

Wavelength Calibration, Overranges & Zero Order Troubleshooting Hints For The 4100 MP-AES

Stephen Anderson, September 2012



The Successful Wavelength Calibration

- Requires the following

1. Zero order location within ± 2 pixels of last position
2. Location of the following emission wavelengths within ± 1 pixels of the predicted position
 - Zn 213.857, Cd 228.802, Mn 257.610, Mn 279.482, Cu 324.754, Cu 327.395, Ni 352.454, Cr 357.868, Al 396.152, Sr 407.771, Sr 421.552, Ba 455.403, Ba 493.408, Ba 614.171, K 766.491, K 769.897
 - Signal/background > 20 for Zero Order & > 5 for Element Emission Lines
3. Wavelength Check
 - Uses zero order and a subset of the calibration wavelengths
 - **Zn 213.857** , Cd 228.802 , Mn 257.610 , **Mn 279.482** , Cu 324.754 , **Cu 327.395** , Ni 352.454 , Cr 357.868 , Al 396.152 , **Sr 407.771** , Sr 421.552 , Ba 455.403 , **Ba 493.408** , **Ba 614.171** , **K 766.491** , K 769.897
 - Must be located within ± 0.035 nm of the calibrated position

Checklist of Possible Root Causes of Wavelength Calibration Failure

- Incorrect wavelength calibration solution
- Failure to locate Zero Order or an Emission Line used in the calibration
- Monochromator not initialized
- Incorrect Software
- Damage to the instrument
- Monochromator drive motor problem

Zero Order Failure

- On Instrument start
 - Firmware looks for zero order
- On encountering the Zero Order error
 - Perform an Instrument Wavelength Calibration
 - Resets the Zero Order Position
 - Searches for predefined emission lines
 - Do not use Zero Order Check in the Instrument Calibration diagnostics
 - Only CHECKS the zero Order Position
 - Does not reset the Zero Order Position

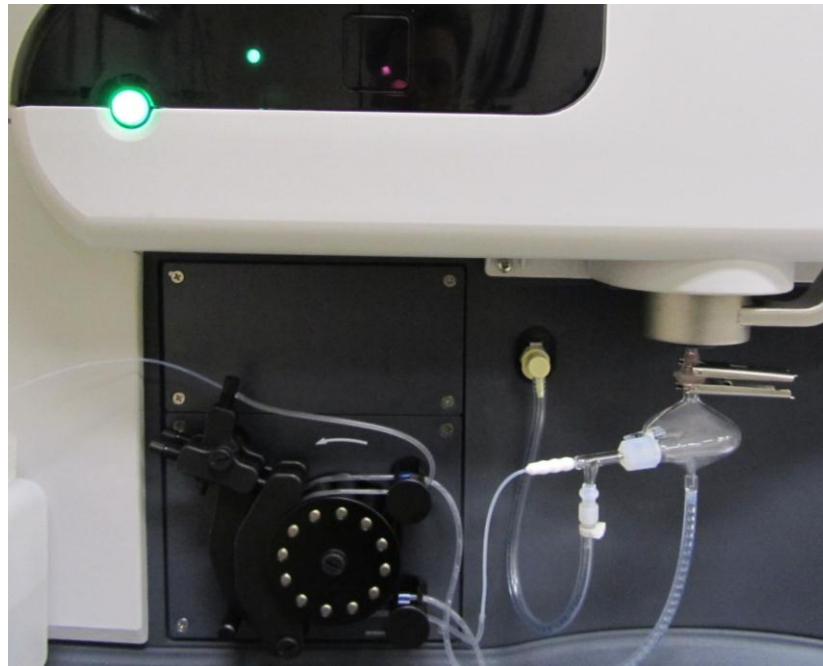
Emission Line Overrange or Peak Not Found

