Resonance Ltd.
143 Ferndale Drive North, Barrie, Ontario, L4M 9V9, Canada
Tel: (705) 733-3633 Fax: (705) 733-1388
Email: res@resonance.on.ca

OPERATION MANUAL

Atomic Oxygen Absorption System

Bill Morrow, PhD March 10, 2006

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Background on Atomic Oxygen Absorption System

The method for making Atomic Oxygen measurements is Atomic Absorption Spectroscopy (AAS). This method employs a light source (lamp) with an atomic emission line or lines (Oxygen lamp) that are absorbed by atoms in the measurement region. In a typical layout the atoms are created by a plasma generator in a flowing stream of O_2 or O_2 plus a rare gas. The atoms can be switched on and off by the plasma generator providing a convenient way to measure the transmittance of the gas (atoms vs. no atoms). The transmittance (I/Io) is measured by placing the lamp and a wavelength specific Cesium Iodide (CsI) detector of the absorbed spectral lines opposite each other across the stream. These lines at 130.22, 130.46 and 130.61 nm are detected by the CsI detector with a spectral response from 110 to 180 nm. The lamps are very clean and the only significant non absorbed lines in that region are around 135 nm from singlet oxygen. These "impurity lines" are strongly absorbed by molecular oxygen making it possible to have a nearly monochromatic lamp in the VUV range of the detector when a mixture of N2 and O2 is used to purge the gap between the lamp and detector. The Atomic Oxygen (AO) density is given by the Beer-Lambert Law (equation 1).

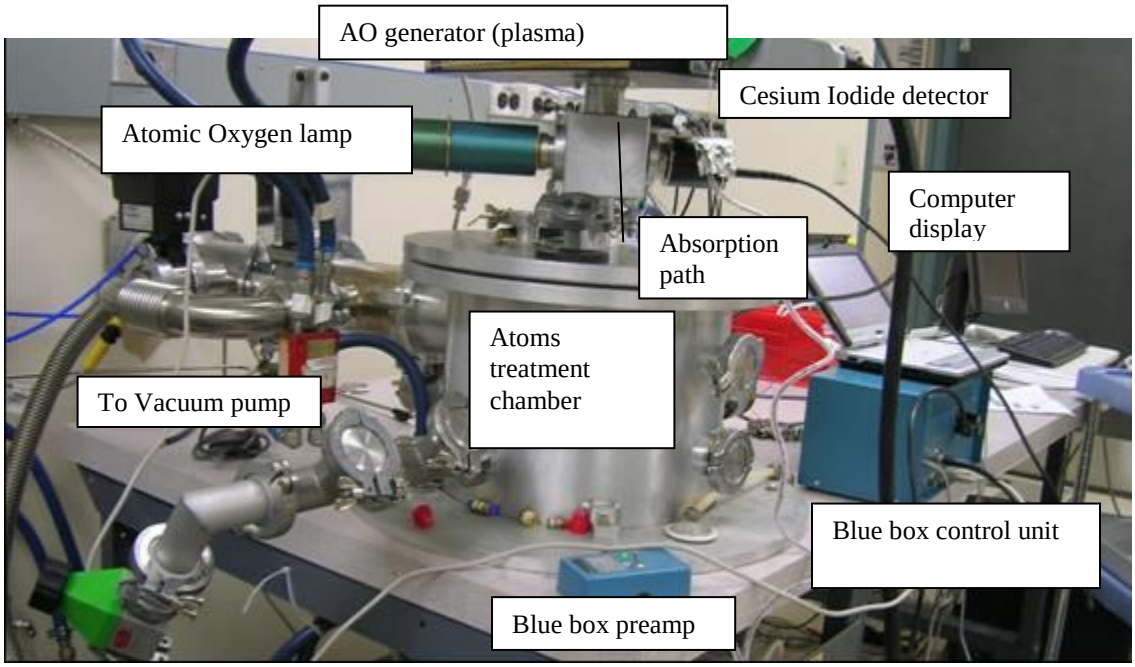


Figure 1 - A typical AO absorption system setup

Linearity and calibration

The use of Oxygen lamps is discussed in the paper given by **Dickenson, 1980**. This paper describes the relationship between the spectral line shape of the lamp and the sensitivity to atomic oxygen. The first lamps used by Dickenson in his pioneering rocket campaign were developed and fabricated by the Author.

