Standard Operating Procedure

Thermo iCE 3500 Flame Atomic Absorption Spectrophotometer (Acetylene/Air)

Introduction

Flame atomic absorption spectroscopy is a sensitive elemental analysis technique used to determine the metal concentration within water samples at the milligram per litre level.

Purpose

The purpose of this document is to introduce the user to the operation of Thermo iCE3500 Flame Atomic Absorption Spectrophotometer (Thermo iCE3500) when using the acetylene/air fuel source in the TRACES Centre. This document also outlines the special safety concerns introduced when using the instrument.

Scope

This procedure applies to direct determination of a metal* in water samples using the Thermo iCE3500. Detection limits for each element at specific wavelengths are reported in the Thermo iCE 3500 Operations Manual (below)*. Individuals must complete a hands-on one-on-one training session with a TRACES staff member.

Referenced Documents

- iCE 3000 Series AA Spectrometers Operators Manuals ver.1 (p/n 9499 500 23000)
- Thermo FAAS Methods Manual version5 (p/n 9499 230 24011)
- Thermo iCE SOLAAR Software Manual ver 2 (p/n 9499 400 30011)
- Flame Atomic Absorption Spectrometry Analytical Methods ed. 8 (p/n 8510000900)
- Perkin Elmer Analytical Methods for Atomic Absorption Spectroscopy rev.D (p/n 0303-0152)

Responsibilities

1. **TRACES Users**
   - 1.1. All Users must obtain training with TRACES Staff prior to system operation. It is the responsibility of the User to ensure they have a good understanding of the instrument and all operation protocols.
   - 1.2. If additional training sessions are needed it is the responsibility of the User to schedule these with TRACES Staff.
   - 1.3. Instrument time must be booked by the User via the online booking system prior to system operation.

2. **TRACES Staff**
   - 2.1. It is the responsibility of TRACES Staff to provide introductory and, if required, subsequent training to all users.

Equipment

- Thermo iCE 3500 FAAS
  .1. AA Grade Acetylene
  .2. Zero Air
- Type 1 (18.2MOhms) Deionzed Water

Safety

IMPORTANT:
You will be using the Thermo Scientific iCE 3500 spectrometer equipped with an air-acetylene flame ~2300 °C.

- Approach the instrument with caution – the flame is very hot!
- Do not open the flame door, this should remain closed for the duration of the experiment
- If at any point you have questions or concerns consult with the TRACES Staff
- **Flame must never remain unattended**
- Seek assistance from TRACES Staff if you have any concerns

Initial Setup

1. Turn on the acetylene. Check to see that MORE than **500 kPa** is remaining in the cylinder. If you expect a long run time contact the TRACES Lab Manger
   a. Do not touch the regulators; they have been set for you
      i. Ensure the gas pressures entering the FAAS are set to the values below
         1. Acetylene: 10 psi
         2. Air :30 psi
2. Remove the burner unit and fill with water
   a. Fill till water is visibly trickling down the waste tubing
   b. Check the waste container to ensure it is at least ¾ empty
3. Startup the Solaar software located on the desktop
   a. Username: Solaar
4. Ignite the flame (button should be flashing)
   a. Allow the flame to warm up for 5 min
5. Click on the lamp icon 
   a. ID the lamp of choice and click the 'Off' button to 'On'
   b. Non-Thermo lamps MUST be manually configured. Please ensure that the correct amperage is applied or damage to the lamp and instrument is inevitable. *
   c. Allow 15-20 minutes of warm up time

Method Setup

6. Load/New Method window is chosen (Found under **Edit**) 
   a. New method: set the parameters

i. Technique: Flame
ii. Element of interest and mode of operation under the 'General' tab*
iii. 'Sequence' tab
    1. Setup the appropriate sequence that matches your method
    2. Ensure the standards are measured first
iv. 'Spectrometer' tab
    1. D2 Quadline :On/Off
    2. Cook Book illustrates default values and reference guide to interferences and quantitative limitation for specific AB lines
    3. Wavelength of interest selected
v. 'Flame' tab :
    1. Unless otherwise noted use the default values set by Cook Book
        a. Gas Flows, Nebulizer Flow, Burner height
vi. 'Calibration' tab:
    1. Calibration Model: Choose from the following selection:
        a. Linear (select in most cases)
        b. Segmented
        c. Quadratic
    2. Standard Stock Concentrations
    3. Units of measure

b. Load method: review the parameters
    i. Adjust accordingly
    ii. ENSURE that the SEQUENCE and CALIBRATION tabs are correctly set
c. Save Method using a new name with the element as an identifier (i.e. Tony Zn 2020.m)

Instrument Setup

7. Click on 'Flame Setup'
    a. Unless otherwise noted use the default values set by Cook Book 
8. Click on 'Optics Setup' 
    a. Wait till all Optics Parameters
    b. Wavelength should be appropriately set
    c. Absorbance should read zero
9. Auto zero the spectrometer 
    a. If the absorbance does not read zero
    b. This can be done during long wait periods between analysis
10. Manually setup the optics (if necessary)
    a. The primary line is chosen from the Cook Book, alter if need be.
    b. Use a typical test solution (i.e middle standard) to test flame and optics selections
    c. Check to maximize the absorbance value
        i. Align the Burner Laterally
        ii. Align the Burner Rotationally (Do not wear gloves or loose clothing, roll up your sleeves)
        iii. Align the Impact Bead position
            1. You may need to play with this to maximize the signal

Analyze

11. Select 'Analyze' 
    a. Save the data with a date specified name (i.e. jun21_2012_Cu)
12. Follow the prompts as they appear
    a. The prompts should follow the sequence setup in the method
    b. Introduce each solution into the spectrometer using the tubing at the front of the spectrometer
    c. Once introduced, wait 1-2 seconds, and click 'Run Sample'
    d. Clean the tubing before and after each sample introduction using a Kimwipe

Evaluate

13. To evaluate the results, click on the Results tab
    a. Under the Options tab:
        i. Select data set by element
        ii. Select data set by type
        iii. Select data set by date
    b. Under the Change Line Fit tab:
        i. Select a new calibration curve
    c. To remove data
        i. Click on the individual data line and Delete Results
14. To print the data sets:
    a. Click Print
        i. Select Signal Graph: To print the absorbance vs time graph
        ii. Calibration Graph: To print the calibration curve
        iii. Results: To print all data collected and instrument parameters
    b. Click Print Options
        i. Select data parameters to be printed with the Results Report
    c. Export Data
        i. As a .csv or .txt file

Instrument Shutdown

15. Shutdown Procedure
    a. <u>Shutoff the acetylene at the source in the flammable cabinet</u>
        i. **Vent the acetylene from the instrument by clicking on the red button**
            1. **Before you are complete BOTH regulators MUST read 0psi**
            2. **Contact the TRACE Manager immediately if you are unsure or if you are unable to vent the gas**
                a. The Air cannot be vented
                b. The Argon does not need to be vented
        ii. Click all the lamps to the 'Off' position
16. Logoff

*The TRACES Manager will provide further details during hands-on training.