the Regional SRP Electronics Module and connect the DB-37 Pin cable to the corresponding connector on the back of SRP-07 Electronic Module. The DP-37 pin cable that was shipped with SRP-07 should now be plugged into the back of the Regional SRP, and then the other end will be connected to the DB-37 pin connection on the back of the Regional Computer.

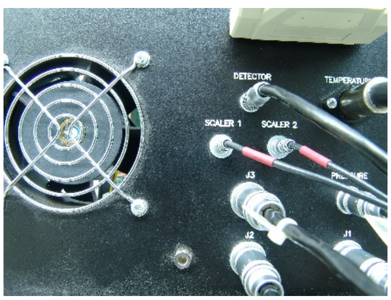


Figure 9.3.7.1

Connect Power Cable to rear of Electronics Module (100-120 VAC required) to a power strip that has a breaker in it. Use the power strip to power the SRP on and off. Now power up the two SRPs and the computer.

Once the computer has booted up, start the SRP Software by double-clicking the SRPControl icon on the desktop or single click the SRPControl icon on the windows menu bar. The SRPControl.exe file can be found in C:\Program Files\SRP2002. On newer computer models, it may be found at C:\Program Files (x89)\SRP2002.

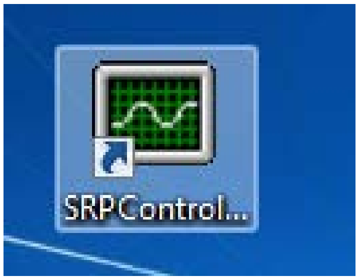


Figure 9.3.7.2 Desktop Icon ***Figure 9.3.7.3 SRP Software Opening Screen***

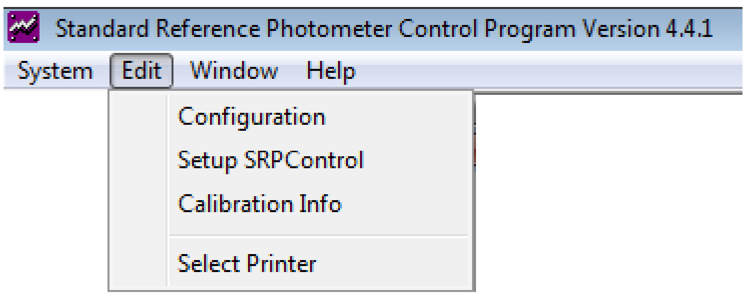


Figure 9.3.7.4. SRP Software Opening Screen Select Configuration

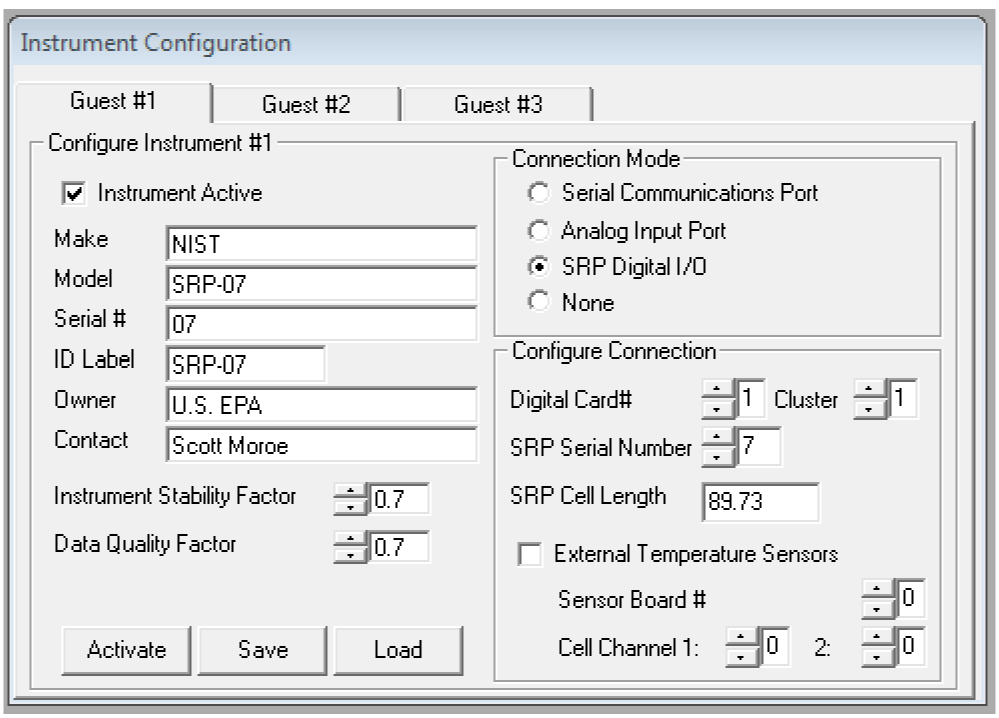


Figure 9.3.7.5 Set SRP-07 as Guest1

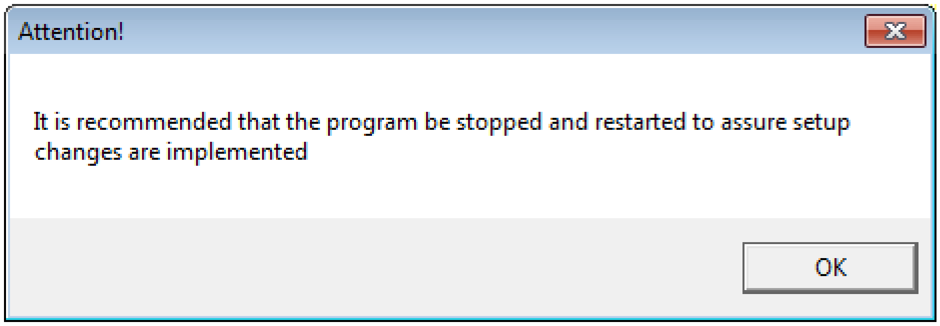


Figure 9.3.7.6

9.3.8 Connect Power Cable to the SRP

Connect Power Cable to rear of Electronics Module (100-120 VAC required) to a power strip that has a breaker in it. Use the power strip to power the SRP on and off.

9.3.9 Initial Startup

Ensure that the three switches on the front of the Pneumatics Module are in the "off" position. At this point, you can power on the NIST SRP. The front panel displays will show all or mostly E's (example, EEEEEE). You may hear a high frequency sound coming from the Electronics Module for at least one minute, and then the sound should decrease. The sound is caused by the PCI 2400 UV lamp power supply, which has a one minute high power start mode.

Figure 9.3.3.9 SRP Electronics module (initial start-up)

9.4 Pneumatics Connection Setup

9.4.1. Remove Caps and Plugs

Remove the caps and plugs from the Teflon tubing and the pneumatics module and save for re-packing.

9.4.2 Connecting the Teflon Lines

Connect the Teflon lines labeled “To 1" and “To 2" to the Teflon bulkhead connectors on the rear of the Pneumatics Module labeled “To Cell 1" and “To Cell 2".

For the Teflon lines that are connected to stainless steel Swagelok fittings, ensure they are hand-tightened plus a quarter inch turn with a wrench. For the Teflon lines that are connected to the Teflon Swagelok fittings, ensure they are hand-tightened. (Be careful not to strip the threads of the Teflon fittings.)

