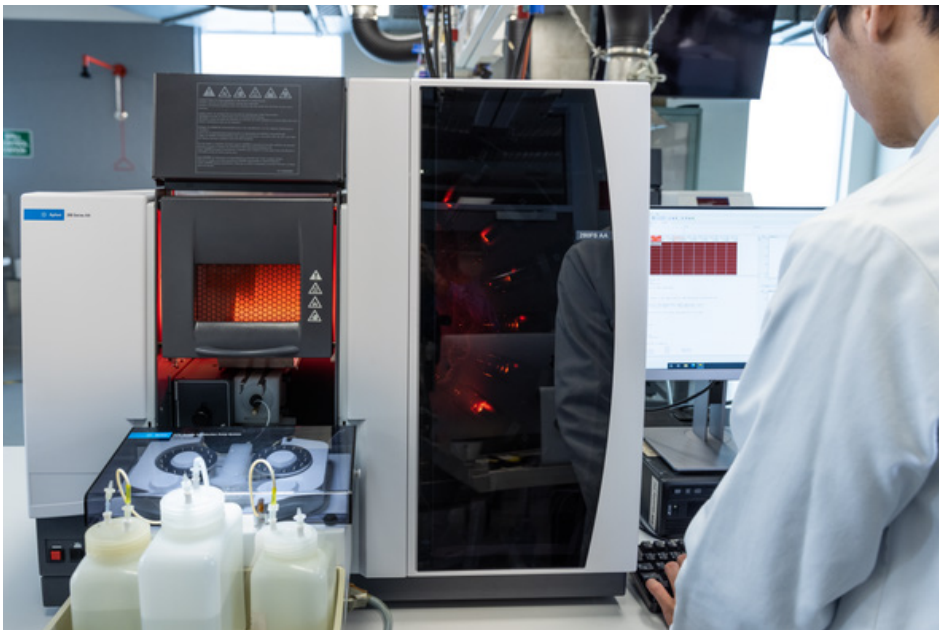
Technical Overview

Agilent
Trusted Answers

Health Checks for Flame and Graphite Furnace AAS



Good Laboratory Practices, quality control methods and other regulatory requirements may require proof that your atomic absorption (AA) spectrometer is working to specification. But you may also like to check this just for your own peace of mind. This procedure outlines test procedures that you can use at any time to demonstrate that your flame AA or graphite furnace AA instrument is operating correctly.

Flame AAS Health Checks

Background:

This test checks the performance of the burner and sample introduction system. Specifically, the test examines the sensitivity and precision of the system with the nebulizer optimized for maximum signal using the air/acetylene burner.

The test is performed using a copper lamp at the 324.8 nm emission line. A detergent solution is aspirated for 60 seconds, followed by distilled water, followed by a 5 mg/L Cu standard solution. The nebulizer uptake rate, absorbance and precision are measured using 10 replicates of 5 seconds integration with the burner position, nebulizer and impact bead optimized for maximum signal.

If routine maintenance is required before completing these tests, refer to the Maintenance schedule section of the online Help for guidance on daily maintenance tasks and cleaning procedures.

What you will need:

- An Agilent copper (Cu) hollow cathode lamp (coded or uncoded)
- A 5 mg/L copper (Cu) standard solution
- A dilute (1%) detergent solution
- A 0.1% nitric acid rinse solution
- Distilled de-ionized water
- An air/acetylene burner
- A 10 mL graduated measuring cylinder

Test procedure:

1. Develop a worksheet for this test. Select the flame method for Cu from the cookbook.
2. Click “Edit Methods...” Check that the sampling mode is set to manual, the measurement mode is set to Integration and set 10 replicates of 5 seconds duration with a pre-read delay of 5 seconds.
3. Click OK to Save the method changes.
4. Install the Cu hollow cathode lamp. Ensure that the lamp position in the Cu method matches the position where the lamp is located.
5. Click on the Analysis tab and Press the Optimize button to go to the Optimization display.
6. Select the Cu method from the list and click OK. Verify that the Cu lamp is switched on.
7. Ensure that the optical path through the sample compartment is free of obstructions. Optimize the lamp alignment by slowly turning each of the adjustment knobs on the lamp holder in turn to maximize the signal bar, rescaling the display where necessary. Once the lamp alignment is complete, record the % Gain displayed and compare this with the expected value.
8. Press the Optimize Signal button.
9. Check that the mixing paddles are fitted inside the spray chamber. Disassemble the spray chamber and re-install the mixing paddles if required.
10. Verify that the liquid trap is filled with water.
11. Verify that a 30 cm length of the wide bore capillary tubing (part number 9910024800) is connected to the nebulizer.
12. Verify that the air/acetylene burner is fitted.
13. Press the Ignite button to light the flame. Allow the burner to warm up for at least 10 minutes.
14. Aspirate the nitric acid rinse solution and perform an Instrument Zero.
15. Aspirate the 5 mg/L copper standard solution and adjust the nebulizer, burner position and impact bead to achieve the highest absorbance signal.
16. Aspirate distilled de-ionized water and use a measuring cylinder to measure the volume of solution aspirated by the nebulizer in one minute. Record the nebulizer uptake rate (mL/min.).
17. Press OK and then Cancel to close the Optimization display.
18. Aspirate a detergent solution for 60 seconds, followed by distilled de-ionized water.
19. From the Analysis window, click on Sample 001 and press Read.
20. Ensure the distilled de-ionized water is aspirated for the Instrument Zero reading. When prompted, aspirate the 5 mg/L copper standard solution.Record the Mean Absorbance and %RSD of the readings obtained for the 5 mg/L copper standard solution.
21. At the end of the reading, aspirate distilled de-ionized water for at least 10 seconds.
22. Press Stop to exit the Read mode and then press the Flame Off button to extinguish the flame.
23. Compare the readings obtained for the 5 mg/L copper standard solution with the expected results. If the achieved results do not meet the expected result, refer to the Troubleshooting section below for suggested corrective actions. Retest as appropriate. If the expected results still cannot be obtained after trouble shooting and retesting, a service call may be required.