• Check Solutions

- Confirm reagent water meets ASTM Type 1 standard (18 M Ohm)
- Standards
 - Use ICP grade, or higher, standards only
 - Prepare fresh Test Solution using the Agilent Multielement Standard
 - Beware of standards prepared from single element standards
 - AA grade standards are not acceptable
- Acids
 - Confirm acids meet purity requirements (minimum Analytical Reagent grade)
- Solution Storage/Preparation Containers
 - Ensure storage/preparation containers are cleaned prior to use

Emission Line Overrange or Peak Not Found

- Check Sample Introduction System
 - Standard Glass Concentric Nebuliser
 - White/White Sample Pump Tubing
 - Blue/Blue Drain Pump Tubing
 - Single Pass Glass Cyclonic Spray Chamber



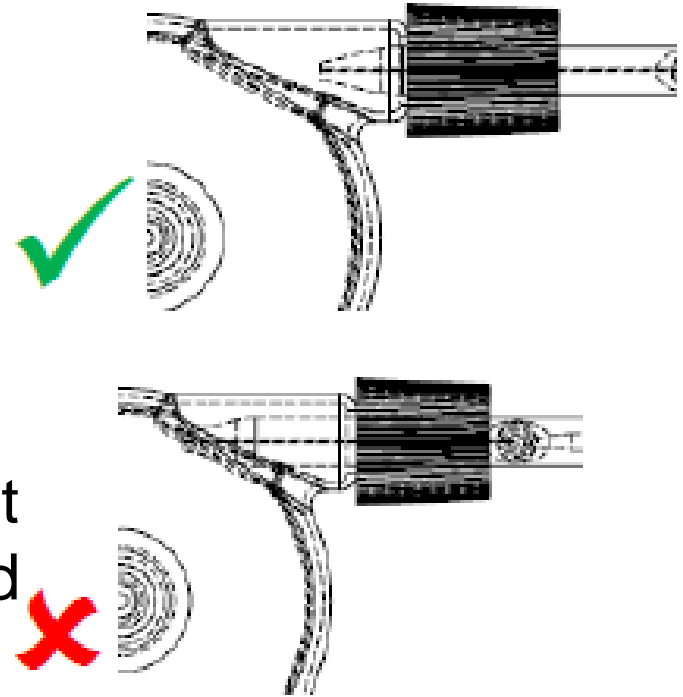
Nebulizer Position in the Spray Chamber

- Instrument performance is very sensitive to the nebulizer position in the spray chamber

- The more the nebulizer protrudes into the cyclonic action of the spray chamber the lower the sensitivity

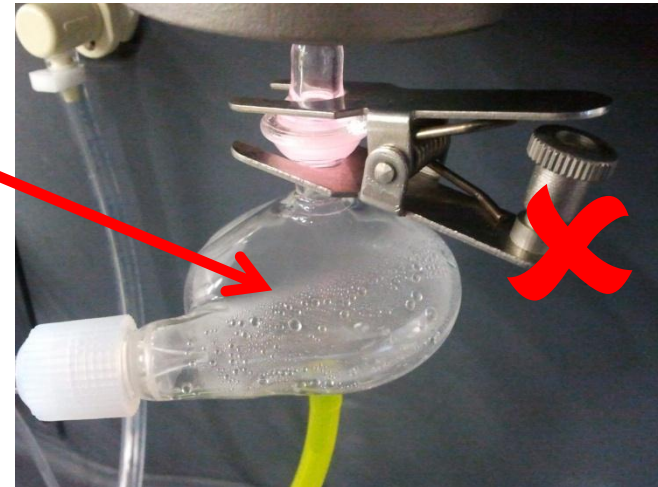
- Carefully retract the nebulizer and observe the effect on sensitivity

- DO NOT retract the nebulizer such that the nebulizer cannot be reliably secured in the spray chamber



