

Varian SpectrAA 400 Atomic Absorption Spectrometer Instrument Protocol

(RevD; 07Apr04)

Gas Pressures (stage 2): Air @ 60psi Acetylene @ 11psi N₂O @ 60 psi

Note: To avoid acetone contamination DO NOT use acetylene at cylinder pressures below 100psi

* = Instructions applicable only if SIPS and/or SPS5 Autosampler used

1. Open database if not already open. Initiate run log by choosing "Perform An Analysis" from main menu

2. Turn on: spectrometer, autosampler, SIPS, acetylene, air valve, and N₂O if used.

Note: DO NOT turn off main air valve on wall.

*3. Fill autosampler rinse reservoir with water and diluent carboy with solution to match sample/standard matrix.

*4. Attach proper SIPS tubing. Adjust clamp pressure settings in "Workgrp.ini" file on desktop; 120 for Neoprene, 150 for Tygon.

*5. Load samples, std, QC into autosampler rack(s); place rack(s) in appropriate position on autosampler table.

*6. Lower autosampler probe down into rinse.

7. Complete required (blue) fields in log to enable SpectrAA icon then click to open program.

8. Click on 'Worksheet'

-To use an old worksheet as a template, click on 'New From' and choose the appropriate file

-'New' allows you to create a new worksheet with new analysis parameters (see Tom for New Worksheet development assistance)

-'Open' opens an old worksheet for you to look at.

9. Click on 'Labels' and enter sample labels, there is an autocopy option.

10. Confirm correct burner head is in place.

11. Optimize the Lamps

a) Highlight column with element to be optimized

b) Click on optimize, once lamp is lit turn out flame (flame need not be lit to turn on lamps for manual run) ; OK to prompt

c) Optimize element lamp – use two knobs at base of the lamp to maximize the signal, rescale if necessary, check versus the last optimization (see historical data)

d) Record signal and gain on log form

e) Repeat steps a - e for all lamps to be used in analysis

NOTE: It is not usually necessary to re-light flame to optimize other lamps

f) Clean burner with alignment card

- g) Use alignment card to align burner with hollow cathode (HC) lamp, check middle, front, and back of burner for alignment; rotate if necessary
- h) If deuterium background correction is being used, turn the lamp turret to take HC lamp out of alignment and align the D₂ lamp to the burner alignment card by turning the two knobs, if necessary

12. Light flame – push button down until flame lights then let go.

*13. Pull SIPS tubes into slots.

*14. To Prime SIPS and Autosampler:

- Choose 'Instrument' → 'Flame Facilities'
- 'Load pumps'
- 'Rinse pumps'; allow ≥ 3 minutes to rinse
- 'Stop Pumps'

15. Optimize Signal (**NOTE:** It is not always necessary or desirable to optimize for max sensitivity. Sometimes analysis quality can be improved by operating at less than max. sensitivity)

- a) Light flame
- b) Click 'Optimize'; OK to prompts
- c) Click 'Optimize Signal'
- d1) If using in Manual mode, introduce high standard of appropriate element directly into capillary tube to nebulizer
- *d2) If using SIPS and/or Autosampler, a dialog box will appear:
 - 1. Enter rack and tube position of high std of appropriate element
 - 2. Click 'Go to Rack / Tube'
 - 3. If SIPS is used, click 'Start Pump'; dialog box will close
- e) Adjust nebulizer impact bead position, burner position, & gas flow setting to maximize signal. Remove mixing paddle if necessary.
- f) *If Autosampler and/or SIPS is used, go to 'Instrument', 'Flame Facilities', 'Rinse', 'Stop Pumps'

16. Click on 'Select' and highlight the samples to be run.

17. Click 'Start' – start with calibration

18. To Print

- Right click on the data – click 'Reports' → 'Report' → 'Print'

19. After Run:

- *-Unload pumps (Instrument → Flame Facilities)
- *-Pull up Autosampler probe
- *-Take tension off pump tubes
- *-Cover modifier and return to storage
- Close acetylene cylinder valve. Light flame to bleed remaining gas in lines.
- Turn off power; close N₂O, and air valves.
- Complete Run Log Form and Billing Entry Form. All elements/analyses for one worksheet may be combined into one BILLING FORM. A SEPARATE RUN LOG must be completed for each element on each worksheet.**

20. Clean up all glassware and dispose of waste properly.

Standard Preparation for High Concentration Alkaline Earth (Ca, Mg, Na, K) Analysis

Bulk Standard

Recipe for 50 ml (can be scaled up if needed):

Element	vol. of stock std (ml)	stock conc. (ppm)	bulk std conc. (ppm)
Ca	1.0	1000	20
Mg	1.0	1000	20
Na	0.5	10,000	100
K	0.25	1000	5
HNO ₃	0.357	70 %	0.5%

Quality Control – VHG Labs Water Pollution Standard (property of LTER)

Make the QC in a 50 ml volumetric flask

Add 1.667 ml of VHG Labs QC

Add 0.357 ml HNO₃

Element	stock conc. (ppm)	final conc. (ppm)
Ca	500	16.67
Mg	100	3.33
Na	500	16.67
K	100	3.33

** To check SIPS dilution accuracy, also run the QC solution undiluted as a sample at end of run.

Modifier:

Use Lanthanum at 1,000 ppm FINAL concentration.

50,000 ppm Lanthanum Stock

- Add 250 mL HCl SLOWLY to 58.65g Lanthanum Oxide in a 1L flask
- Swirl gently until all the Lanthanum Oxide dissolves.
- Dilute to 1000mL with Nanopure water.
- Use 1 part La Stock to 50 parts Sample/Standard/Blank, etc.

** If using SIPS for dilutions, remember to use 1,000 ppm La + acid as diluant!