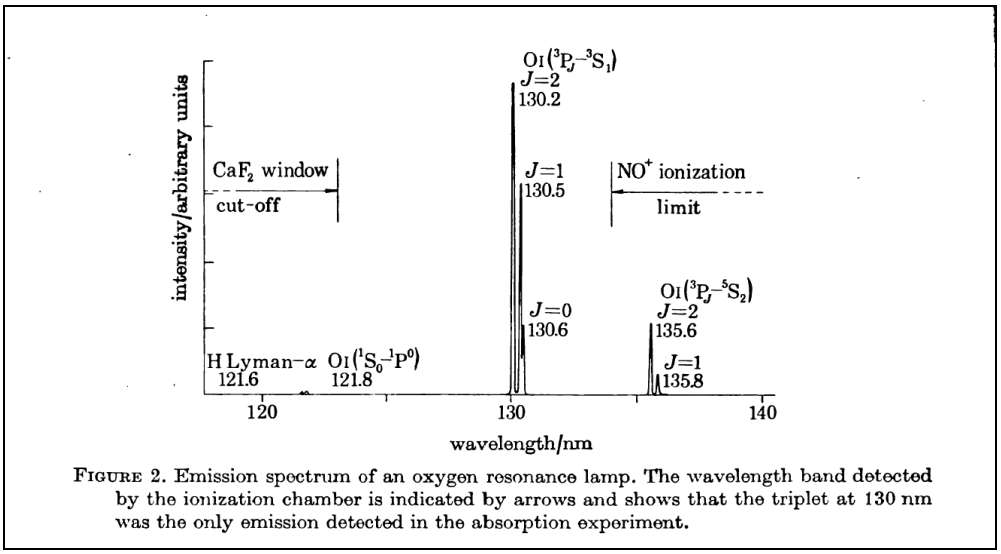


FIGURE 2. Emission spectrum of an oxygen resonance lamp. The wavelength band detected by the ionization chamber is indicated by arrows and shows that the triplet at 130 nm was the only emission detected in the absorption experiment.

Figure 2 - Excerpt from Reference 1 showing the emission spectrum of a typical atomic oxygen lamp used for atmospheric measurement

The system response to Atomic Species is dependent on the optical depth which is determined by the number of atoms per cm^2 in the optical path. The optical depth can be controlled by the optical path length for absorption of lamp radiation. A measure of the optical depth is the ratio of I/Io, that is the lamp/detector signal with the atoms generating plasma on divided by that with the plasma off.

Measurements with atomic oxygen lamps manufactured by Resonance Ltd. indicate this sensitivity follows an exponential relationship (Beer-Lambert Law) with an absorption cross section that is dependent on the absorbance.

Equation 1 - The Beer-Lambert Law with an absorption cross-section dependent on transmittance

