Sulfur Dioxide Monitor Instrument Handbook

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## Acronyms and Abbreviations

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<tr>
<td>AAF</td>
<td>ARM Aerial Facility</td>
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<td>AMF</td>
<td>ARM Mobile Facility</td>
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<td>AMF1</td>
<td>first ARM Mobile Facility</td>
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<td>AOS</td>
<td>Aerosol Observing System</td>
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<td>DQPR/DQR</td>
<td>Data Quality Problem Report/Data Quality Report</td>
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<td>SGP</td>
<td>Southern Great Plains</td>
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<td>SO₂</td>
<td>sulfur dioxide</td>
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1.0 Instrument Title: Sulfur Dioxide Analyzer

Sulfur Dioxide (SO$_2$) Analyzer. Instrument is pictured in section 4.0, and more information is available at the manufacturer’s website.

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1-508-520-0430

4.0 Instrument Description

As of 2015-11-22, Thermo Fisher Scientific Sulfur Dioxide Analyzer instruments are deployed in the AAF, MAOS-C and SGP AOS (the SGP unit is scheduled for activation in late spring of 2016).
Figure 1. Model 43i trace level-enhanced pulsed fluorescence SO$_2$ analyzer (AAF, AMF2, AMF3, ENA, and MAOS C) (from sulfur dioxide manual).

The Sulfur Dioxide Analyzer measures sulfur dioxide based on absorbance of UV light at one wavelength by SO$_2$ molecules which then decay to a lower energy state by emitting UV light at a longer wavelength. Specifically,

$$\text{SO}_2 + h\nu_1 \rightarrow \text{SO}_2^* \rightarrow \text{SO}_2 + h\nu_2$$

The emitted light is proportional to the concentration of SO2 in the optical cell.

External communication with the analyzer is available through an Ethernet port configured through the instrument network of the AOS systems. The Model 43i-TLE is part of the i-series of Thermo Scientific instruments. The i-series instruments are designed to interface with external computers through the proprietary Thermo Scientific iPort Software. However, this software is somewhat cumbersome and inflexible. BNL has written an interface program in National Instruments LabView that both controls the Model 43i-TLE Analyzer AND queries the unit for all measurement and housekeeping data. The LabView vi (the software program written by BNL) ingests all raw data from the instrument and outputs raw data files in a uniform data format similar to other instruments in the AOS and described more fully in Section 6.0 below.

Modifications for the instrument include:

1. Addition of a 47-mm x 5-µm Teflon filter on the inlet line.
2. Internal plumbing changes to allow filtered (to remove sulfur dioxide) ambient air to be used for zeroing the instrument.

3. Addition of an external source of calibration standard to be added to the inlet sampling manifold to measure the response of the instrument.

5.0 Measurements Taken

The primary measurement output from the Thermo Scientific Sulfur Dioxide Analyzer is the concentration of the analyte (SO₂) reported at ~1-s resolution in units of ppbv in ambient air. There is some ‘jitter’ in the reporting frequency and a true 1-s interval can have 0, 1 or 2 measurements reported. The values reported are stamped with the time (referenced to the site NTP server) they are received by the instrument computer. The final output stream, reports at a monotonic 1-s time base used in the AOS systems. If there are zero points reported in a 1-s interval, an empty field is assigned. When there are two points output by the instrument in a 1-s interval, the average of the two is assigned. Accompanying instrument outputs include sample temperatures, flows, chamber pressure, lamp intensities and a multiplicity of housekeeping information. There is also a field for Operator comments made at anytime while data is being collected.

6.0 Links to Definitions and Relevant Information

6.1 Data Object Description

6.1.1 Raw Data

The ‘raw’ instrument data stream outputs include all parameters measured by the instrument. Metadata are included automatically in each hourly file.