9.4.3 Identifying the Correct Cell Lines

The cable tie markers attached to the Teflon lines sometimes break off. When one of the regional SRPs comes to RTP for repair or maintenance, the external Teflon tubing will be replaced and a label will be put on the end of the tubing to identify where it will be connected. If the labels should fall off, the operator will need to identify where the proper connections are. First, connect any tubing that is properly identified and through the process of elimination, the others may fall into place. If some or all of the markings have fallen off, the operator will need to identify the Cell 1 and Cell 2 lines. As you look at the Optical Bench, the Temperature Block and UV Source should be to your left and the detector to your right. The Scalars are matched with the Cells, that is, Scalar one is Cell one and Scalar 2 is Cell 2. Underneath the Cell Block to your left, there are two Teflon lines coming out. The “To Cell One” is towards the front, closest to you, and then “To Cell Two” is towards the back. Go ahead and connect “To Cell One” to the appropriate Teflon bulkhead fitting on the back of the back of the SRP-07 Pneumatics Module.

9.4.4 Connecting the Stainless Steel (SS) Fittings to SRP-07 Pneumatics Module

Connect one of the SS fittings from the cells to the SS bulkhead label 1 on the back of the Pneumatics Module, but only hand tighten. Connect the Sample and Reference lines to the manifold at this time. Now watch the rotameter on the front panel for Cell 1 (the one to the left), and turn the sample pump on for just a moment. If the glass ball in the rotameter jumps, the connection is correct If the glass ball in the rotameter does not jump, then switch to the other SS fitting from the cell and repeat. Rotameter 1 should now work.

9.4.5 Connecting the Teflon Fittings to SRP-07 Pneumatics Module

Connect the Teflon lines labeled “1" and “2" to the stainless steel bulkhead connectors on the rear of the Pneumatics Module labeled “From 1" and “From 2".

9.4.6 Connecting the Zero Air to SRP-07 Pneumatics Module

Connect the laboratory supply of zero air to the stainless steel bulkhead connector labeled “Air In”. The pressure of this line must be kept between 15 and 30 psi. An adequate Zero Air Generator should be able to supply up to 30 liters per minute at 35 psi. Most any brand-named Zero Air generator should work well.

9.4.7 Technical Note on Zero Air Systems

A note should be mentioned here about the commonly used AADCO 737 Zero Air Generator. During the scrubbing process, the AADCO Zero Air Generator will alternately scrub out the O_2 and N_2 and then re-mix them alternately. This process causes the O_3 concentration to alternate between high and then low. Therefore, when generating a high O_3 concentration, you could end up with undesired stability issues. The solution is an optional part from AADCO called a Mixer/Receiver that will remix the O_2 and N_2 at stable concentrations.

9.4.8 Verification Check of Zero Air Source

If problems with your Zero Air are suspected, a check that can be done to verify your Zero air supply would be to buy a commercial tank of Ultra-Pure air and set up a purge line directly to the manifold and replace either the "reference in" or the "Ozone In" and then run some zero readings from the two different sources. If you get an unusually high or low Zero, then there is an issue. If the Zero is 0 ± 0.2 ppb, then the air supply should be adequate. This test should be performed at least once per year.

9.4.9 NIST Recommendation for Zero Air used for an SRP

NIST recommends the zero air generator have a minimum input pressure of 15 psig (34.7 psia, 239.2 kPa) of clean dry air, preferably zero air, containing no significant impurities, having less than 1 ppb total hydrocarbons by volume, and containing 20–21 % oxygen. The NIST SRP has internal devices to control the amount of air required for operation. No external control is necessary. A zero air supply of 20 standard liters/min (sLpm) will be adequate for most verification work. These units are available through commercial suppliers of air monitoring equipment. An SRP draws 2 sLpm into each cell (4 sLpm total), plus some excess is required in the sample and reference manifolds, and additional flow requirements for instruments under calibration.

9.4.10 CFR Statement on Zero Air

CFR 50 Appendix D Section 4.1 defines Zero Air as follows: "The zero air must be free of contaminants which would cause a detectable response from the O_3 analyzer, and it should be free of NO, C_2H_4, and other species which react with O_3. A procedure for generating suitable zero air is given in Reference 9. As shown in Figure 1, the zero air supplied to the photometer cell for the I0 reference measurement must be derived from the same source as the zero air used for generation of the O_3 concentration to be assayed (I measurement). When using the photometer to certify a transfer standard having its own source of O_3, see Reference 8 for guidance on meeting this requirement."

9.4.11 The SRP Glass Manifold

The Dual Glass Manifold is separate from the SRP Pneumatics Module. Care should be taken to locate the Pneumatics Module as close to the Manifold as possible, taking care not to kink any of the Teflon tubing. Then connect the Sample and Reference lines to the Manifold. SRP-07 may be used to generate O_3. Zero Air would only need to be supplied to the SRP that would be generating O_3. To convert flow and O_3 control over to SRP-07, simply unplug the J4 from the host SRP and plug it into the back of the Pneumatics Module on SRP-07. If control does not automatically take place, it will be necessary to restart the SRP Control Software and in some cases, the computer and the SRPs may need to be rebooted. Figure 9.4.11.1 is a schematic of the Dual Glass Manifold. NIST has modified their Manifold to be approximately 1/8" larger inner diameter. In some areas, the #15 SLV Thread may not be available, so another thread size would be acceptable, but all new Solid Caps and Caps with ¼" Aperture would need to be ordered with the Manifold.

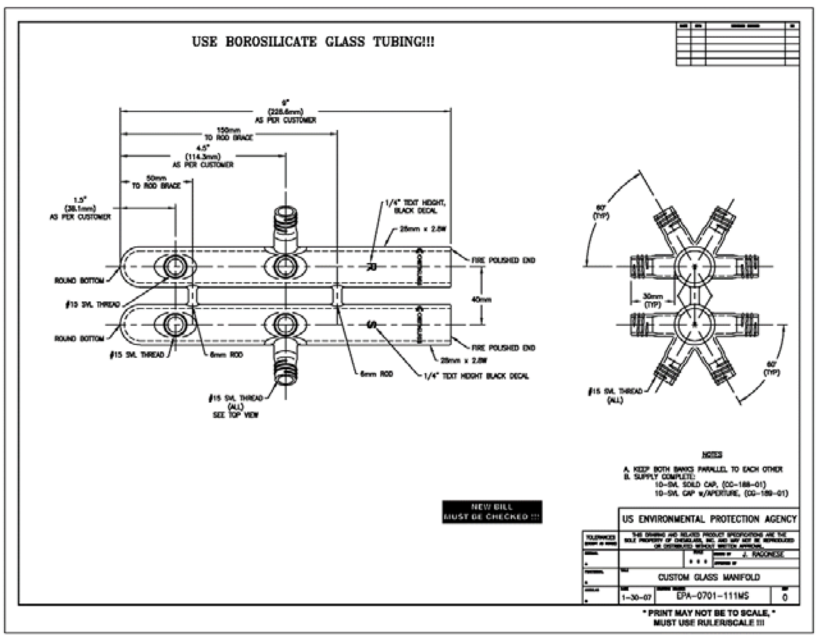


Figure 9.4.11.1 Dual glass manifold schematic

9.4.12 SRP Warm-Up Time

Plug in the power cable and place the power switch on the electronics module in the "on" position. Allow the SRP an initial warmup time period of at least four hours for the system to become fully stable. Normally the SRP would be left on after the initial setup with the modules kept in idle mode (with the pump switch in "off" position) until needed for a verification or any routine system characteristics data checks. It is recommended that a power strip be used for turning the SRP on and leaving the power switch in the on position. The power toggle on the SRP is slightly under-rated for the equipment; a newer version will soon be available to all the operators.

9.5 SRP-07 Standard Operating Procedure

9.5.1 Procedure and SRP Control Software

The following is the procedure for running SRP-07 (the host) against a Regional SRP (the guest). However, the principles apply equally to any other Guest instrument that is brought in to a Region for Qualification, Verification or Re-Verification. Any references to the NIST SRP Control Software will be kept to a minimum for two reasons. The first reason is to keep this document from becoming too large and too difficult to handle. The second reason is that the software will be written up in a separate SOP, detailing commercial instrument setups in more detail and more detailed information on various parameters of the software.