Expected performance:

% Gain for the Cu lamp	< 55%
Nebulizer uptake rate	Within range 4.0 – 6.5 mL/min.
Absorbance for 5 mg/L Cu standard	≥ 0.4 Absorbance
Precision for 10 replicates	< 1.0 %RSD

Trouble shooting:

Observed Problem	Recommended Check/s
Poor precision	– Check the mixing paddles are installed in the spray chamber and fitted correctly (pip in the centre of the paddles facing towards the nebulizer) – Check burner is aligned correctly – Check nebulizer is set up correctly by measuring the uptake rate – Check impact bead adjustment. Try adjusting the impact bead adjuster half a turn clockwise and repeat the test – Check all flame shields and chimney in position – Check nebulizer, burner, spray chamber and paddles are clean – Check purity of gas supplies, especially air supply for carryover of moisture or oil
Low absorbance	– Check burner is aligned correctly – Check nebulizer is set up correctly and check for possible blockage of the capillary by measuring the uptake rate – Check impact bead adjustment. Try adjusting the impact bead adjuster half a turn anti-clockwise and repeat the test – Check the standard solution used. Prepare a fresh solution and repeat the test – Check nebulizer, burner, spray chamber and paddles are clean – Check the hollow cathode lamp alignment and function
Low uptake rate	– Check sample tubing for blockages – Check impact bead adjustment. Try adjusting the impact bead adjuster half a turn anti-clockwise and repeat the test – Check nebulizer is set up correctly and check for possible blockage of the capillary by measuring the uptake rate – Check nebulizer, burner, spray chamber and paddles are clean

Graphite Furnace AAS Health Checks

Background:

This test checks the performance of the graphite furnace atomization system. Specifically, the test checks the analytical sensitivity and precision of the graphite furnace system. This is suitable for either the Zeeman graphite furnace AA systems, or the graphite furnace system installed as an accessory to the flame AA instrument.

The test is performed using a copper lamp at the 324.8 nm emission line. The sensitivity and precision is determined by injecting three replicates of a 25 µg/L Cu standard solution into the graphite furnace.

If routine maintenance is required before completing these tests, refer to the Maintenance schedule section of the online Help for guidance on daily maintenance tasks and cleaning procedures.

What you will need:

- An Agilent copper (Cu) hollow cathode lamp (coded or uncoded)
- A 25 µg/L copper (Cu) standard solution
- A 0.1% nitric acid blank solution

Test procedure:

1. Develop a worksheet for this test. Select either the Zeeman or the furnace method for Cu from the cookbook.
2. Click “Edit Methods...” Check that the Sampling Mode is set to Automix, measurement mode is set to Peak Height and 3 replicates have been selected.
3. Go to the Standards page and verify the following concentration values have been defined:
   - Standard 1: 12.5
   - Standard 2: 25.0
   - Standard 3: 37.5
4. Go to the Sampler page and verify the following locations for the solutions are set:
   - Bulk Standard Position: 51
   - Make Up Position: 52
   - Total volume (µL): 25
   - Sample Volume (µL): 10
   - Modifier Volumes (µL): 0
   - Bulk Concentration: 25.0
5. Click OK to Save the method changes.
6. Install the Cu hollow cathode lamp. Ensure that the lamp position in the Cu method matches the position where the lamp is located.
7. Click on the Analysis tab and Press the Optimize button to go to the Optimization display.
8. Select the Zeeman or the furnace method for Cu from the list and click OK. Verify that the Cu lamp is switched on.
9. Ensure that the optical path through the sample compartment is free of obstructions. Optimize the lamp alignment by slowly turning each of the adjustment knobs on the lamp holder in turn to maximize the signal bar, rescaling the display where necessary. Once the lamp alignment is complete, record the % Gain displayed and compare this with the expected value.
10. If not already in position, install the workhead of the graphite furnace AA into the sample compartment. Using the workhead positioning height and/or lateral adjustment knobs, adjust the position of the workhead to maximize the light passing through the furnace.
11. Press OK and then Cancel to close the Optimization display.
12. If not already in position, install the furnace autosampler of the graphite furnace AA onto the instrument.
13. Select Furnace Facilities from the Instrument menu and then click “Align”.