Metadata are now included in every hourly data file. The metadata are:

- Row 1: Filename
- Row 4 (col 1 only): ARM Climate Research Facility
- Row 5: SitePlatform
- Row 7: Last revised date
- Row 9: Instrument
- Row 10: Instrument Serial Number/ARM Inventory Number (WD#)
- Row 13: Instrument Mentor/Affiliation
- Rows 14-19: Comments (operational conditions, calibrations, etc.)
- Rows 21-24: Constants (usually defined in Comments)
- Row 35: Column title
• Row 36: Column units line 1
• Row 37: Column units line 2
• Row 40: First row of data

Data fields in the raw output begin on Row 40 and are:

• Date Time: Primary Date/Time stamp yyyy-mm-dd hh:mm:ss as set by the instrument computer and referenced to the site NTP server (or if unavailable, linked to the ‘master’ computer in the AOS)

• Inst. Time: Time set on the internal instrument computer hh:mm:ss. This should be equal to the primary Date/Time, but can vary if Operator has not set the instrument time. This value is usually discarded.

• Inst. Date: Date set on the internal instrument computer mm-dd-yyyy. Note this is not a standard format. This value should be equal to the primary Date/Time, but can vary if Operator has not set the instrument time. This value is usually discarded.

• Flags: %hhhhhhhh (eight hexadecimal digits) showing the state of the instrument. The meaning of individual flags is described in Figure B-1 of the vendor manual, shown here:

![Figure 2. Bit by bit meaning of status flag.](image)

• SO2: Mixing ratio (in ppbv) of sulfur dioxide corrected to STP (O°C, 1 atm) based on instrument measurement of temperature and pressure.

• Int. Temp.: Internal temperature of the instrument (°C)

• React Temp: Temperature inside the measurement cell (°C). Used to correct measurement to STP.
• Pres: Pressure inside the measurement cell (mm Hg). Used to correct measurement to STP.
• Flow: Sample flow (LPM). Since fluorescence detectors are concentration sensitive, flow measurement is only an indication of transit time through the instrument.
• PMT volt: Cathode voltage of the photomultiplier tube. Changing this value does change the response sensitivity of the instrument.
• Lamp volt: Voltage driving the flash lamp used to excite SO₂ molecules.
• Lamp int: Lamp intensity (%). Normally about 90% but will decrease over time as the lamp ages.

The above signals are reported at nearly 1 Hz. The next signals change infrequently and are reported much less often by the instrument.
• Avg Time: Averaging time (s). Normally set to 1.
• SO2 Bkg: Background signal used by instrument for zeroing. Never changed. Chemical zero is used to find background for subtracting.
• SO2 Coef: Slope used for calculating instrument response. Never changed. Standard additions are used to find real-time instrument response.
• Sp conc: Span concentration (unused in AOS systems)
• Acg int: Automatic gain control of reference channel (%). An early coding error mistyped the header for this stream as acg instead of the correct agc. To preserve the body of processing code, this error has not been corrected.
• Range: Range of the analog output (ppbv). Since the analog output is unused in AOS systems, this parameter is irrelevant.
• Lamp setpoint: Internal setting of the flash lamp. This is adjusted whenever the flash lamp is replaced.
• Diag Volt mb 24: Diagnostic point for the 24V PS on the motherboard
• Diag Volt mb 15: Diagnostic point for the 15V PS on the motherboard
• Diag Volt mb 5: Diagnostic point for the 5V PS on the motherboard
• Diag Volt mb 3.3: Diagnostic point for the 3.3V PS on the motherboard
• Diag Volt mb -3.3: Diagnostic point for the -3.3V PS on the motherboard
• Diag Volt mb 24: Diagnostic point for the 24V PS on the measurement interface board
• Diag Volt mb 15: Diagnostic point for the 15V PS on the measurement interface board
• Diag Volt mb -15: Diagnostic point for the -15V PS on the measurement interface board
• Diag Volt mb 5: Diagnostic point for the 5V PS on the measurement interface board
• Diag Volt mb 3.3: Diagnostic point for the 3.3V PS on the measurement interface board
• PMT status: State of the PMT (on/off). This must be on for operation.
• Test LED: State of a test LED in the optical chamber (on/off). Provides a crude test of PMT operation. This must be off for operation.

• Flash lamp: State of the flash lamp (on/off). This must be on for operation.

• Temp Comp: Whether data are numerically converted to conditions of 0°C. This should be on for AOS operation.

• Pres Comp: Whether data are numerically converted to conditions of 1 atm. This should be on for AOS operation.

• Purge: Status of ’purge’ mode. Not used in AOS operation and should be off.

• Gas Units: Units concentration is reported (PPB for AOS operation).