9.5.2. QC Checks

9.5.2.1 Data Sheet

Before running a Qualification, Verification, Re-Verification, Audit Level Comparisons or a Zero – Span, a QC Check on the SRP needs to be performed. The frequency of the QC Checks could be referenced to the International Standards Organization (ISO) 17025 definition of determining the time span in-between calibrations by establishing a historical record of intervals needed to keep a current calibration without having to make adjustments. The Pressure and Temperature circuits have a tendency to drift ever so slightly over a period of two to three days. Therefore, it is essential to run the QA/QC as often as possible, even daily. Section 9.6 contains an example of the Excel QA Check Sheet.

9.5.2.2 Title Heading

In the heading of the QA check, enter the current Date, SRP Serial Number, the Location of the SRP, the Ambient Room Temperature and the name of the analyst who will be conducting the tests. In the current QA Spreadsheet, some of these functions can be invoked by clicking on a box or selecting from a drop down menu (see QA SRP FORMAT.xls). The QA spreadsheet will be in an Excel Spreadsheet with macros added in to help automate the QA process. Additionally, the QA spreadsheet will assist the SRP operators in determining the PASS/FAIL of a QA check. The following sections describe the information required for each section of the QA spreadsheet.

9.5.2.3 Section 1: Equipment Calibration Validation:

Section 1 should include the date that any equipment was last calibrated or certified. Such items would include the SRP that is being used, the pressure monitor, the volt meter, the STOLAB Temperature Calibrator and the temperature meter. Also include the last time the Zero Air System had any maintenance performed on it and also the pressure (psi) that Zero Air is being fed to the SRP. To maintain proper NIST Traceability, it is important to document all devices used in association with the SRP. Please contact your Program Coordinator for further assistance with these items. The following images (figures 9.5.2.3.1 and 9.5.2.3.2) are examples of the temperature/voltmeter being used in RTP.

Figure 9.5.2.3.1 Temperature/voltmeter

Figure 9.5.2.3.2 Temperature/voltmeter

9.5.2.4 Section 2: Warm-up Time

Section 2 (Line 1) is to document the warmup time for the SRP and STOLAB Calibrator. SRP-07 should be allowed to warm up for a minimum of four hours. However, it may take even longer depending on the SRP. A good rule of thumb would be to let it warm up overnight. A major factor in the time it takes to warm up is the Source Block Temperature and how well the heating element is working. An older heating element may take longer that a newer heating element, or there may be a slight difference in the size, wattage and power. Once the Source Block reaches the desired temperature, it will still take the

Proportional-Integral-Derivative Controller (PID Controller) a little while longer as it tunes itself to the desired set-point. If the Source Block temperature has not had sufficient time to equilibrate properly, the SRP will not pass the “Stability Test”.

Section 2 (Line 2) is intended to record the date and time that the STOLAB Calibrator is plugged into the back of the Electronic Module. A minimum of 15 minutes is required for the STOLAB to heat up all the internal elements. Either the 30 °C (Fig 9.5.2.4.1) or the 100 °C (Fig 9.5.2.4.2) STOLAB Calibrator may be used.

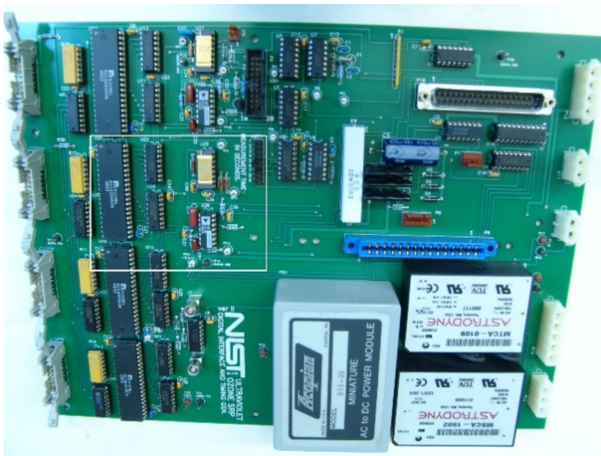
30°C Calibrator **100°C Calibrator**

Figure 9.5.2.4.1 ***Figure 9.5.2.4.2***

Section 2 (Line 3) is intended to record the date and time that the QA/QC is started. The date and time in this line needs to be 4 hours greater that line 1 and 15 minutes greater that line 2.

9.5.2.5 Section 3: Temperature Check

The temperature is a critical measurement in the calculation of the concentration of O_3, so special care needs to be taken in the QA/QC of the temperature resistance temperature detector (RTD). Once the O3 concentration is calculated, it is adjusted to ambient temperature and pressure. There are two separate functions of the entire temperature circuit in the SRP. The first check is to validate that the STOLAB Temperature card is functioning at the proper voltages as specified by the manufacturer, and the second half is verifying the Zero and Span of the temperature circuits on the SRP mother board (see Figure 9.5.2.5.1).



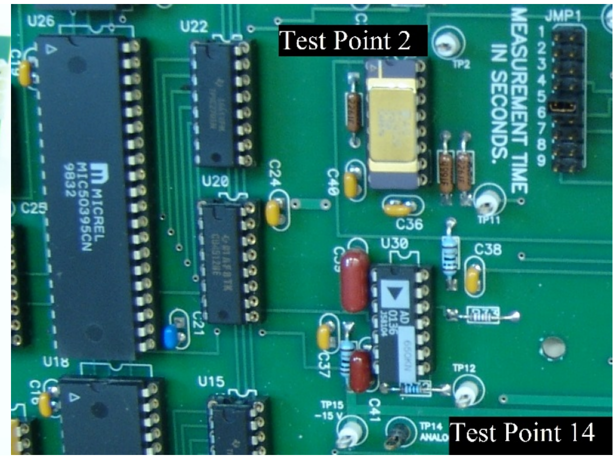


Figure 9.5.2.5.1 SRP Mother Board ***Figure 9.5.2.5.2 Close up showing TP2 and TP14***

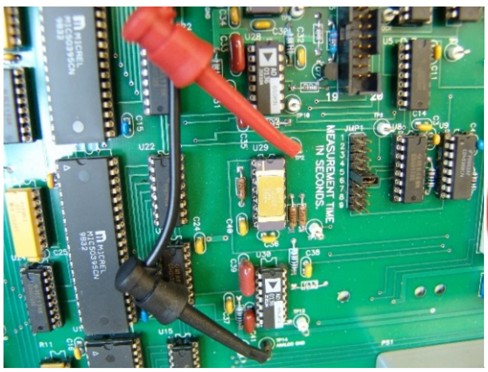
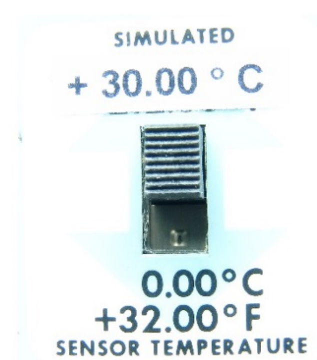


Figure 9.5.2.5.3 TP2 and TP14 connected. ***Figure 9.5.2.5.4 TP2 Minigrabber Set*** ***Figure 9.5.2.5.5 Volt Meter Probes***

STEP 1: Connect the Mini-Grabber (see Figure 9.5.2.5.4) red lead to TP2 and the Mini-grabber black lead to TP14 (see Figure 9.5.2.5.2 and Figure 9.5.2.5.3) and plug the other end of the cable into the Positive (red) and the Negative (black) terminals of the volt meter. The Volt Meter needs to have a resolution of at least 0.1 mV. With the STOLAB Calibrator set to 0° C (see Figure 9.5.2.5.6) record the Volt Meter reading in millivolts (mV).