Emission Line Overrange or Peak Not Found

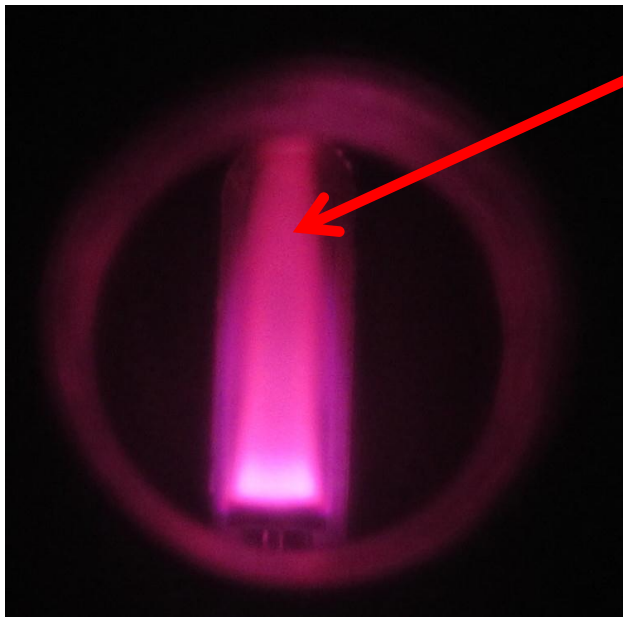
- Correctly wetting spray chamber should not have large visible droplets on internal surfaces
- If not wetting correctly clean with a laboratory grade detergent, 5%-10%
 - Problem spray chambers may need to stand overnight in the detergent solution
- After cleaning rinse the spray chamber thoroughly with Deionised water
 - Do not dry the internal surfaces of the spray chamber with compressed gases



Confirm Vista Tune Solution is being Transported to the Plasma

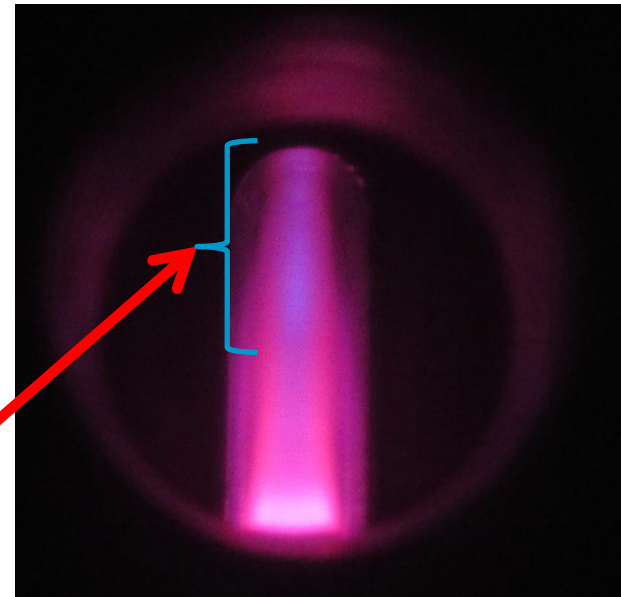
- Observe plasma, tip of the plasma should show a distinct BLUE colour (switching between blank and standard emphasises the difference)

