Standard Operating Procedure

Thermo Flash 2000
Elemental Analyzer (NC Operational Mode)

Introduction
The Thermo Flash 2000 Elemental Analyzer (EA) bases its operation on the well-known process of Flash Dynamic Combustion, which produces complete combustion of the sample followed by an accurate and precise determination of the elemental gases (nitrogen and carbon dioxide). This in turn will provide detection of total carbon (TC) and nitrogen (TN) from 100ppm to 100% level.

Purpose
The purpose of this document is to introduce the user to the routine operation of Thermo Flash 2000 Elemental Analyzer (EA) with a dedicated Thermal Conductivity Detector. This document also outlines the special safety concerns introduced when using the instrument.

Scope
This procedure applies to established procedures of analysis using the EA in NC mode to determine the TC and TN in soils, leaves and sediments. Individuals must complete a hands-on one-on-one training session with a TRACES staff member.

Referenced Documents
- Thermo Flash 2000 Elemental Analyzer: Instruction Manual ed.4th (p/n 31709912)
- Thermo Flash 2000 Elemental Analyzer: Consumables ed.11th (p/n 31708250)
  AN 633 FlashEA™ 1112 NC Soils Analyzer: Reproducibility of Nitrogen/Carbon determination

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. User must ensure that EA is fully operational before commencing use of the instrument.
   1.4. 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.
Standard Operating Procedure

Equipment (Standard Configuration)
- Thermo Flash 2000* (Go to Initial Setup for more details)

Safety

**IMPORTANT:**
- Oxygen is an oxidizing gas keep away from flammables
  - Take the appropriate precautions
- Seek assistance from TRACES Staff if you have any concerns

Initial Setup
1. Ensure there is ample oxygen and helium to run your experiments. Minimum 1000 psi if an overnight run is to be initiated. Please check with the TRACES Lab Manager if this is your intention.

2. The EA should already be powered up
   a. EA should be in “Stand By’ mode
   b. Please inform TRACES Staff if the Safety Cut Off is lit

3. Start the computer
   a. Username/Password is given verbally by TRACES Staff
   b. If not Click on Instrument 1 Online

4. Start the software
   a. Select the **Eager Experience** icon located on the desktop
   b. Select the **NC Soils** icon in the Eager Window

5. Confirm that all the Instrument Actuals (Figure 1)* are not greyed out.

![Figure 1](image)

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EA Setup Section

6. Method
   a. The method of choice is NC.eam (please see TRACES Staff for more details)
      i. This method is set for the determining of TC and TN in soils, leaves and sediment
b. Establish that the NC.eam method parameters are correctly set (see below) by clicking on the Parameter button set in the Main Menu

c. NC.eam Parameters (set values)
   i. Temperature:
      1. Left Furnace: 950 °C; Right Furnace: 840 °C; Oven: 50°C
   ii. Flow/Timing (ml/min)
      1. Carrier Flow: 140; Reference Flow: 100; Oxygen Flow: 250
   iii. Detector
      1. TCD: On    Gain:1    Prep:0

d. Ready the EA System
   i. From the ‘Stand By’ mode
      1. Manual Ready
         a. Select Status window
            i. Uncheck the ‘Standby’ box
            ii. Click ‘Send’ to send the wake-up call to the EA
            iii. Click Okay
      2. Automated Ready (advanced users)
         a. Activate ready using the Auto Ready from the Status window
         b. Ensure the correct date is set and that the gas meet minimal levels

7. Instrument Warmup time
   a. 60 minutes warmup time from this point

8. Leak Test
   a. Can be accomplished while instrument is warming up
   b. Go to the Status window
      i. Select Special Functions
      ii. Click on Leak Test
      iii. Start
   c. The Leak Test will ask you if you want to perform an Auto-Zero function
      i. Select YES
   d. After 30 seconds
      i. Reference flow should drop to 0-2 mL/min.
   e. After 240 seconds
      i. Carrier flow should drop below 5 mL/min.
   f. Select Done when you are satisfied with the results.
   g. Wait 5 minutes BEFORE starting any analysis

9. Checking System before Running
   a. Select Parameter
      i. Under the ‘General’ tab ensure all the Actuals equal the Set parameters listed above (6c)
EA Loading Samples

10. Using the Ultra-Balance
   a. **Extra care must be given to both the handling and recording of the sample and standard weights, as this is the area of GREATEST ERROR.**
   b. A Mettler-Toledo XP6 Ultra-balance is located at the SW corner of TRACES.
      i. This device has a MAXIMUM capacity of 6 grams and a readability of 1 ug
      ii. When re-weighing samples; the difference must not be greater than 0.01mg*
      iii.