• Gas Mode: Operational mode of the instrument (sample/zero/span)

• Comment: This field is the last field in the data stream. It allows operators to enter free form text from the Graphical User Interface at any time. Operational notes or disruptions may be entered here.

These data are save unaltered from what is produced by the instrument. Processing of the raw data must be able to deal with more than 1 record per second and time periods with either no data or only a date/time stamp in the record. If the instrument does not put out a number, the Instrument Computer can include a record of empty fields. Since neither the instrument clock nor the instrument computer clock are perfect, minor irregularities (dithering) in the output data stream can occur.

6.1.2 Mentor QA/QC’d Data Stream

Mentors provide data that has been processed from the ’raw’ data stream. In general data is delivered in 1-month chunks (for the AAF, the division is by flight). For each month (or other period) three files are produced. The naming convention is:

[site][platform][subsite].[species].[resolution].[version].[date].[time].[processing level].[delimiter]{.log.txt | .plots.pdf}

Where:

• [site]: The site of the measurement (usually the 3-letter specifier of the nearest airport)

• [platform]: The structure used for the instrument (aos | maosa | maosc)

• [subsite]: The subsite of the sampling site (m1 : main site | s1 : supplemental site)

• [species]: Species measured – for this instrument this is ‘SO₂’

• [resolution] [xxx]: (xx: two significant figures, s: time units string – s = seconds, m = minutes, h = hours, d = day, w = week, M = month) Typically this is always s or m

• [version]: Version of this data. Always use the highest version number with this name

• [date]: Date of first point in file (yyyyymmdd)

• [time]: Time of first point in file (hhmms)
[processing level]: This is either ‘raw’ for ‘raw’ data or ‘m02’ for Mentor QA/QC’d data with only ambient measurements (zeros and calibrations removed), vetted and all appropriate calibration factors used in processing.

[delimiter]: Typically this is ‘tsv’ for tab-separated values, but could be ‘csv’ for comma separated values.

{".log.txt | .plots.pdf}: Optional extension. In addition to the data file, Mentor prepares a .log.txt file containing explanations of processing unique for this time period and a plots.pdf file with time series plots of processed data.

The data for this instrument (both .raw and .m02) follow the file structure used for most DOE/BNL/AOS instruments:

File Structure:
- Row 1: Filename
- Row 4 (col 1 only): ARM Climate Research Facility
- Row 5: SitePlatform
- Row 7: Last revised date
- Row 9: Instrument
- Row 13: Instrument Mentor/Affiliation
- Rows 14-19: Comments (operational conditions, calibrations, etc.)
- Rows 21-24: Constants (usually defined in Comments)
- Row 35: Column title
- Row 36: Column units line 1
- Row 37: Column units line 2
- Row 40: First row of data

Raw data now (beginning in 2014) have all the Meta data described above. Data is given in an arbitrary number of columns. The first column is date/time

- Time: Time is reported in UTC as set by an NTP server. Following convention, the time is the beginning of the period. The parameter reported at this time is the average of all points >= the time and < the next time. Data are reported at 1- s resolution. All non-operational periods have been removed (empty field or NAN). As follows convention, data are reported as tab-delimited ASCII files. Files are formatted such that they are self documenting.

Data Columns (for Mentor QA/QC data):
- Column 1: Date Time – All times are in UTC as yyyy-mm-dd hh:mm:ss{.ss} and are the beginning of the time period. All data reported here correspond to data from samples taken >= this time and < than this time + the data increment.
• Column 2: [SO2] – Mixing volume of SO2 in ambient air (no water vapor correction). This is this instrument’s primary measurement.

• Column 3: [SO2] – Column 2 with a running numerical average of 60-s (arithmetic mean of the present time point and the next 59 points).

6.2 Data Ordering

Sulfur dioxide data collected are distributed through the ARM Data Archive and are presently updated monthly.

6.3 Data Plots

Mentor provides monthly data plots of raw, housekeeping and final data sets. Typical plots, with explanations are shown below.

Figure 3. Raw sulfur dioxide data for 1 month.

The raw data plot shows all signal data recorded from the instrument. Processing software is used to parse the data into sampling and zero/calibration periods. Every midnight and noon UTC, the instrument goes
into an automatic zero check with a scrubbed sample air zero. Every 03:00 and 15:00 UTC, the instrument goes into an automatic span check. A blow-up of one of a zero period and a standard addition is shown here:

**Figure 4.** Typical zero and span check.

Sample periods are automatically delineated with time allowed for measurements to restabilize.
Figure 5. Housekeeping data for 1 month.