$$nl = \frac{-\ln(I/I_0)}{\sigma(I/I_0, T)}$$

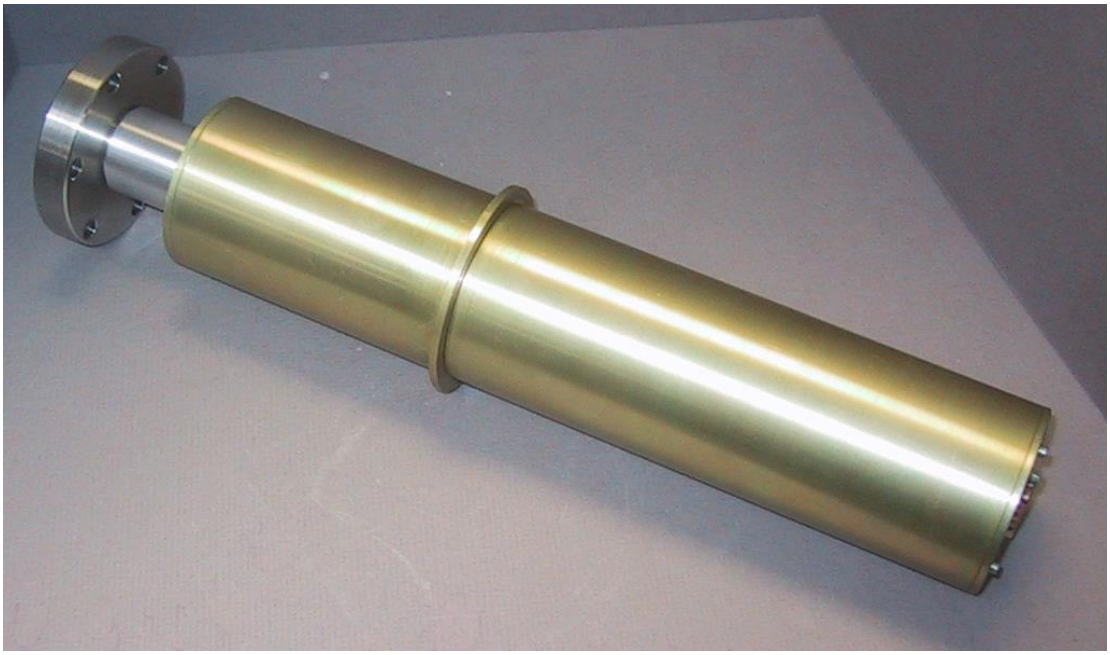
nl = the number of atoms per cm^2 in the optical path
I/Io = the transmittance (absorption signal O generator on over O generator off)
σ = the absorption cross section (cm^2) of the oxygen triplet for a transmittance I/I0 of O atoms at temperature T. This absorption cross section has been successfully derived from a simple polynomial.

In later papers, Dickenson came up with a standard lamp fill that consistently produced experiments with the same sensitivity. This prescription was used on more than 100 rocket flights employing RF powered Oxygen Lamps. For a lamp excited with 100 MHz RF (at approximately 1 Watt) and a fixed O2 density and Helium pressure, the line profile of the lamp is constant. The effective cross section, σ, is a function of the transmittance and the temperature of the atoms. For a given temperature and lamp condition the absorbance I/I0 can be related to the absolute atomic oxygen density in the optical path.

Step by Step Operation

1) Connect AO lamp and CsI detector to vacuum chamber
   a) Connect CF flanges to the vacuum chamber.
   b) Ensure that there is a clear path between the lamp and detector ports.
   c) Ensure that the AO beam will intersect the path between the lamp and detector ports.
   d) Screw 1 inch 20 tpi adapters into CsI and lamp assemblies. The o-rings should be fully compressed.
   e) Screw the lamp and detector assemblies to CF flanges making sure that o-rings are fully compressed.
2) Check Vacuum integrity
   a) Pump down vacuum system.
   b) Lamp and detector interfaces are designed to pump down to < 10^-6torr. If pump down does not occur, check seating of o-rings and CF gaskets.
3) Interconnect CsI detector to small blue box control unit
   a) Connect BNC to CsI detector and to connector labeled “preamp”.
4) Interconnect lamp to large blue box control unit
5) Interconnect large blue box unit to PC
   a) Connect RS232 to phone jack adapter to large blue box control unit.
   b) Connect computer to RS232 either directly or through USB to the RS232 bridge.
   c) The Fujitsu tablet PC employs USB connection on the right.
6) Switch on detector preamplifier
   a) Green led should light.
7) Open TF tools by clicking icon on desktop
8) Set communication parameters for TF tools
   a) 19.2 kBaud
   b) 8 bit
   c) 1 stop bit
   d) Hardware software control off
9) Open program window using File/Open/PSD8MKS.tfb(or latest program version)
10) Using ComPort Menu, set up Capture to File so that data will be saved
11) Go to terminal window
12) Switch on Blue box
   a) TXF Basic logo should appear in terminal window
13) Run program PSD8MKS by moving to program window and hitting Alt R
14) Quickly go through startup menu (defaults are OK so one can quickly start by hitting return several times). This is important since the lamp operates best in pulsed mode. If the program is started but not acquiring data, the lamp will run in CW mode which reduces its lifetime.
15) Check that lamp is on by observing plasma through the view hole in the front of the can.
16) Wait for the lamp to stabilize.
17) Set I_0 with the space bar
18) Observe absorption by change in signal level and I//Io