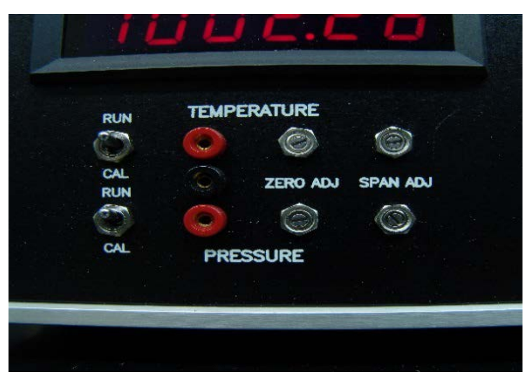


Figure 9.5.2.5.6 ***Figure 9.5.2.5.7*** ***Figure 9.5.2.5.8***

STEP 2: Switch the STOLAB Calibrator to 30°C (see Figure 9.5.2.5.7) and record the Volt Meter R\reading.

STEP 3: Insert the red Volt Meter Probe into the upper red socket on the front panel of the Electronics Module (see Figure 9.5.2.5.8) and the black Volt Meter Probe into the black socket in-between the two red sockets. Remove the banana plugs for the Mini-Gabbers and plug in the banana plugs Volt Meter Probe (red to red and black to black). Flip the switch for the Temperature from RUN to CAL (see Figure 9.5.2.5.8) and record the Volt Meter reading.

STEP 4: In order for the front panels to display the temperature in this step the Stability Diagnostic needs to be running in the SRP Software. Flip the switch for the Temperature from CAL to RUN (see Figure 9.5.2.5.8) and switch the STOLAB (temperature) Calibrator to 30°C. Now the reading is taken from the Electronics Module front display. Record the temperature read-out and record the value.

STEP 5: PASS / FAIL Criteria

STEP 1: > 0.0 mV and <1.0mV

STEP 2: 300.0 mV ± 0.1 mV or 1000.0 mV ± 0.1 mV

STEP 3: >0.0 mV and <1.0 mV

STEP 4: 30°C (or 100°C) ±0.01°C

STEP 6: If all four PASS then proceed to the Pressure Check. If any one of the four steps FAILS, then take the following corrective measures:

STEP 1: Adjust the Zero on the STOLAB Temperature Board at STEP 1 settings

STEP 2: Adjust the Span on the STOLAB Temperature Board

STEP 3: Adjust the Zero on the Electronics front panel.

STEP 4: Adjust the Span on the Electronics front panel.

Then repeat STEPS 1 through 4 and move on to the Pressure Check.

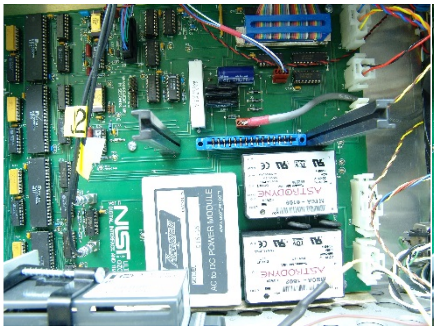


Figure 4.2.5.9

SRP Mother Board

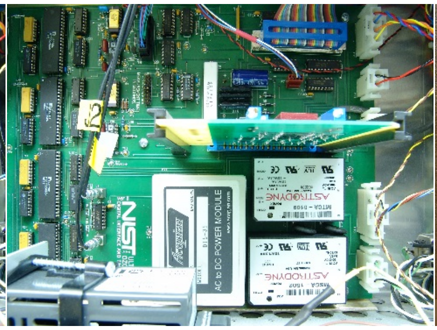


Figure 4.2.5.10

SRP Mother Board with STOLAB Temperature Card Installed

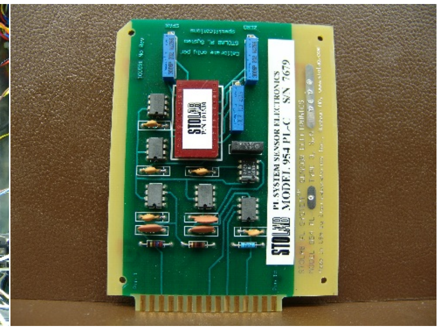


Figure 4.2.5.11

STOLAB Temperature Card

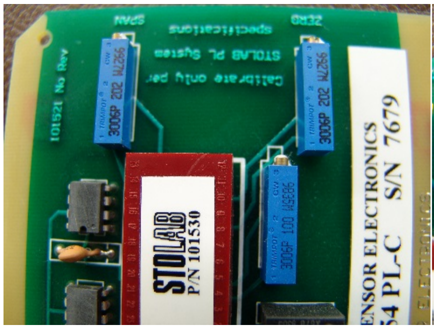


Figure 9.5.2.5.12

Zero and Span Adjustment Pots

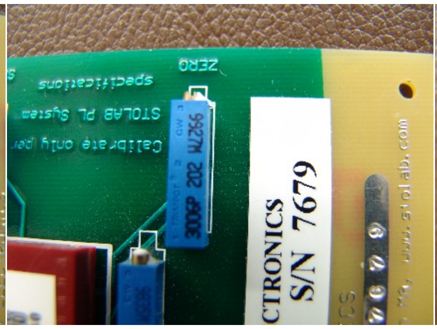


Figure 9.5.2.5.13

Zero Adjustment Pot

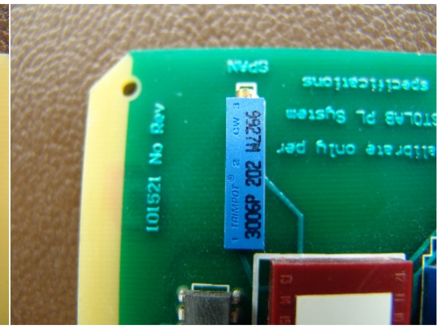


Figure 9.5.2.5.14

Span Adjustment Pot

9.5.2.6 Section 4: Pressure Check

Pressure is also a critical measurement that is applied in the calculation of the concentration of O_3. Once the O_3 concentration is calculated, it is adjusted to ambient pressure and temperature. There needs to be an accurate Lab Standard that can be used as a reference. As an example, in Region 4, RTP, a Mensor Series 600 Automated Pressure Calibrator is used as a Lab Standard. Every year this instrument is sent to an ISO 17025 Certified Laboratory for certification. There are three steps to the pressure check.

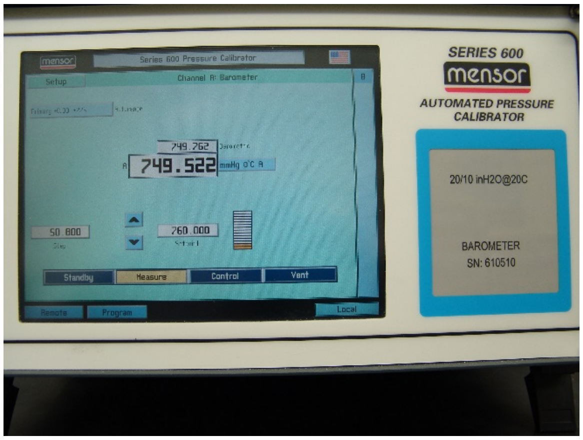


Figure 9.5.2.6.1 Mensor Lab Standard

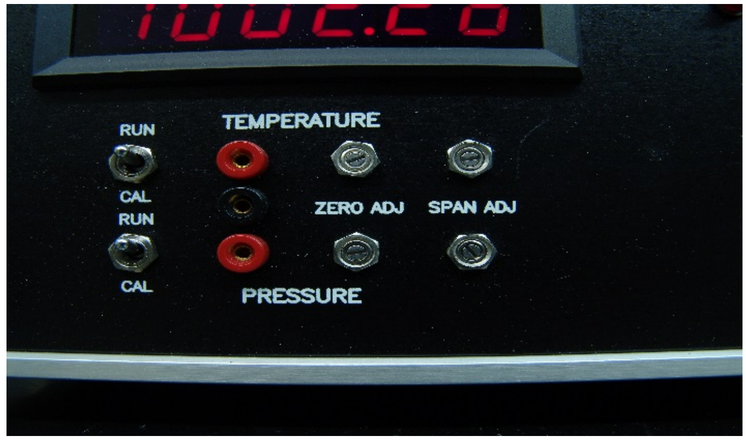


Figure 9.5.2.6.2 Zero Adjust and Span Adjust for Pressure

Before starting the Pressure Check, turn off the SRP Sample pump and disconnect the "Sample In" and "Reference In" lines from the manifold. Turning off the Zero Air Supply to the SRP will accomplish the same goal if there are no other instruments attached to the manifold and drawing air out of it. Also, have the Stability Monitor or the Stability Multi-Monitor running so that the readings on the front panel will be updated every few seconds.