Blank



No Blue Tip

Vista Tune Solution



Blue Tip

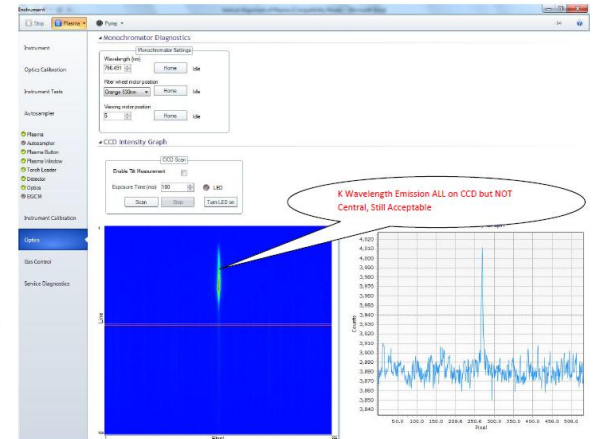
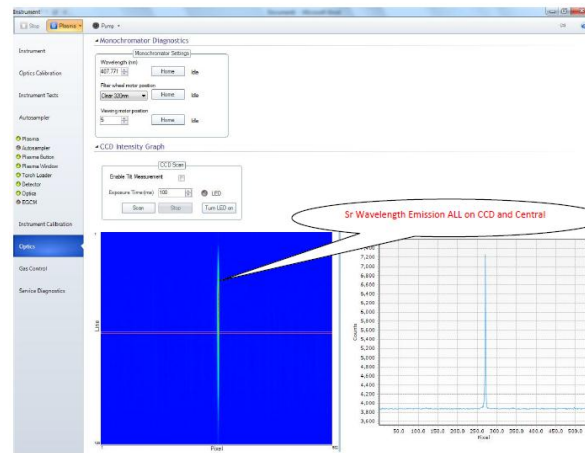
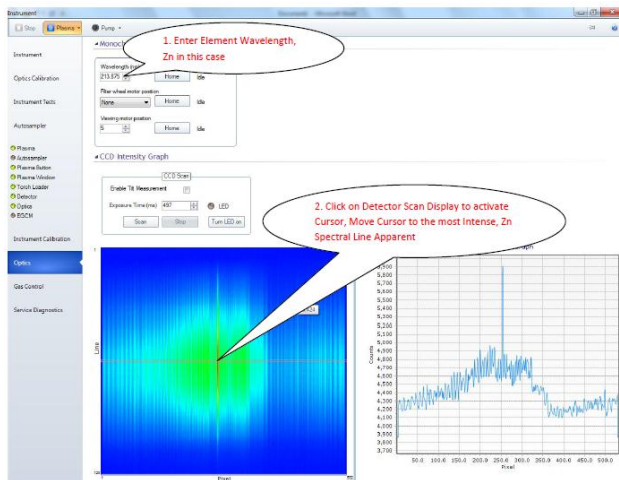
Confirm Detector Rotation

- Impacts Performance sensitivity

The screenshot displays the 'Instrument' software interface with the 'Optics' tab selected. The 'Wavelength (nm)' is set to 0.000, 'Filter wheel motor position' is 'None', and 'Viewing motor position' is 420. The 'CCD Intensity Graph' section is active, showing a 'CCD Scan' control panel with 'Enable Tilt Measurement' checked, 'Exposure Time (ms)' set to 10, and an 'LED' indicator. A 'Scan' button is visible. Below the controls is a 'CCD Line 62 Intensity Graph' showing a single sharp peak at approximately 260 pixels. A 'Pixel' axis is shown at the bottom of the graph. A 'Pixel' axis is also shown at the bottom of the main image area. Three callout boxes provide instructions: 1. 'After Detector Scan Completes Check Tilt Measurement' (pointing to the 'Scan' button), 2. 'Two New Cursor bars Appear Above & Below Main Bar' (pointing to the horizontal red lines in the main image area), and 3. 'For Non-Rotated Detector Metre Should be in the GREEN Region' (pointing to the green bar in the 'CCD Intensity Graph').

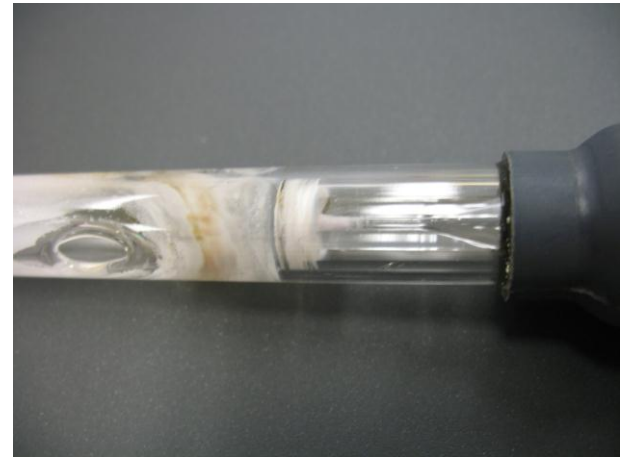
Confirm Vertical Alignment

- Impacts Performance sensitivity
- Check Low, Medium and High wavelengths
 - Usually (Zn 213.857), Sr (407.771) and K (766.491)
 - Confirm emission lines are completely on the CCD