OPERATION MANUAL
Optically Thin Atomic Oxygen
RF Powered Line Source



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Resonance RF Line Sources

Resonance RF powered line sources are designed to be a reliable and maintenance free compact source of emissions with an operating life in excess of 4000 hours. Our line sources are sealed RF excited sources with a window in an EMI shielded enclosure. The lamp mounts to a 2.75 inch or larger CF type flange. The lamp assembly has an integral RF exciter that is powered by a small wall plug power supply.

Resonance Ltd offers the following bulb types. They include:

1. Kr - Lines at 116.5, 123.6 and 557.0 NM (only a partial list) with a typical 116.5 plus 123.6 flux > $3x10^{15}$ photons per second per steradian and 557 flux > $1x10^{13}$ photons per second per steradian
2. Xe - Line at 147.0 NM (only a partial list) with a typical flux > $5x10^{15}$ photons per second per steradian
3. Ar - Lines at 106.0, 733.4 and 763.5 NM (only a partial list) with a typical flux > $3x10^{13}$ photons per second per steradian in the 106 resonance line
4. Hg - Lines at 184.9 and 254 NM with a typical flux > 8 $x10^{15}$ photons per second per steradian in the 254 line
5. O - Lines at 115.2, 130.2, 135.6, 777.4 and 844.6 NM (only a partial list) with a typical flux of > $3x10^{13}$ photons per second per steradian in the 130.2 resonance triplet
6. N - Lines at 120.1, 149.3, 174.3 NM with a typical flux of > $5x10^{12}$ photons per second per steradian in the 120.1 resonance line
7. Cl - Line at 118.9nm (only a partial list) with a typical flux of > $5x10^{12}$ photons per second per steradian in the 118.9 resonance line
8. H - Lyman alpha at 121.6 NM with a typical flux of > $1x10^{14}$ photons per second per steradian in the 121.6 resonance line

