

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: Operating Manual rev. D (p/n 31712052)
 - Thermo Flash 2000 Elemental Analyzer: Software Manual rev. C (p/n 31711055)
 - Thermo Flash 2000 Elemental Analyzer: Instruction Manual ed.4th (p/n 31709912)
 - IODP CHNS Analyzer User Guide: FlashEA 1112 Elemental Analyzer : User Guide ver1.1
 - 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.

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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 lite



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.

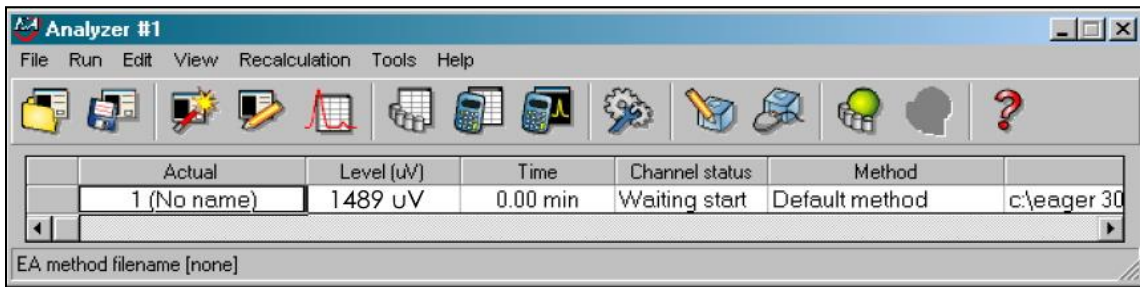



Figure 1

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

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- 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)

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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
 - i. When re-weighing samples; the difference must not be greater than 0.01mg*
 - ii.

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*




Figure 2

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  sequence.
 - 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*

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- 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.

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- iii. Also view the Blank analysis for N or C peaks in the analysis
 - 1. Peaks should be $<100k \text{ uV} \cdot \text{sec}$
- 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.