Check Torch Condition

- Plasma torch should not have any cracks or large white frosted areas (devitrification)
- Ensure the gas sealing surfaces are free of particulate materials
- Ensure gas orifices are not blocked
- Install a new Plasma Torch



Clean the Plasma Viewing Window

- Plasma viewing window should NOT have any deposits or coatings
- Clean or replace

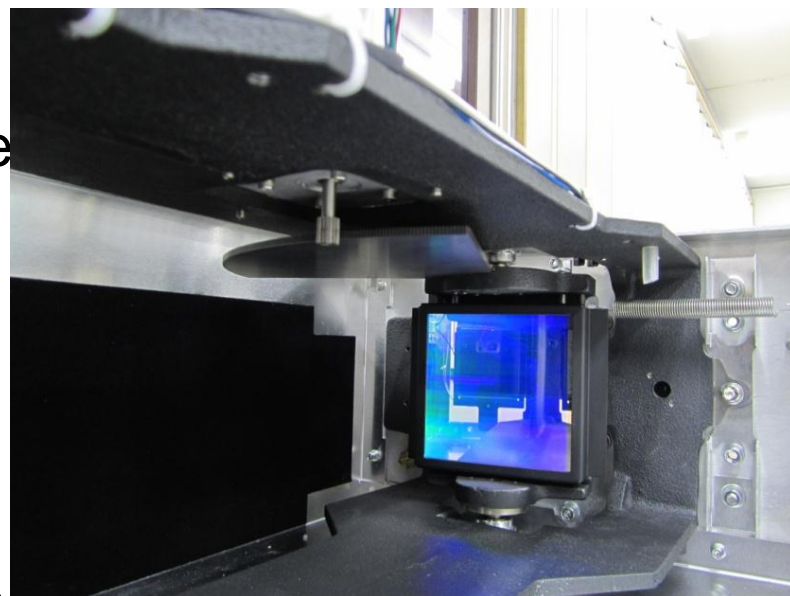


Monochromator not Initialized

- Instruments prior to serial # AU12250226
 - Important Monochromator Drive initialization procedure was not performed on some 4100 MP-AES instruments shipped from the factory
 - Service note [SN AA-263](#) details a field Monochromator Initialization procedure designed to replicate the factory initialization procedure and qualify the success of this procedure.

Inadequate Monochromator Stabilization Time

- Scanning of the Monochromator requires precision control
- Part of the control is a “stabilization” time which allows the grating drive components time to settle before beginning signal measurement
- Too short a stabilization time may cause OVERRANGES, resulting in a failed Wavelength calibration
- Some 4100 MP AES units have been identified to require a longer settle time
- MP Expert software releases post 1.0.2.43659 have the longer stabilization times.