    The sampler will position the capillary at position 1 in the fully down position. Check and adjust (if necessary) the alignment of the probe to ensure the probe is correctly located in the center of the vial, and not touching the base.

    Click “Align” again. The probe will move to the furnace workhead and lower into the furnace. Check and adjust (if necessary) the sampler position to ensure that the probe, when lowered, enters the injection hole of the graphite tube without hitting the sides – and that the probe is set to the correct depth in the tube. Press Rinse to return the probe to the rinse station.
14. Press “Close” to close the Furnace Facilities display.
15. Place the blank (make-up) and the 25 µg/L Cu standard into suitable vials located in positions 52 and 51 (respectively) of the autosampler carousel.
16. From the Analysis window, click on Standard 2 in the Calibration window to select this – and press Start GTA. The solutions to prepare standard 2 will be collected in the probe and then injected into the graphite tube. The furnace program is then initiated.
17. Record the Mean Absorbance and %RSD of the readings obtained for the 25 µg/L copper standard solution.
18. Compare the readings obtained for the 25 µg/L copper standard solution with the expected results. If the achieved results do not meet the expected result, refer to the Troubleshooting section below for suggested corrective actions. Retest as appropriate. If the expected results still cannot be obtained after trouble shooting and retesting, a service call may be required.

Expected performance:

% Gain for the Cu lamp	< 55%
Absorbance for 25 µg/L Cu standard	≥ 0.15 Absorbance (≥ 0.10 Absorbance for Zeeman systems)
Precision for 3 replicates	≤ 4.0 %RSD

Trouble shooting:

Observed Problem	Recommended Check/s
Poor precision	– Recheck alignment of the dispensing capillary to confirm probe positioning is correct and droplet is being dispensed into the centre of the tube – Check the age of the graphite tube and replace tube if necessary – Check that the tip of the dispensing capillary is clean and free of deposits – Check that the rinse solution in the rinse bottle of the autosampler is acidified with 0.01% nitric acid. The addition of 0.002% Triton X100 is also often useful. – Perform a Rinse of the furnace autosampler and while manually lifting the probe, verify that there is a continual flow of rinse solution from the dispensing capillary – Check that the standard and make-up (blank) solutions have been prepared in an acid matrix
Low absorbance	– Recheck alignment of the dispensing capillary to confirm probe positioning is correct and droplet is being dispensed into the centre of the tube – Check the age of the graphite tube and replace tube if necessary – Check that the standard and make-up (blank) solutions are located in the nominated positions in the autosampler carousel – Check that the standard and make-up (blank) solutions have been prepared in an acid matrix – Check when the standard was prepared. If this is more than a few days old, prepare a fresh standard – Confirm that you are using argon as the inert gas supply to the graphite furnace AA – Check the condition of the electrodes in the furnace workhead. Clean the surface to remove any loose graphite or other material that may be present

Need further guidance?

If you would like additional advice combined with tips and tricks to help ensure you are able to achieve the best performance, and your maintenance SOPs are robust and reliable, refer to the Agilent AA Troubleshooting and Maintenance Guide available from the Agilent website.

Stay up to date with the best AAS practices for instrument maintenance and operation.

Explore the AAS resource hub, visit explore.agilent.com/aas-resource.

Recommended AAS consumables to keep your lab online and productive

Labs today are often operating with restricted access to meet strict social distancing requirements. If you are looking to keep your lab operational and striving to achieve optimal performance, Agilent is here to make sure you have everything you need, every step of the way.

With that in mind, Agilent technical experts have compiled a list of recommended products designed to:

- Help you keep your operations running as efficiently and productively as possible.
- List recommended supplies for different matrices to ensure you have the correct configuration for your analysis.
- Simplify installation and ensure your lab continues to produce reliable results.

It's all part of our commitment to helping your lab navigate its "new normal" in these unprecedented times.

Recommended Supplies for Flame AAS

Recommended Supplies for Graphite Furnace AAS

www.agilent.com/chem/aas-healthcheck

DE93034863

Published in the USA, December 9, 2021
5994-4382EN

Agilent
Trusted Answers