The chassis and optical cell temperatures are shown in the top panel. The cell pressure and flow are shown in the second panel. Cell temperature and pressure are used internally by the instrument to report sulfur dioxide concentrations at STP (1 atm, 0°C). The twice daily negative spikes in pressure are momentary and due to flow interruptions proximate to the zero checks. The photomultiplier tube voltage and the flash lamp voltage are shown in the bottom panel. Flash lamp voltage is automatically raised by the instrument as the lamp ages. Values above ~1300 V indicate lamp replacement is needed. Generally the lamp and the electronic ‘flash pack’ are replaced together.
Figure 6. Zero and span check stability over 1 month.

A 1-month record of zero and span checks are shown above. The dotted lines are the responses for lo and high level span checks calculated from the standard add concentration. The difference between the measured span and the calibrated span value is usually less than 15%. This difference is judged to be instability and drift in the dilution flow within the trace gas manifold and NOT in the measurement cell. Drift in the span response is monitored over long periods (> 1 year) and drifts of more than 10% indicate need for multipoint recalibration in zero air.

Finally, a plot is produced of the entire month (and also weekly plots) of the reported, ambient SO$_2$ concentrations adjusted for any baseline drift.
Figure 7. Processed data for 1 month.

This plot shows a typical month’s ambient data record. The black trace shows the unfiltered (~1-s) instrument record. The red trace shows data filtered to 60 s. The 2x daily spikes are due to a slight residual tailing from the standard addition.

6.4 Data Quality

The first level of data quality is automatic flagging of data when instrument changes states during zero and span checks. Normally this eliminates the first 50 seconds after the zero is actuated and then the first 270 seconds after a span check. The ‘centroid’ of each state is taken as an average of the ‘valid period,’ i.e., once a stable level has been achieved. Valid ambient samples are not taken until 250 seconds after a change in state.

The second level of data quality is inspection of the 2x daily zeros and span checks. A time series plot over the course of the month typically shows <3-5% relative standard deviation and minimal drift (<3-5%). Values greater than this indicate need for instrument recalibration with zero air by Mentor.
Under normal operation two factors can change the instrument flow rate: dirt accumulation on the inlet filter or degradation of the internal diaphragm pump. Given the 2-week filter change schedule, dirt accumulation is not observed to reduce flow. Pump life under continuous operation is 2-3 years.

The third level of data quality and assessment deals with visual inspection of the output data stream. Periods of instrument inoperation are identified and flagged. Either instrument failure or inlet failure cause such periods. In some cases Operators note occurrences, but more often it is up to the Mentor to recognize nonsensical values and determine the cause(s). The complete recording of all instrument and inlet housekeeping aids in this diagnosis. Except for pump/inlet failures and several instances where the inlet filter was replaced with an impermeable spacer (documented below), Mentors have not experienced any pattern of failures that could be algorithmically identified. Failures, their identification and remedy and the Mentor write up in the DQPR/DQR system tend to be unique individual events.

Local sources can in some instances be identified by referring to the wind direction measured at the point of sampling. HOWEVER, identification of local interference is quite subjective.

### 6.5 Calibration Data Base

The Thermo Scientific Sulfur Dioxide Analyzers are calibrated for response upon receipt from the Manufacturer. This is done by the Mentor at the Brookhaven National Laboratory. These results are tabulated by the Mentor and for inclusion into the OSS.

### 7.0 Technical Specification

#### 7.1 Units

The measured quantity of interest is the mixing volume of analyte. This is reported in units of parts-per-billion by the instrument.

#### 7.2 Range

The full range of this model is somewhat arbitrary. It extends well past conceivable ambient levels in a non-power plant location. All zero and span checks are done through the ambient inlet filter so should buildup on the filter cause SO$_2$ hold up or destruction, it would be obvious in the span checks.