Instrument Damage

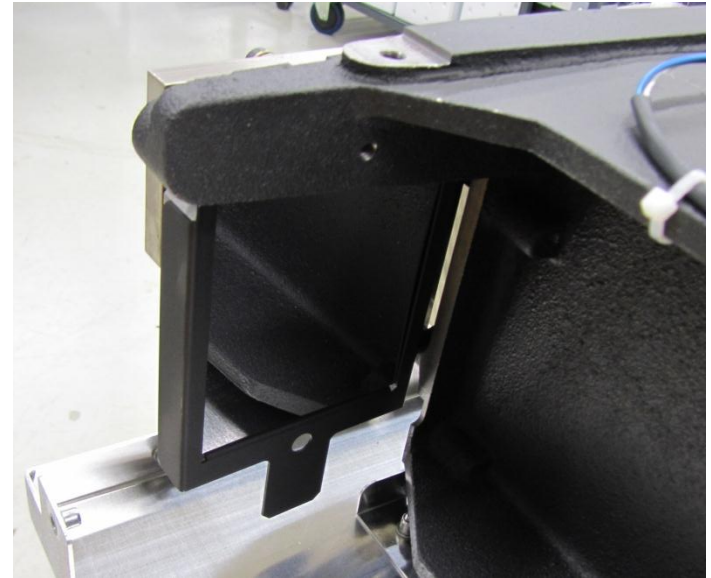
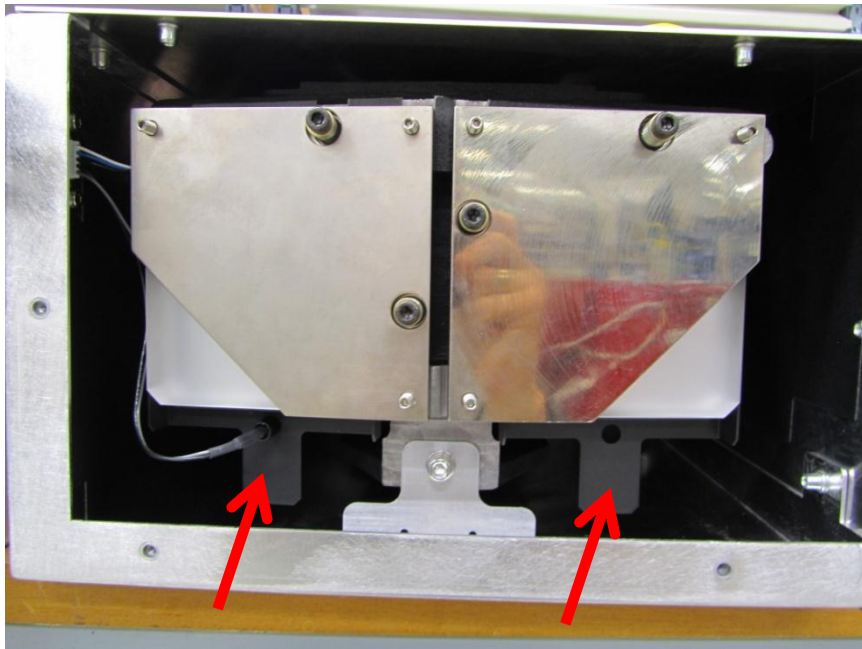
- If the following has been confirmed
 - Solution used to Calibrate the instrument is correct
 - Sample introduction system is correctly configured and performing
 - The latest software is installed
- Suspect a hardware problem
 - This may be a consequence of
 - Inappropriate handling resulting in damage to the instrument
 - Component failure or wear

Possible Hardware Problems

1. Collimating or Focus Mirror Mask dislodged
2. Monochromator drive Tension Spring detached
3. Monochromator Drive Motor not correctly secured to casting
4. Transit/installation damaged monochromator support bracket
5. Faulty Monochromator drive motor