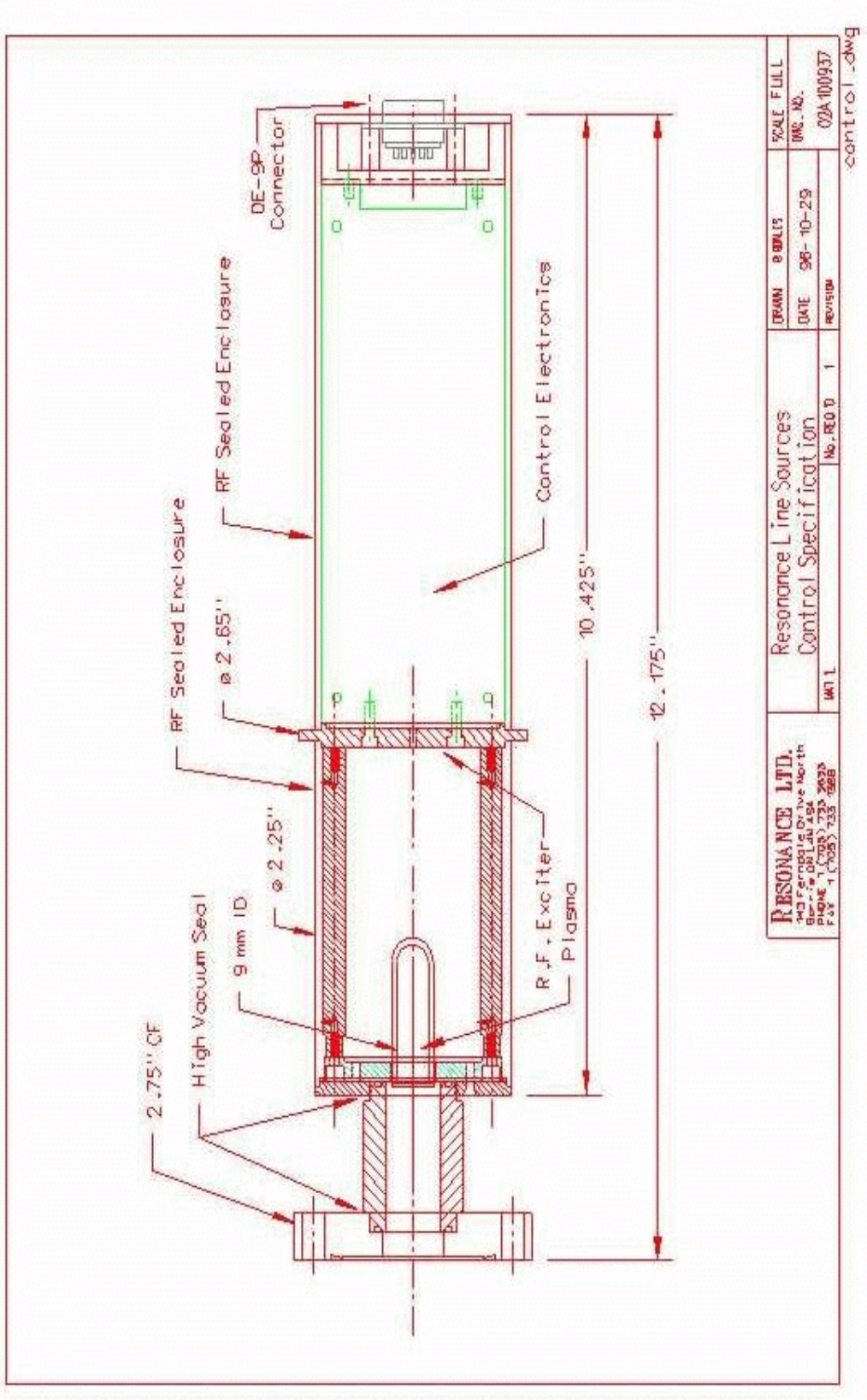


Figure 3 - configuration of O, N, Cl, CO and all optically thin line sources (drawing)

General Specifications:

Lamp Bulbs:

1. Lamp bulb plasma cavity 30mm x 3mm ID for rare gas line source
2. Lamp bulb plasma cavity 30mm x 9mm ID for optically thin line sources (O, N, Cl, H, etc.)

Integrated Units:

1. Includes lamp bulb in housing with EMI shielded exciter/controller, 2.75 in CF adapter, wall plug power supply
2. Input power (Voltage/Power) 8 to 35V/0.3 to 20W
3. CW or pulsed operation DC to 10kHz

Environment:

Case temperature range 0 to +55 degrees C

Running Life:

Min. 1500 hrs > 4000 hrs typical

Stability:

Max drift of +/- 1% per hr < +/- 0.2% per hr typical

Calibration:

1. Absolute intensity determined by photoionization or by traceable NBS standard
2. Spectrum of entire UV region

Accessories:

1. Short adapter for wide angle output
2. Lens assembly
3. Modulator
4. UV diodes
5. PSD and Pulse counting
6. Detector assemblies

Special Options:

1. Space qualification
2. Miniature low power configurations
3. High flux, high power configurations

Operating Instructions:

***** WARNING EYE HAZARD*****
Do not look directly at the lamp plasma unless wearing glasses. Normal eyeglasses will block extreme UV of all lamps except Mercury and D2. For these lamps use special UV blocking glasses.
***** WARNING AVOID DAMAGING WINDOW SEAL *****

Do not use chloroform, acetone or xylene to clean the lamp window. Use of these (or similar based solvents) might dissolve the window or the window seal.