Step 1: Record the Lab Standard reading in millibars (mb) in the PRESSURE Section of the QC Document next to "Lab Standard" and under "Unadjusted".

Step 2: Record the Pressure readout on the SRP Electronics Module front display. The display output is in millibar pressure and record this value in the QC Check List. **The PASS/FAIL criterion for this reading is ± 0.2 mb from the Lab Standard.**

Step 3: Find the RUN/CAL switch to the left of the PRESSURE ZERO ADJ and the SPAN ADJ potentiometers on the Electronics Module Front Panel and flip the switch to the CAL position. Record the value after the reading stabilizes. **The PASS/FAIL criterion for this reading is ± 0.1 mb from 700.00 mb.**

Corrective Action: If any one of these three steps FAILs, then adjustment to the SPAN and ZERO will need to be made. Keep in mind that the ZERO ADJ will affect the span, and then the SPAN ADL will affect the zero. In the meantime, the barometric pressure may also be changing. Therefore, keep referencing the Lab Standard, the SPAN and the ZERO to make adjustment using the potentiometers for the PRESSURE on the front panel. Try adjusting the ZERO first and then move back to the SPAN. It will be necessary to move back and forth several times for it to pass, but once it does pass, repeat steps 1 through 3, record the values and move on to the next section.

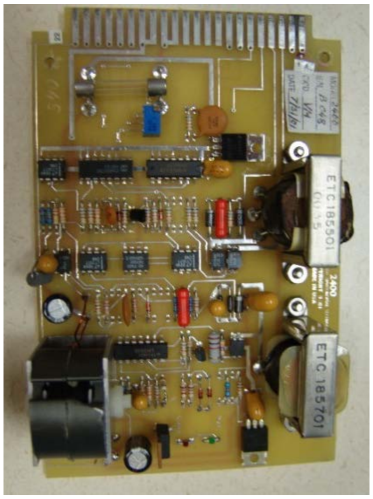
9.5.2.7 Section 5: UV Source Lamp and Block Temperature

Section 5 is the area where the Ultraviolet (UV) lamps are documented, and the temperature of the heated block is recorded. Two different lamps are used in each SRP: the UV Source Lamp and the O_3 Generator Lamp. The temperature for the Source Lamp Heated Block is located on the Watlow PID Controller inside the Electronic Module. The temperature for the O_3 Generator Heated Block is inside the Pneumatics Module. Ensure that the temperature readout is the same as the set point. If they are not within ±0.1°C, then that may be an indication of a problem or the heated blocks have not reached the set point. Failure to reach the set point can lead to stability issues that will be discussed in Section 8. A future version of the/ QC Work Sheet will also include the date that each lamp was installed. The O_3 Generator Heated Block temperature is usually kept a little lower because the block is located right next to the MFC and temperature will not significantly affect the stability of O_3 generation. There are no PASS/FAIL criteria for these temperature settings other than that they need to be stable.

9.5.2.8 Section 6: Total Scalar Counts

If the Stability program is running, this would be a good time to stop it and record the Scalars from Cell 1 and Cell 2. Each Cell has a UV Detector that measures the intensity of the UV light being transmitted through the Cell. The more O_3 that is in the Cell, the lower the Scalar count will be. As the UV Light hits the detector, the detector will respond with a frequency. The more UV light, the higher the frequency. The SRP will then sum the frequency, at a given Hertz (Hz) for a total of five seconds. The summed frequencies of the given Hertz is the value that will be displayed on Scalars 1 and 2. **The PASS/FAIL criteria for the Scalars should be >100,000 and <250,000. There are three methods that can be used to increase the Scalar Counts.**

The first method is to adjust the potentiometer on the O_3 card to apply more power to the lamp. Two styles of boards are seen in Figures 9.5.2.8.1-9.5.2.8.3, below. Find the blue potentiometer on the board and turn it so that the Scalars move up. The stability should be running during the adjustment. If the scalar counts do not increase sufficiently, then the next method to try is to replace the UV Lamp. And finally, if those two methods do not increase the scalar counts sufficiently, it may be time to replace the board.



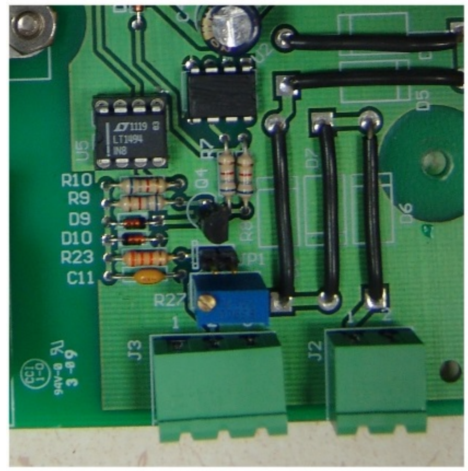


Figure 9.5.2.8.1 Old Style Board *Figure 9.5.2.8. 2 New Style Board* *Figure 9.5.2.8.3 close up*

9.5.2.9 Section 7: Dark Counts

To check the dark counts, start with the SRP Control Software in the Standby Mode. On the SRP control panel, click on the “SHUUER” button, and you should hear the shutter close. Then start the “Stability” or “Multi-Stability” Diagnostic Tool. The Dark Count will then be displayed on the front panel of the SRP and on the computer screen. Record the Scalar Counts for the Cells on the QA sheet. Proceed with the adjustment of the Dark Counts by adjusting the potentiometers on the top of the detector. The software will run the Dark Count Stability test for only 10 to 15 minutes before it will abort the run. Restart the Dark Count Stability again if more time is needed. The acceptance criteria for the Dark Counts are >5 and <25 counts. In some labs where Relative Humidity may fluctuate throughout the day, you may want to add a packet of desiccant to the bottom of the detector or add a small purge line of Zero Air to flush the detector to help stabilize the Dark Counts.

9.5.2.10 Section 8: Stability Test

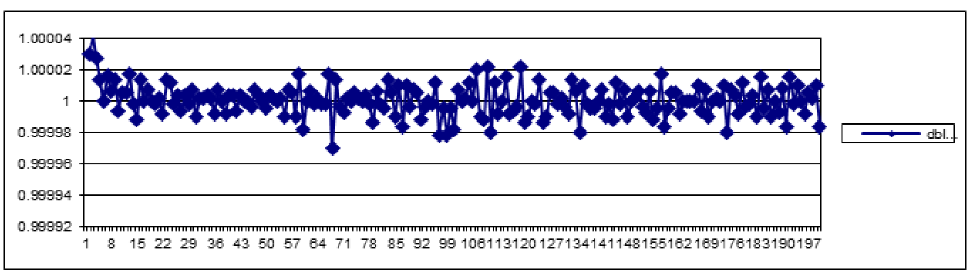
Once all the QA Checks have passed, the Stability Test needs to be run. The Stability test will run only one SRP at a time, where the Multi-Stability can run up to four SRPs at a time. In the Stability test, 20 repetitions will be averaged together and a total of 10 cycles will be run.