#### 7.3 Accuracy

Calibrations are done 2 x day intervals with an internal zero and 2 x day span checks. These reveal short term (daily) and long term (annual) drifts. At present, the span check level is dependent on an accurate measurement of the dilution (the inlet manifold flow). The flow manifold does not have a data record and is normally measured by the Mentor during system integration at each site. The initial (on receipt) calibration is within 1-2% accuracy. However, field conditions vary substantially. Variation from the
original measurement standard would appear to be ~5-10%. The present (as of 2015-11-24) system of in-field calibration is being phased out by a new, more accurate system of standard addition.

### 7.4 Repeatability

Precision (repeatability) is given here as the noise of the 1-s signal. Under quiet ambient conditions, this has been measured as:

$$[\text{SO}_2] \sigma = 1 \text{ ppbv}$$

For the 60-s averaged data, the noise goes down approximately as $60^{-0.5}$

$$[\text{SO}_2]_{60\text{-s avg}} \sigma = 0.15 \text{ ppbv}$$

Further averaging is probably not useful as instrument drift and baseline uncertainty exceed this value.

Therefore, for normally distributed noise, $\pm 2\sigma$ encompasses 95% of the points. The precision of the instrument under average ambient conditions is then given as:

$$[\text{SO}_2]_{1\text{-s}} 95\% \text{ Confidence Interval} = \pm 2 \text{ ppbv}$$

$$[\text{SO}_2]_{60\text{-s avg}} 95\% \text{ Confidence Interval} = \pm 0.3 \text{ ppbv}$$

Note that these Confidence Intervals represent repeatability over a relatively short period of time. Day-to-day and month-to-month repeatability has a larger confidence interval and approaches the accuracy uncertainties given in the previous section. The Manufacturer reports zero noise as 0.25 ppb RMS (for a 60-s average) which is slightly better (smaller) than the values observed under field conditions and reported above.

### 7.5 Sensitivity

Sensitivity as a lower detectable limit is reported as 95% confidence interval above baseline which in this case would be 2 ppbv for data as reported to ARM (1 value per second). Assuming the noise decreases inversely with the square of the integration time, the measurement sensitivity under field conditions is similar to the 0.1 ppb ‘Lower detectable limit’ reported by the Manufacturer for a 60-s average.

### 7.6 Uncertainty

Uncertainty is an integral of all errors. It is a combined measurement of accuracy and precision (repeatability) discussed above.

### 7.7 Output Values

Described in Section 6.0
8.0 Instrument System Functional Diagram

Based on experience, the Sulfur Dioxide Analyzer is modified upon receipt. These changes are shown here:

Figure 8. Diagram of flow schematic (from Thermo Scientific Manual, Figure 1-1).
Figure 9. Modifications to the TEI Model 49i by mentor.

Red boxes indicate components that are removed and green boxes indicate components added. The original (as received) configuration is shown in the top and the current configuration is shown in the bottom panel. The changes are summarized as:

1. The 5-µm Teflon filter (47-mm diam) (not shown) is installed on the sample line.

2. Instead of supplying zero air to the analyzer, the instrument pump draws ambient air through a zeroing filter. The filter is a 47-mm paper filter impregnated with potassium carbonate and moistened with a humectant (Ref. 1).

3. The instrument driven calibration is available, but not used in the AOS systems.

The trace-gas inlet used in the AOS systems consist of a high-flow ½” o.d. PFA tubing sampling from under the aerosol inlet rain hat at ~10-m AGL. Air is pulled into the container at 30 LPM as controlled by a rotometer and the sum of instrument flow rates sampling from the manifold. The residence time to the back of the instrument is ~1-2 s.
Figure 10. Ground inlet schematic A (present installation in MAOS C).
The original installation in MAOS C uses a standard addition in the high flow, trace gas inlet. This adds a small quantity (~40 sccm) into the total flow of the trace gas manifold. The concentration seen by the instrument is thus this amount diluted by the sum of instrument flows plus the bypass flow. This value is measured experimentally upstream of the calibration tee. However there is at least a 10-20% uncertainty in this measured flow over the course of a typical IOP. For this reason a new calibration method is being retrofitted, beginning with the SGP AOS.
Figure 11. Ground inlet schematic A (for installation in SGP AOS).
9.0 Instrument/Measurement Theory

The following description is taken from the Thermo Scientific Manual:

“The sample is drawn into the Model 43 i Trace Level-Enhanced through the SAMPLE bulkhead, as shown in Figure 8 (Figure 1-1 of the vendor documentation). The sample flows through a hydrocarbon “kicker,” which removes hydrocarbons from the sample by forcing the hydrocarbon molecules to permeate through the tube wall. The SO$_2$ molecules pass through the hydrocarbon “kicker” unaffected.