Step 1: CLEAN THE LAMP WINDOW
Inspect the front of the lamp window and clean it, (following the lamp window cleaning instructions) if contamination is suspected.
Step 2: MOUNT THE LAMP
Ensure that the lamp is properly mounted.
(see mounting and heat sinking instructions in Appendix A).
Step 3: POWER UP THE LAMP
Plug power supply into the wall at 110 volts. Plug the lamp into the power supply. If the lamp does not light or problems occur refer to the troubleshooting guide.

LAMP WINDOW CLEANING INSTRUCTIONS

The lamp window is polished lithium fluoride, magnesium fluoride, sapphire or quartz and its vacuum ultraviolet transmission will be degraded if it is touched or otherwise contaminated. In all but the best vacuum systems a slow loss of window transmission will result from photopolymerization of organic materials on the outside window surface. Both of these problems may be overcome by proper cleaning of the window. A small bottle of polishing powder and cotton tipped applicators are included with the lamp unit.

Inspect the window for any signs of gross contamination, such as fingerprints. If there are signs of this, first clean the window with polishing powder (aluminum oxide) following the instructions below.

All cleaning operations are carried out with cotton tipped applicators or with lint free Kimwipe tissues. Apply the polishing powder to an applicator tip and gently polish the window. Repeat until there is no evidence of a film on the window when it is viewed with reflected light. Wipe away excess powder with a dry applicator. A few specks of powder on the window will have a negligible effect on the optical transmission. The final bits of powder may be removed by directing a stream of ultra-high-purity helium, nitrogen or argon across the window. Never use a lab source of air for this process because it may contain compressor oil.

TROUBLE SHOOTING GUIDE

Symptom	Remedy
Lamp intensity appears to go down	This is often caused by contamination of the lamp window. Clean the window according to the window cleaning instructions in section 2.
Lamp does not start	Often after sitting for a while the lamps are hard to start. Repeat the starting procedure until the plasma strikes.
The lamp current checks out but the lamp still does not start.	As a last resort you can start the lamp by holding a Tesla coil in the vicinity of the lamp window. Be VERY CAREFUL that the coil does not arc to the window or lamp can as this can damage the window, the lamp electronics, and even the power supply.

MOUNTING AND HEAT SINKING

We recommend operating the lamps either with the heat sink (supplied with later units) or with a small cooling fan. While this is not necessary for lamp operation in normal lab conditions, cool operation will enhance the spectral purity of the lamp output and prolong the life of the electronics.

MOUNTING NORMAL LAMPS

Refer to the configuration drawing above. The normal way to mount the lamp is with a 2.75 inch CF type flange. The lamp has been vacuum tested and should bolt onto a standard flange or adapter. The lamp is designed to operate on High Vacuum equipment at pressures <10(-7) torr. The seals in the mounting adapters are viton and should operate to vacuums <10(-9) torr. The lamp is not designed to be bakable to more than 100 C. Precautions should be taken to ensure the lamp does not get heated above this temperature during system bake-outs. Also, the lamp should not be operated during system bake-outs.

MOUNTING RE-ENTRANT TUBE LAMPS

The re-entrant tube lamp is mounted on a 2.75"conflat type flange. The general mounting directions for Normal lamps above apply to the re-entrant style lamps as well. This flange can be bolted directly onto the vacuum system using copper or Viton gaskets. The depth may be adjusted in the following manner:

i) When the chamber is at atmospheric pressure, loosen the clamp rings.
ii) Using a rod inserted into the holes in the side of the o-ring retaining ring, unscrew the ring to loosen it. The lamp will now slide.
iii) Adjust to the desired depth and tighten the o-ring retaining ring.
iv) Adjust the clamping ring so it is flush against the o-ring retaining ring.
v) Tighten the clamping ring.

Frequently Asked questions

Temperature Sensitivity (lamp, detector, electronics, window, and spectral lines)

Lamp temperature sensitivity

The key parameter for lamp control is the temperature of the oxygen furnace which is controlled. Temperature drift is not an issue here.

Detector temperature sensitivity

The detector is a vacuum photoelectric diode which has a dark current much lower than the smallest signals.