The following is an example of a Stability Test on SRP-01 from 09/12/2014:

Diagnostic Test Report
Stability Monitor

Calibrating Institute: EPA RTP **Date:** 9/12/2014
Operator: Scott Moore **File Name:** dstab147.xls
Instruments: SRP-01
Comment: Reps = 20; Cycles = 10

		Temp	Pressure	Scalar1	Scalar2	Ratio
1	Average:	24.52	1005.0	126383	100155	1.261868
	Std Dev:	0.05	0.0	5	9	0.000065
2	Average:	24.58	1005.0	126393	100170	1.261788
	Std Dev:	0.00	0.0	3	3	0.000010
3	Average:	24.56	1005.0	126404	100180	1.261771
	Std Dev:	0.01	0.0	2	1	0.000008
4	Average:	24.54	1005.0	126413	100187	1.261769
	Std Dev:	0.01	0.0	6	5	0.000009
5	Average:	24.50	1005.1	126424	100195	1.261775
	Std Dev:	0.01	0.0	5	3	0.000019
6	Average:	24.46	1005.2	126430	100197	1.261814
	Std Dev:	0.01	0.0	3	3	0.000016
7	Average:	24.42	1005.2	126448	100210	1.261822
	Std Dev:	0.01	0.0	6	5	0.000008
8	Average:	24.39	1005.2	126468	100225	1.261842
	Std Dev:	0.01	0.0	**8**	**6**	**0.000010**
9	Average:	24.35	1005.3	126474	100229	1.261851
	Std Dev:	0.01	0.0	**2**	**2**	**0.000006**
10	Average:	24.32	1005.3	126484	100238	1.261843
	Std Dev:	0.01	0.0	**8**	**6**	**0.000010**
	average	1.000001				
	stdev	1.00E-05				



The Pass/Fail criteria are based on the last three Std. Dev. For Scalar1 and Scalar2, they should be <15 and the Std. Dev. For the three Ratios should each be <0.00003.

Last 3 < 0.0030 Ratio Std Dev dstab131 or other

Last 3 < 15 Count ratio

9.6 QA Data Sheet

QC CHECKs

DATE:	
SRP S/N :	SRP-01
Location :	Research Triangle Park, NC
Ambient Lab Temperature (° C) :	
Operator :	

1. EQUIPMENT CALIBRATION VALIDATION:

	DEVICE	SERIAL NUMBER	DATE CALIBRATED	DATE DUE
	STOLAB (PL 0-30)	6210	1/10/2014	1/10/2015
	STOLAB PL 0-100)	4771	1/10/2014	1/10/2015
	Fluke 743 B	7115612	2/15/2014	2/15/2015
	Fluke 744	9816004	11/20/2013	11/20/2015
	Mensor	621604	6/11/2014	6/11/2015
	K-Type TC	SN 03926	12/31/2014	12/31/2015
	Source Lamp	SN 00580576	6/2/2014	NA
	Ozone Generator Lamp	SN 0001188172	6/2/2014	NA

2. WARM-UP TIME

	DATE AND TIME SRP WAS TURNED ON	01/01/15 08:00 AM	OK

	DATE AND TIME STOLAB PLUGGED IN		

	DATE AND TIME TEMPERATURE QA START		

3. CALIBRATION OF TEMPERATURE STANDARD

STOLAB MODEL NUMBER	**PL-30**

	Unadjusted	Adjusted	Status
A: Circuit Reading (mV)			
B: Circuit Reading (mV)			
C: Circuit Reading (mV)			
D: Display Reading (°C)			

4. PRESSURE

	Unadjusted	Adjusted	Status
A: Lab Standard			
B: SRP Readout			
C: Zero Readout			

5. UV SOURCE AND GENERATOR BLOCK TEMPERATURE

	Set Point	Actual	Adjusted
A: UV Source Block (° C)			
B: UV Generator Block (°C)			

6. TOTAL COUNTS

	Unadjusted	Adjusted
Cell #1		
Cell #2		

7. DARK COUNTS

	Unadjusted	Adjusted
Cell #1		
Cell #2		

8. PRECISION

Run the Stability Monitor Diagnostic Program (10 sets of 20 replicates per set); printout and attach.

9.7 Re-Verification Procedure

9.7.1 The Re-Verification may begin after all the QC Checks have passed. (see section 9.5.2 QC Checks)

9.7.2 Check the Temperature and make sure the RTD is plugged in and not the STOWLAB Calibrator. Also check the Temperature calibration switch on the front panel of the Electronics Module is in the “UP” position.

9.7.3 Check the Pressure calibration switch on the front panel of the Electronics Module is in the “UP” position.

9.7.4 Plug the J-4 cable into the back of the pneumatics module of the SRP that will be generating Ozone. Preferably the regional SRP that is being Re-Verified should generate Ozone, however SRP-07 could also be used.

9.7.5 Connect the Sample Output line from the Ozone Generating SRP to the bottom of the “Sample” side of the manifold.

9.7.6 Connect the Reference Output line from the Ozone Generating SRP to the bottom of the “Reference” side of the manifold.

9.7.7 Connect the Sample lines from both of the SRPs to the upper ports of the “Sample” side of the manifold.

9.7.8 Connect the Reference line from both of the SRPs to the upper ports of the “Reference” side of the manifold.

9.7.9 Turn on the Zero Air to the SRP that will be generating Ozone. Operating pressure should 15 to 25 pounds per square inch of pressure.

9.7.10 Turn on the Flow Controller Power located on the front of the Pneumatics Module of the SRP that will be suppling Ozone. The power to the Mass Flow Controller should never be ON when there is no Zero Air being supplied to the SRP.

9.7.11 Turn on the Ozone Generator Lamp Power located on the front of the Pneumatics Module of the SRP that will be suppling Ozone.

9.7.12 Inside the Pneumatics Module, on the right hand side, there are two switches that allow the computer to control the Mass Flow Controller and the Power to the UV Lamp in the generator. Make sure that both are in the “ON” position.

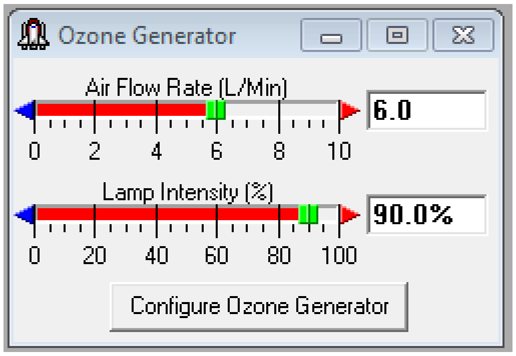
9.7.13 Shut down any other software that may be open (this includes any Excel spreadsheets).

9.7.14 Shut down the SRP Control Program. (System <ENTER>, Exit <ENTER>)

9.7.15 Restart the SRP Control Program.

9.7.16 Use the Ozone Conditioning Tool to find the settings for the upper Ozone concentration and the Lower Ozone concentration. (System <ENTER>, Diagnostic <ENTER>, Ozone Conditioning <ENTER>. Then select all the instruments that will be used.