The sample then flows into the fluorescence chamber, where pulsating UV light excites the SO$_2$ molecules. The condensing lens focuses the pulsating UV light into the mirror assembly. The mirror assembly contains eight selective mirrors that reflect only the wavelengths which excite SO$_2$ molecules.

As the excited SO$_2$ molecules decay to lower energy states they emit UV light that is proportional to the SO$_2$ concentration. The bandpass filter allows only the wavelengths emitted by the excited SO$_2$ molecules to reach the photomultiplier tube (PMT). The PMT detects the UV light emission from the decaying SO$_2$ molecules. The photodetector, located at the back of the fluorescence chamber, continuously monitors the pulsating UV light source and is connected to a circuit that compensates for fluctuations in the UV light.

As the sample leaves the optical chamber, it passes through a flow sensor, a capillary, and the “shell” side of the hydrocarbon kicker. The sample then flows to the pump and is exhausted out the EXHAUST bulkhead of the analyzer.

10.0 Setup and Operation of Instrument

Instrument is permanently installed in the AOS systems. This includes:

1. Physical mounting of the instrument in a shock-isolated 19” instrument rack,
2. Plumbing of the sample line into the fast flow ½” PFA trace gas manifold line with the associated 47-mm PFA filter and filter holder,
3. Connection of the RJ-45 ethernet output to the AOS Instrument Network switch, and
4. Connection of the 110 VAC power line to the appropriate Power Distribution Unit outlet.

Initialization involves only making sure the ½” PFA trace gas manifold line runs up the aerosol stack to under the 14” rain hat and turning on the power.

After power is turned on, the instrument goes through self checks and commences putting out data. Note that after extended shut down, this warm up period can be 5-10 minutes or more for temperature and lamp stabilization.
11.0 Software

A Graphical User Interface has been written by BNL for the Instrument Computer (‘brick’) acquiring data from the Sulfur Dioxide Analyzer. This GUI is similar to other AOS instrument GUIs. The zero and span checks are controlled by the GUI. (In MAOS C the standard addition is controlled separately by the TEI Model 149i calibrator).

12.0 Calibration

Calibration procedures are described earlier. These include 2 x daily zero and span checks.

13.0 Maintenance

Maintenance is minimal on this instrument. Mentor advises changing the inlet particle filter every 2 weeks. Filter is 47-mm diam. 5-µm PFA membrane filter Type LS (Millipore Catalog # LSWPO4700). Note that the filter is not directional (either side up). The filter is white and is packed in a stack separated by blue plastic spacers. DO NOT USE THE SPACER! USE THE WHITE FILTER. (This error has been made multiple times.)

Figure 12. 47-mm PFA filter holder.

Note the arrow showing flow direction.
The green filter holder wrenches were delivered with the instrument. One end goes over the orange locking ring and the other (smaller) end goes over the PFA body. When opening the holder note (and report) if the previous filter appears damaged. The filter being replaced should have at most a faint circle of trapped dirt. If the circle is visibly dark, increase the change frequency and notify the Mentor.

The ¼” PFA fittings on the ends of the filter have an integral ferrule in the nut (no separate ferrule needed). These are finger tightened, but should be quite snug on the ¼” PFA tubing.

The old filter may be disposed of in regular garbage.

### 14.0 Safety

Unit has no safety concerns during normal operation. The unit has a flash lamp that emits light in the UV and should not be viewed directly. If lamp operation must be verified, looking at the reflected flash will suffice. The internal instrument pump has an exposed shaft that drives the diaphragm on one end and a hard plastic fan on the other end. Both sides are open inside of the enclosure and pose a hazard to fingers. Older versions of this instrument had exposed electrical terminals on the pump motor. The current ARM version does not seem to have any exposed terminals with 110 VAC, but normal electrical procedures and cautions should be used. Instrument should not be operated with the cover off without proper training, precautions and approvals.

It has been observed that the PFA tubing inside the instrument can abrade even when rubbing against another PFA tube. Thus, all tubing must be strain relieved (with tie wraps) to prevent any rubbing.
15.0 Citable References