Electronics temperature sensitivity

Since the measurement is a ratio, temperature drift is not an issue for the electronics unless a significant offset is generated by heating. We have not found this to be the case.

Windows temperature sensitivity

Temperature drift of the optics is possible since fluoride crystals exhibit decreased transmission with increasing temperature. This may come about when heating occurs from O atom recombination on the crystal surfaces. The O 130nm line is quite a bit higher than the Magnesium Fluoride cutoff and it is known that the temperature effect decreases as one gets away from the band gap energy. This effect was not seen in an oxygen system that experienced significant heating from O atoms which suggests that this effect was not significant compared to the large absorption signal.

Spectral lines temperature sensitivity

One would predict that the absorbance by the atomic oxygen will change with the temperature of the gas. This relationship should go roughly as the square root of temperature since the line peak will decrease as Doppler broadening gets larger.

Base noise level, temperature sensitivity

The signals are large (10^{10} photoelectrons per second), therefore, the shot noise limit to the signal to noise ratio is on the order of 100,000. The main noise limit is the preamplifier which limits the SNR of the ratio to about 1000. However, the changes in absorbance we are looking for are many percent so the limitation on precision seems to be less than a percent. Systematic signal drift seems to be a much more important issue.

We observe the baseline signal drift which appears to result from changes in the window transmission. This drift seems to be related to window contamination and subsequent cleaning due to heating or oxygen bombardment. This drift seems to be fairly systematic and can be at least partially compensated for by switching the plasma on and off and tracking the baseline changes.

Gas pressure Line broadening and scattering of light

Pressure broadening is smaller than Doppler broadening in the low torr region. Line broadening due to temperature is a root T effect and will decrease the sensitivity and increase the dynamic range of the system. For example, a change from 1000 to 2000 K should decrease the effective cross-section by a factor of 0.707. This seems like it could be an issue if the temperature is expected to vary widely with the conditions.

Multiple scattering of lamp light

As the O atom density increases the multiple scattering (which can scatter lamp light back into the optical beam) can be an issue. However, multiple scattering models by the author indicate that for absorbance in the 50 percent range this effect is small.

Scattered light from AO generator plasma

Scattered light from the plasma is not an issue since the detector signal is processed by subtracting lamp off from lamp on signals.

Interference by other gases

Our experiments indicated that interference effects occurred from wall or window changes. For the most part, these effects seemed transitory and were much smaller than the oxygen signals.

Some gases seemed to have significant molecular gas phase absorption as well but these effects were less than 3 percent of the oxygen signal. Also, the addition of some gases

seemed to increase the efficiency of the O generation. This is due to decrease in the re-combination of O on the walls which is a well known effect.

Oxygen lamp lifetime and failure modes

The light source has a lifetime limited by the depletion of the oxygen source and or absorbing getter. This lifetime is ~1,000 hours. This is likely much more than a year since the method can determine the oxygen density very quickly and the lamp can be turned off.

The lamp can fail if the heater overheats because of an electronic malfunction. The lamp bulbs themselves are very robust, having flown on rockets and having been used in labs for many years. Leaks are unlikely because of the hard seal of the window and lack of bulb feedthroughs (electrodeless). The large getter maintains the gas in the lamp in a pristine condition.