9.7.17 The Ozone Generator Control Box will now be activated.



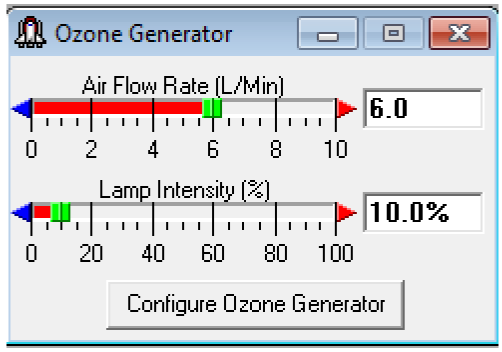


Figure 9.7.17.1
Ozone Generation Control Box
High Ozone Concentration Example

Figure 9.7.17.2
Ozone Generation Control Box
Low Ozone Concentr5ation Example

Generally, 6.0 liters per minute with a Lamp Set to 90.0% should generate close to 1000 ppb Ozone with enough flow to supply both SRPs with a Standard Deviation < 0.7. And with the Lamp Set to 10.0% it should generate close to 15-20 ppb Ozone with enough flow to supply both SRPs with a Standard Deviation < 0.7. Determine the Upper and Lower concentration point to use and record it for use in the Re-Verification.

9.7.18 Set the Lamp to 0.)% and then exit the Ozone Conditioning Diagnostic (System <ENTER>, Script Control <ENTER>, Abort <ENTER>)

9.7.19 Ready to start the Verification. System <ENTER>, Calibration <ENTER>, Standard <ENTER> and following box (Fig 9.7.19.1) will pop up.

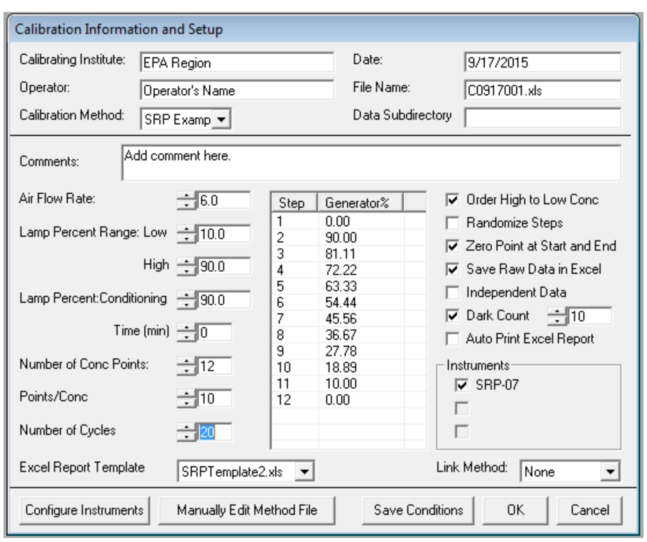


Figure 9.7.19.1 Verification Control Box

9.7.19.1 Calibration Institute: Please use EPA Region #. City and state may also be included.

9.7.19.2 Operator: Please include the Operator's full name.

9.7.19.3 Calibration Method: Once a method is generated for Re-Verifying an SRP a copy can be saved and used again later on.

9.7.19.3 DATE: Current date is automatically entered.

9.7.19.4 File Name: File name is automatically entered.

9.7.19.5 Data Subdirectory: The subdirectory automatically defaults the "Data" directory. However an alternate subdirectory may be entered here at the operator's discretion.

9.7.19.6 Comments: Please4 enter any comments such as which SRP is generation Ozone or any repairs or annual maintenance performed prior to the Re-Verification.

9.7.19.7 Air Flow Rate: 6.0 liter per minute is recommended for two SRPs. The excess flow from the Sample and Reference Manifold should be verified with a rotometer to make sure there is at least 1.0 liters per minute excess flow.

9.7.19.8 Lamp Percentage Range – Low: Use the Low Range established from the Ozone Conditioning Control Box (see Section 9.7.15 above) or from a recent Verification.

9.7.19.9 Lamp Percentage Range – High: Use the High Range established from the Ozone Conditioning Control Box (see Section 9.7.15 above) or from a recent Verification.

9.7.19.10 Lamp Percent Conditioning: Generally use the same value for the Lamp Percentage Range – High used above (9.7.17.9).

9.7.19.11 Time (min): Set to zero if conditioning is not required. If new tubing or a new pump were recently installed, then running the conditioning for 15-30 minutes would be required.

9.7.19.12 Number of Conc Points: To maintain consistency across all the EPA Regions select 12 concentration points. With the "Zero Point at Start and End Box" checked this would be 10 actual concentration points and two Zero points.

9.7.19.13 Points/Conc: This the number of concentration points that are averaged together to complete a calibration step. To maintain consistency across all the EPA Regions select 10.

9.7.19.14 Number of Cycles: Set this value for the maximum of 20 cycles and allow the Verification to run overnight. Each cycle will take about two hours. Allowing it to run overnight at least 6 cycles or more. The minimum for an SRP Verification is 6 cycles. The Step/Generator% List to the right will automatically calculate even step for each concentration point. Specific concentration s are not required for a Verification.

9.7.19.15 Excel Report Template: Default templates are found in C:/Program Files/SRP2002/Templates or C:/Program Files (x86)/SRP2002/Templates. Open SRPTemplate-A4.xls and edit cell A2 to "Verification Report". In all three Guest pages. Save the file as "Verification.xls" and use this file as the default report for all Verifications.

9.7.19.16 Order High to Low Conc: Check the box.

9.7.19.17 Randomize Steps: For an SRP Verification leave this box unchecked.

9.7.19.18 Zero Point at Start and End: Check the box.

9.7.19.19 Independent Data: For manual data entry thei box would be checked, but for automatic data entry leave this box unchecked.

9.7.19.19 Instruments: Check the SRP-07 box and any other instrument that applies.

9.7.19.20 Link Method: Leave unchecked.

9.7.19.21 Save Conditions: Clicking on this button will prompt you to save the method. The method can be saved over an existing method or a new method name can be saved.

9.7.19.22 OK: Selecting this button will begin the Verification.

9.7.20 Allow the Verification to run overnight for a minimum of six complete cycles.

9.7.21 Copy all the data runs, QC Checks and Stability to an E-mail and send it to RTP for data validation.

9.7.22 Once a Summary Report has been received from RTP documenting that the Regional SRP has passed its annual Verification, then SRP-07 may be boxed up and returned to RTP. With the summary Report there will also be the last Verification of SRP-07 and a copy of the last NIST Verification. These reports should be saved and provided to any guest Verification upon request.

10. Data Records and Management

The RTP and Regional SRP operators are responsible for creating, retaining, and maintaining the forms and reports generated by the use of the SRP in comparison to the traveling SRP, and in the comparison to the state, local, tribal and CASTNET ozone primary standards that are required to be compared to the SRPs every year. Along with the QA Data Sheet described in Section 9.6, a verification report, like the example in Figure 10.2 is required to be developed and saved as evidence of an acceptable verification.

U. S. Environmental Protection Agency
Office of Research and Development
Air Pollution Prevention and Control Division
Technical Services Branch
109 T.W. Alexander Drive
RTP, NC 27711

	Primary Standard		Guest Information	
U.S. EPA	Agency:	EPA RTP	Agency:	EPA R4
Scott Moore	Contact:	Scott Moore	Contact:	Mike Crowe
4930 Old Page Rd.	Make:	NIST	Make:	NIST
RTP, NC 27709	Model:	SRP	Model:	SRP
(919) 541-5104	S/N:	01	S/N:	10
moore.scott@epa.gov	NIST Ver.	Apr 18, 2013	Offset:	0
	Level	1	Status:	PASS

	Slope	Intercept	R^2
Averages:	1.0007	0.0092	0.99999987
Upper Limit:	1.0100	1.0000	NA
Lower Limit	0.9900	-1.0000	NA

