

Flame Atomic Emission Spectrometry of Sodium

The objective of this laboratory experiment is to introduce the concept of flame emission as applied to analytical atomic spectroscopy, and to explore the working concentration ranges in an air acetylene flame which is commonly used in atomic emission spectroscopy.

FLAME START-UP PROCEDURE

Before you begin the experiment, it is extremely important to point out the precautions and rules to follow when using the Varian AA-1475 atomic absorption/emission spectrophotometer. FAILURE TO OBSERVE ANY OF THE FOLLOWING PRECAUTIONS CAN AND WILL RESULT IN A VIOLENT EXPLOSION.

1. Pull off the burner head to check if the liquid trap is filled. Check by pouring distilled water into the spray chamber through the burner head collar until you observe water running through the drain tube into the waste bucket. Replace the burner head, making sure that it is secure.
2. Make certain that the waste bucket has been emptied before you start. After emptying the bucket, replace the drain tube. When replacing the tube, make sure that the end of the tube is NOT submerged.
3. Turn on the exhaust hood before any fuel or oxidant lines have been opened. Also, turn on the power switch on the Varian spectrophotometer.
4. Open the oxidant line by turning the switch on the left-hand front panel. With the oxidant switch open, make sure that the oxidant flow gauge ball is even with the red line. Check the air pressure gauge to be sure that the pressure is approximately 60 psi.
5. Open the valve on the acetylene tank. Adjust the fuel flow so that it reads between 3 and 4 on the gauge scale. Once this is adjusted, return to the acetylene tank and check the tank pressure. It should read between 8 and 10 psi with the fuel valve open on the instrument.
6. With both the air and acetylene lines open, press the ignite button on the instrument's front panel. A bright yellow-white flame should result. Adjust the fuel control so that less acetylene is allowed into the combustion mixture. You should continue the adjustment until you see a crisp blue feather and non-luminous flame (The feather should be sharp and well defined).
7. Aspirate distilled, deionized water for approximately 5 minutes to allow the flame and instrument to stabilize. Aspiration of water also serves to clean out the spray chamber and nebulizer, which may have residue left from previous samples.

SHUT-DOWN PROCEDURE

To shut the instrument down the following procedure should be used.

1. At the end of use, aspirate distilled, deionized water for approximately five minutes.
2. To shut the flame off, turn the fuel flow gauge control off (completely to the right). Let the air run for about 1 minute to flush any remaining acetylene from the system. Shut the air off using the oxidant switch on the front panel.
3. When shutting down at the end of the day, be certain that the excess acetylene has been drained from the fuel lines. To do so, shut off the fuel valve at the tank, open the flow valve on the spectrophotometer, and let all gas bleed out until the tank gauge reads zero pressure.
4. Turn off the exhaust hood when finished.

EXPERIMENTAL PROCEDURE

You will be provided with a 1000 ppm Na stock solution. You will need to dilute this to obtain the series of standard solution needed. Your instructor will provide you with a liquid unknown that needs to be diluted to 100 mL. Report the concentration of this diluted sample in ppm Na.

Na Determination and Self-Absorbance

Refer to procedure 4.13 on page 50 in the Varian AA-1275/1475 Operation Manual for specific directions regarding flame emission operation with this instrument.

To shut off AA lamp: Press Lamp 1, type 0 on the keyboard, then press Lamp 2, and again type 0. Return the instrument to flame emission mode by pressing the "Emission" key.

Using your 10 ppm Na standard, adjust the instrument wavelength setting to 589 nm (slit setting 1). Once adjusted, maximize the signal intensity using the analog PMT current meter. If this meter deflects off the maximum scale, readjust this signal amplification using the SB AUTOGAIN button. Pressing this button will return the meter to an "on-scale" position.

Once the signal is maximized, use the highest standard in the series of standards (100 ppm) to set the maximum scale amplification. Aspirate the highest concentration Na standard. Press "SB AUTOGAIN" to reset the instrument amplification. Once on scale, aspirate distilled-deionized water. Allow the digital readout to stabilize to a constant level and press "CALIBRATE". The digital output should read 0.

Record and later plot emission readings for the following series of standard Na solutions: 0, 1.0, 3.0, 5.0, 10.0, 20.0, 50.0, 100.0 ppm Na. When performing the measurements, use a 3 second integration time. Also record the horizontal and vertical burner positions that you use. Measure the standards in increasing order of concentration and discuss your results in terms of the phenomena of self-absorbance. Next, measure the Na concentration in tap water and in

the sample provided by the instructor. Now rotate the burner head 90 degrees so that the flame is perpendicular to the optical path. This should be done with the flame off and care should be taken since the burner will be hot. Repeat the measurement of the Na standards and the tap water samples with the new burner position. When finished, return the burner head to a position parallel to the optical path.

LAB WRITE-UP QUESTIONS:

1. What is the working concentration range useful for sodium analysis?
2. The relation between emission intensity and the concentration is theoretically linear. Why does the calibration curve of Na bend? (Give and reference for your answer.)
3. What effect does the perpendicular positioning of the burner introduce on the detection limit?