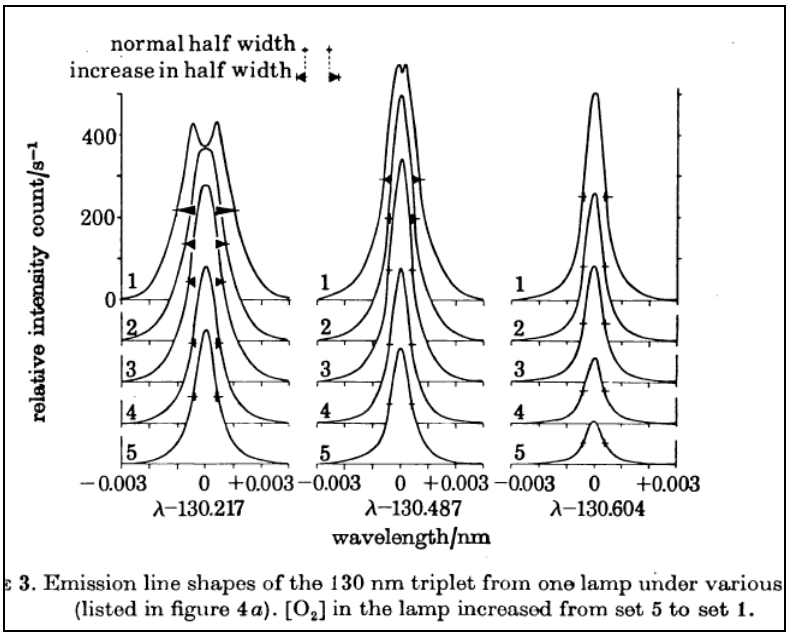
Geometry

The geometry requirements are set by the level of atomic oxygen which is determined by the power of the AO generator and the recombination of the walls. For situations where the atoms are near the target a path of a few centimeters is usually fine. For situations where the atoms are in the delivery pipe, the O density can be quite high and the path may have to be made quite small (~ 1 mm). In all cases the integrated O atoms across the path are measured and the average density can be found by dividing the atoms per cm2 by the path length in cm.

Lamp line profiles

As seen in the figure below (excerpted from Dickenson) the Doppler lamp line width is approximately 0.0008 nm. This is good since the absorption line width will encompass the lamp line width even if the lamp is somewhat optically thick. The Resonance lamp is typically set to curve 5 in the diagram below, which is much narrower than the absorption line in a high power set-up.

In low power setups the gas temperature is still going to be higher than room temperature in the oxygen beam. The actual temperature will depend on the degree of thermalization of the atomic oxygen after dissociation in the plasma and the starting temperature of the oxygen atoms. Typically, even in plasma of a few Watts, the temperature of the O atoms will be 50 to 150 K hotter than room temperature. Thus, the atomic oxygen absorption profile will be larger than the lamp profile which will make the absorption measurement insensitive to small changes in lamp temperature.



ε 3. Emission line shapes of the 130 nm triplet from one lamp under various (listed in figure 4*a*). [O_2] in the lamp increased from set 5 to set 1.

Figure 4 Lamp spectral line profiles measured on a lamp built by Author

The stability issue is a temperature stability issue since temperature will directly change the absorption line width. The lamp stability is important since the atomic oxygen in the lamp will affect the line shapes and ratios. Resonance lamps have been shown to be very stable over long periods of time owing to the dynamic equilibrium between the O source and getter. Resonance lamps were developed for making upper atmospheric measurements of AO, an application which is much more critical than the lab AO generator application, relying on knowledge of the absolute line shapes of the lamp and the atmosphere. The published body of literature on atmospheric application supports the notion that this method is more than adequate for the AO generator characterization.

Data Processing

The raw data is acquired by subtracting lamp off from lamp on signals at a frequency of about 100 Hz. The signals are subtracted in a microprocessor in the blue power supply box which then sends signals to a laptop via a USB port. Two other options that would reduce the system's complexity are available:

1. Use the microprocessor in the lamp to modulate the lamp and process the CsI diode signal. This would eliminate the need for an external signal processor. Data could be sent to a palmtop, laptop or desktop PC via the USB interface.
2. To take a lamp sync signal out of the lamp along with the signal from the CsI system and preamp into a low cost A to D converter that feeds the host computer. Then LabView might be used to process the signal, eliminating the external signal processing electronics except for the preamp and A to D.

It also might be more cost effective to use a PMT instead of the diode. A PMT is an ideal amplifier which eliminates the need for a sensitive preamp. The higher SNR could make a much smaller diameter optical path which would reduce the diffusion problem.

References:

http://elchem.kaist.ac.kr/vt/chem-ed/spec/atomic/aa.htm

P. H. G Dickinson et al, *The determination of the atomic oxygen concentration and associated parameters in the lower ionosphere*, Proc. R. Soc. Lond, A 369, 370-408 (1980)