11. Tin Capsules
   a. Available from TRACES
      i. Tin Capsules Pressed Standard Weight 10x10mm
      ii. Samples (solids and liquids) and Standards are enveloped in each capsule
         1. Using a technique that maximizes closure and reduces spillage*

12. Preparing Samples and Standards
   a. Standards
      a. Aspartic Acid is available from TRACES
         i. Load a **minimum** of five standards with varying amounts
            1. 1-8mg
   b. Blanks
      a. Tin capsules only
   c. Bypass
      a. Tin capsules with aspartic acid
         i. Not weighed-simply for ‘waking-up’ the catalyst as a first run
   d. Samples
      a. Varying amounts of sample depending on the nature of the samples
         i. Organic samples; 1-3 mg
         ii. Soils; 50-70 mg
         iii. Sediments; 5-15 mg
         iv. Filters; 5-25 mg

13. EA Sample Sequence
   a. Click on the **Sample Table** and start a new
      i. Using the wizard
      ii. Manually insert the required information for each A/S position
   b. All sequences should start using the following order
      i. One (1) Bypass
      ii. Two (2) Blanks
      iii. Five (5) Calibration standard (aspartic acid)
      iv. Ten to Fifteen (10-15) Samples*
v. Once the sequence is completed ensure that A/S is correctly in sync with the sample table. Set the ‘Actual’ key in the Sample Table to the FIRST SAMPLE to be run in the sequence.

vi. Click OK.

c. Save the method
   i. Using a unique name (*.eam)
   ii. File under a Directory with corresponding year and month

d. Auto zero/auto adjust the TCD detector.
   i. Should read approximately 1000 uV (+/- 50 uV)

14. RESET the Calibration Factors (Linear Method), this will start a new calibration with your results*

15. **View Maintenance**

   a. Check all maintenance parameter are met BEFORE you continue*

16. Check the **STATUS page** to ensure all the parameters have been populated correctly

17. Auto zero/auto adjust the TCD detector.

**Starting Sequence Run**

18. Select Start sequence

   1. Click ‘Start Now’
      a. Check Force to ‘Stand By’ if you have complete loading samples
      b. Otherwise, run the sequence and keep the system in Ready mode

**Data Evaluation Section**

19. Calibration Curve

   a. Under ‘View’ tab select **Calibration Curve**
      i. Select Component ‘Nitrogen’ for linear calibration $r^2>0.999$
      ii. Select Component ‘Carbon’ for linear calibration $r^2>0.9999$
      iii. These values must be met BEFORE continuing the analysis

   1. If this is not the case speak with a TRACES Staff member*

20. Check Runs

   a. Once the sequence is running ensure that A/S is correctly in sync with the sample table, if not stop immediately and reset the sequence and the ‘Actual’ key in the Sample Table and Save the Method again.

   b. While analyzing the Bypass click under the ‘View’ tab select ‘View Chromatogram’
      i. Check the retention time of the Nitrogen and Carbon peaks.
      ii. The peaks should be separated and within the RT limits set

   1. If so, continue the analysis.
iii. Also view the Blank analysis for N or C peaks in the analysis
   1. Peaks should be <100k uV*sec

c. Viewing Real-time Chromatograms
   i. Shift F1 –view sample being acquired
   ii. Shift F9 –view last sample calculated results

21. Data Analysis*
   a. Data files are located in the folder you have selected to save your sequence method
   b. Offline analyzer can be started to view details of previous runs

   c. Results can be compiled with the' **Summarize Results**' tab
      i. The results can also be exported as either a *.xls or *.txt file
         1. Assign the same number to samples you wish to summarize
         2. Show Summary Sheet of Selected Group
            a. Export to Excel (*.xls)

d. Print
   i. Data, Samples and Chromatographs can be printed using the icon

e. Recalculation*
   i. Function allows the recalculation
      1. Weights
      2. Calibration Factors

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