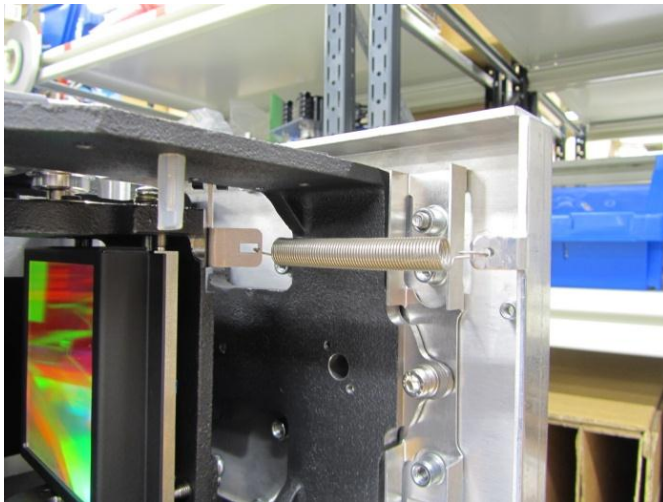
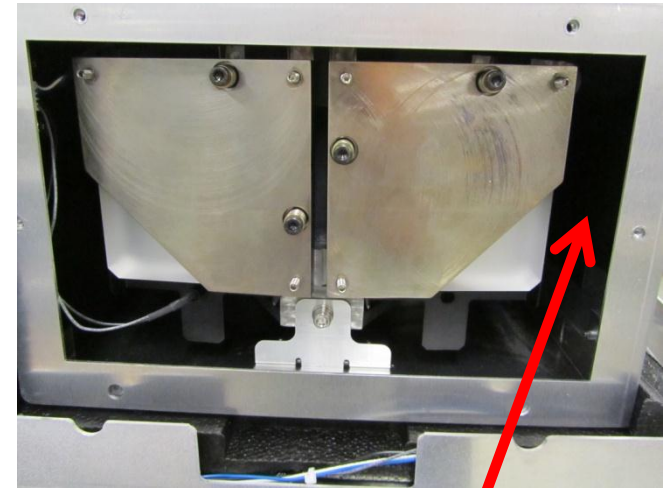
Mirror Masks Detached

- Remove Monochromator rear panel and confirm mask location
- To reinstall mask requires removal of the monochromator covers
- Ensure dust free environment before removing Monochromator covers



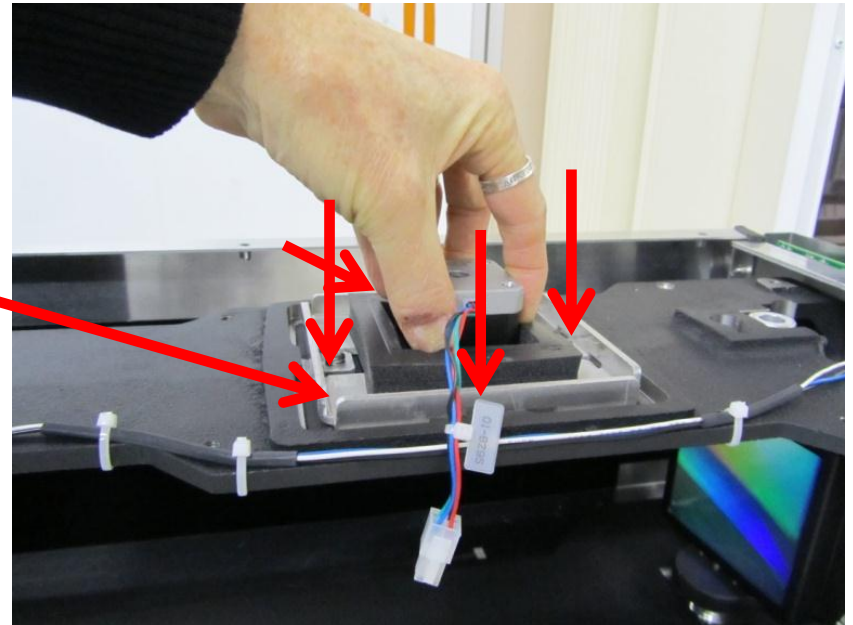
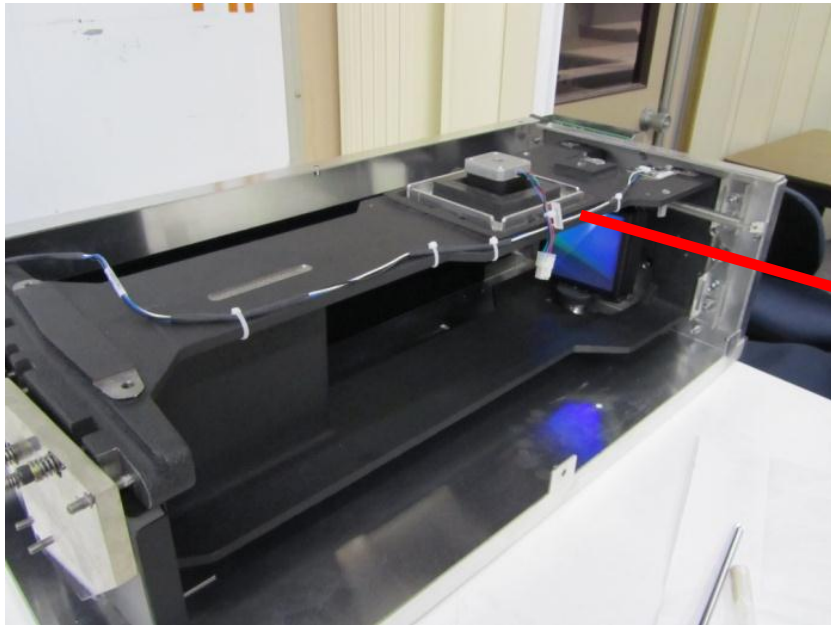
Monochromator Drive Load Spring Detached