Date Start	Time Start	Date End	Time End	File	Slope	Intercept	R^2
04/08/14	18:34	04/08/14	20:38	c0408001.xls	0.9983	0.3562	0.99999951
04/08/14	20:38	04/08/14	22:38	c0408002.xls	0.9998	-0.0197	0.99999992
04/08/14	22:38	04/08/14	0:38	c0408003.xls	1.0002	-0.0090	0.99999990
04/09/14	0:38	04/08/14	2:38	c0408004.xls	1.0004	0.0091	0.99999997
04/09/14	2:38	04/08/14	4:42	c0408005.xls	1.0006	-0.0266	0.99999989
04/09/14	4:42	04/08/14	6:46	c0408006.xls	1.0006	0.0450	0.99999990
04/09/14	6:46	04/08/14	8:56	c0408007.xls	1.0008	-0.0051	0.99999995
04/09/14	8:56	04/08/14	11:01	c0408008.xls	1.0007	0.0152	0.99999985
04/09/14	11:01	04/08/14	13:05	c0408009.xls	1.0006	0.0735	0.99999987
04/09/14	13:06	04/08/14	15:21	c0408010.xls	1.0009	-0.0380	0.99999977
04/09/14	15:21	04/08/14	17:34	c0408011.xls	1.0010	-0.0855	0.99999993
04/09/14	17:34	04/08/14	19:44	c0408012.xls	1.0011	0.0300	0.99999993
04/09/14	19:44	04/08/14	21:59	c0408013.xls	1.0012	-0.0930	0.99999984
04/09/14	21:59	04/08/14	0:14	c0408014.xls	1.0011	-0.0146	0.99999993
04/10/14	0:14	04/08/14	2:36	c0408015.xls	1.0012	-0.0934	0.99999990
04/10/14	2:36	04/08/14	4:54	c0408016.xls	1.0010	-0.0632	0.99999993
04/10/14	4:54	04/08/14	7:03	c0408017.xls	1.0011	-0.0332	0.99999976
04/10/14	7:03	04/08/14	9:12	c0408018.xls	1.0010	0.0124	0.99999988
04/10/14	9:12	04/08/14	11:24	c0408019.xls	1.0008	0.0274	0.99999997
04/10/14	11:24	04/08/14	13:39	c0408020.xls	1.0008	0.0973	0.99999988

Comments: The following parts were replaced on SRP-10 before starting the Re-Verification: A new RTD, all external Teflon tubing, two new rotometer, a new double headed diaphragm pump (with quick connect plugs), two new Ozone Boards, one of the SS bulkhead fittings, the source block heating element, support brackets for the pneumatics module power supply, plexiglas shield over optical bench terminals, a new SRP label plate and bulkhead locks for Teflon fittings. We modified the Pneumatics module so that the rotometer can be installed with the Swage adapter already mounted to the rotometer before installation. Ten concentration points were run, on an average at 1015, 913, 809, 705, 600, 495, 388, 275, 163 and 42 ppb Ozone with a Zero at the start and end of each cycle. Your next re-verification will be due by April 10, 2015.

Scott A. Moore *Scott A. Moore* DATE: April 10, 2014

Figure 10.2 Example Verification/Re-verification Report

11.0 Quality Control and Quality Assurance

EPA SRP Quality Control Procedures are addressed in the Procedures section, along with forms that have to be filled out and acceptance criteria that must be met. Quality assurance for the EPA SRP network is provided by the annual certification of the EPA RTP SRPs against the NIST SRPs, by the NIST SRP maker/ operator. The comparison of the EPA Regional SRPs against the Traveling RTP SRP is, in a sense, a secondary check to ensure traceability to NIST.

12.0 References

12.1 Guidance for Requirements and Use of the EPA SRP Network

Technical Assistance Document (TAD) for Transfer Standards for Calibration of Air Monitoring Analyzers for O_3, October 2013 http://www.epa.gov/ttn/amtic/qapollutant.html. Every reference of this acronym in this SOP applies directly to the document EPA-454/B-13-004.

12.2 Physical Basis of the Photometry Equation

The theory of photometric measurement is very well documented. This portion of the SOP is a guide to the existing documentation, and the reader may follow these links for a deeper understanding of how the SRP calculates the O_3 concentrations. The first place to begin any research on the photometry equation is in the Code of Federal Regulations, and that resource is easily available on line. The following is a list of contents and a web link to all the chapters that pertain to the theory of photometric measurement. The link :http://www.ecfr.gov/cgi-bin/text-idx?tpl=/ecfrbrowse/Title40/40tab_02.tpl

e-CFR 40 CFR PART 50—NATIONAL PRIMARY AND SECONDARY AMBIENT AIR QUALITY STANDARDS

§50.9 National 1-hour primary and secondary ambient air quality standards for O_3.

§50.10 National 8-hour primary and secondary ambient air quality standards for O_3.

Appendix D to Part 50—Measurement Principle and Calibration Procedure for the Measurement of O_3 in the Atmosphere

Appendices H,I, P and U of Part 50 address Interpretation of the 1 and 8-Hour Primary and Secondary National Ambient Air Quality Standards for O_3

Additionally this information is periodically updated. Revisions to the ozone standard, including the Appendix (D) in 40 CFR Part 50 for Ozone, which address the requirements for ozone standard traceability to NIST SRPs, including the addition of recently updated ozone guidance documents, have been proposed. They can be viewed at the following website:

http://www.gpo.gov/fdsys/pkg/FR-2014-12-17/pdf/2014-28674.pdf

The text of interest is near the very end of the proposed revisions,

12.3 Sources of Error in the Photometry Principle

The following references deal with systematic errors and biases of the NIST SRP.

Bass, A.M.. 1977. Ultraviolet photometer for ozone calibration.

Smith. 1986. Standard Reference Photometer Network for Verification and Certification of Ozone Standard.

Paur, R.J., Bass, A.M., Norris, J.E., and Buckley, T.J. (Date Unknown) Standard Reference Photometer for the assay of O_3 in calibration atmospheres" Document Number NISTIR 6963.

Norris, J., Guenther, F.R., (NIST), Wielgosz, R.I., Viallon, J., Moussay, P., (BIPM), Braganza, E., Klamser-Williams,T. (EPA-Las Vegas). NIST Standard Reference Photometer Operations Manual. Document Number "Draft"

Viallon, P., Moussay, F.R., Guenther,F., and Wielgosz, R.I.. 2006. A study of systematic biases and measurement uncertainties in O_3 mole fraction measurements with the NIST Standard Reference Photometer" doi: 10.1088/0026-1394/43/5/016 Metrologia 43 (2006) 441-450

Laiva, M.A. 2011. Uncertainty of ozone measurements with the primary standard reference photometer (SRP 45), *Talanta,* Volume 86, pages 71-81,

Mosquin, P.L.et al. 2011. A model for the estimation of ozone photometer Aggregate Uncertainty for EPA's NIST Traceability Program. Paper 2011-A-580-AWMA, Presented at Annual meeting.

Norris, J. 2013. Temperature measurement and optical path-length bias improvement modifications to National Institute of Standards and Technology ozone reference standards.

12.4 Upgrades of the NIST SRP Hardware and Software

Norris, J.E., Band, A., Bliss, R., and Guenther, F. 2004. Upgrade and intercomparison of the US Environmental Protection Agencies ozone reference standards. AWMA Paper # 04-A-530-AWMA,

Norris, J.E., Choquette, S., Viallon, J., Moussay, P., Wielgosz,R., and Guenther, F. 2013. Temperature measurement and optical path-length bias improvement modifications to National Institute of Standards and Technology ozone reference standards.

12.5 International Photometric Measurements for O_3

The following references are provided for those readers who may want to see published information on the international work on the SRP. As NIST has provided, and continues to provide new SRPS to many of the countries in the world (some have more than one), they have asked the government organization of France (Bureau Internationale des Poids et Measurements, abbreviated the BIPM) that has the parallel role to NIST for that country, to be the international lead on the SRP.

Klausen, J., Zellweger, C., Buchmann, B., and Hofer, P. 2003. Uncertainty and Bias of Surface O_3 Measurements at Selected Global Atmosphere Watch Sites. Journal of Geophysical Research, Vol. 108, No. D19, 4622, doi:10.1029/2003JD003710, 2003

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