

## Determining concentration of aluminum in samples on the atomic absorption spectrophotometer

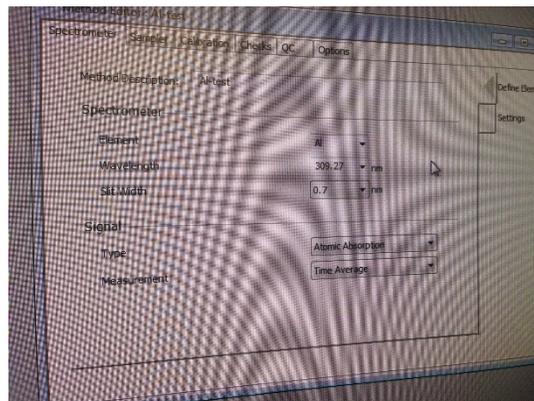
Important: sample prep protocol from EPA method — [“METHOD 7000B FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRY”](#)

4.9.1 Aluminum — Aluminum may be as much as 15% ionized in a nitrous oxide/acetylene flame. Use of an ionization suppressor (1,000 µg/mL of K as KCl) as described in Sec. 4.5 will eliminate this interference.

4.5 Ionization interferences occur when the flame temperature is sufficiently high to generate the removal of an electron from a neutral atom, giving a positively charged ion. This type of interference can generally be controlled by the addition, to both standard and sample solutions, of a large excess (1,000 mg/L) of an easily ionized element such as K, Na, Li or Cs. Each sample and standard should contain 2 mL KCl/100 mL (0.2mL /10 mL) of solution. Use 95 g of potassium chloride in 1 L of reagent water for the KCl solution. *Note: we dissolved 30 g KCl in 1L of solution and added 1 mL of this solution per 10mL of sample to get 11 mL total volume in culture tubes*

### Procedure

1. Make standards in your sample matrix. 0, 1, 5, 25, 50, and 75 ppm. Make 100 mL each of the 1, 5, and 25 ppm standards and 200-300mL each of the 0 and 50ppm standards.
2. Make ~ 500 mL of 5% w/v nitric acid solution to clean aspirator tube between samples.
3. Turn on AA. Make sure that aspirator tube is out of any solution. (Tube should be aspirating air).
4. Open up Syngistix software.
5. Open method file (Al-test). (Element = Al,  $\lambda = 309.27$  nm, slit width = 0.7 nm). Click Analysis > Method. In the method editor that is now open, click Calibrations > Standard Concentrations. Enter both the ID and the actual concentration of each of your standards.
6. Change out burnerhead if necessary. For Aluminum, use the smaller burnerhead (0.2nm) with the magnetic strip facing towards the back. You must push down white button on nebulizer assembly and pull the grey lever completely to the forward position to put the burnerhead in place.
7. Turn on gasses by turning the knob on the top of the tank counterclockwise to completely open—air compressor, acetylene tank, and nitrous oxide tank. Note: plug in regulator on nitrous oxide tank.
8. Turn on flame controls (Instrument > Flame Control) in Syngistix and turn on flame. Allow burnerhead to warm up on air for a few minutes.
9. Switch flame to nitrous oxide/acetylene in the flame controls (must click “Apply”). Allow burnerhead to warm up for another 15 minutes.
10. **SAFETY NOTE: Always wear the welder’s glasses when looking directly at the flame.** Look at the flame. There should be a red “feather” in the middle of the flame. If



Method editor

- the feather is shorter than 1.5 inches vertically, increase the acetylene flow rate to about 8L/minute (must click "Apply" after changing flow rate).
11. Open continuous graphics (Instrument > continuous graphics).
  12. Use burner alignment wizard to automatically adjust vertical and horizontal direction of burner. Follow directions on wizard. You may need to use the burnerhead key that fits in the front hole on the burnerhead to slowly rotate the burnerhead until your 50 ppm standard reads at ~ 0.2 nm in continuous graphics.
  13. Go to Analysis > Sample Info. You can copy/paste your sample IDs into the software here.
  14. Click Results > Results to open results sheet. Then go to Analysis > Analysis. Stick aspirator tube in deionized water and click "Analyze blank" in the Analysis tab. Allow machine to run all three reps. Wait for auto-zero performed readout in the results sheet.
  15. Now you are ready to run your standard curve. Stick aspirator tube in first standard and click "Analyze Standard." Clean tube by aspirating a small amount of the 5% nitric acid for 3-5 seconds between samples. Run the rest of standards and click Results > Calib. Calibration coefficient should be 0.995 or better.
  16. In the analysis tab run a matrix blank after your standard curve to double check calibration. You may insert it as a sample. Click "Analyze Sample."
  17. Now you are ready to analyze your samples.
    - a. Analysis > Sample info > Paste sample table
    - b. Analysis > Check sample info name and save location > change file save path if incorrect
    - c. Repeat step 16 and clean aspirator tube by aspirating a small amount of the 5% nitric acid for 3-5 seconds between samples.
    - d. Check residuals in results tab. If they are higher than 20%, rerun the sample. If any sample is higher than your calibration curve, make a note and dilute that sample to rerun later.
  18. Periodically run a blank to check calibration (~ every 15 samples).
  19. When you are done running samples, switch flame back to air/acetylene in Flame Control (hit "Apply". Allow to run for about 20 seconds and then switch flame off completely.
  20. Close gas tanks (turn tank knob all the way clockwise), turn off air compressor, and unplug nitrous oxide regulator.
  21. In flame controls hit "Bleed gases." **Note: this is an important safety step as it will bleed all gas remaining in the machine tubes.**
  22. Turn off Syngistix.
  23. Turn off AA.
  24. Open Data Manager software on the desktop.
  25. Click "Export."
  26. In data export wizard click "Use existing Design" and open up the file titled "AA\_report.xptx"
  27. Go through the rest of the data export wizard and select desired report output parameters.
  28. Save file.

### **Common issues/troubleshooting**

- Burnerhead not locked – jimmy the burner head around, make sure that grey lever is all the way forward.
- Dirty burnerhead – soak burnerhead upside down (DO NOT submerge magnetic strip – only top of the burnerhead) in 10% nitric acid for 30 mins. Rinse thoroughly with deionized water and let air dry.
- Uneven flame – there may be buildup on the burnerhead. Use graphite stick to clean burnerhead with flame on.
- Flame won't light – use Bunsen lighter to manually light flame.
- Drain Interlock – unscrew the draining lock and pour DIW down the drain tube
- Cannot run samples at the same time as “continuous graphics” window is open
- Clog in nebulizer – nebulizer needs “oiling”, aspirate coffee
- No air pressure error – using a flathead screwdriver, loosen the hose clamp on the waste drain line. Pull the off of the machine and pour a stream of deionized water down the line. Reconnect the line to the machine. Error message should disappear.

### **References**

SW-846 Test Method 7000B: Flame Atomic Absorption Spectrophotometry.

<https://www.epa.gov/hw-sw846/sw-846-test-method-7000b-flame-atomic-absorption-spectrophotometry>