- If correctly connected the spring should be visible at the grating end of the Monochromator by removing the back panel to the Monochromator
- If not visible reattach, requires Monochromator cover removal
- Ensure dust free environment before removing Monochromator covers



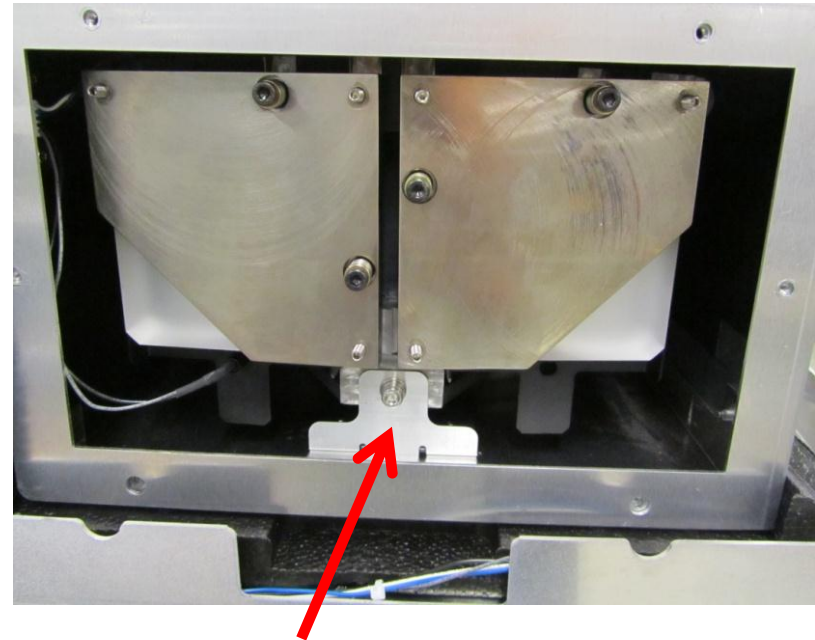
Monochromator Drive Motor not Secured

- Check the motor is firmly affixed to the Monochromator casting by the 4 mounting screws
- Requires removal of Monochromator covers
- Ensure dust free environment before removing Monochromator covers



Damaged Monochromator Support Bracket

- When subjected to sudden excessive forces, the rear Monochromator support bracket has been known to suffer significant distortion
- Remove the rear Monochromator panel and inspect the bracket
- Ensure dust free environment before removing Monochromator covers
- If the bracket is distorted consider Monochromator replacement



Faulty Monochromator Drive Motor

- Some 4100 MP-AES units may have suspect Monochromator drive motors
- If the Monochromator Initialization procedure in [SN AA-263](#) does not register a Complete within the recommended number of cycles, replace the Monochromator drive motor. Please contact the factory 4100 MP-AES PSE for further details

