

CHNS Advance User Guide

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CHNS Elemental Analysis Advanced User Guide (from CHNS_AUG_371T_draft)

Manual Information

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Introduction

CHNS is fundamental element analyses performed in the shipboard laboratories for IODP cruises. Fluctuations in the concentrations and/or content ratios of C, N, and S define the origin, depositional environment, and diagenetic alteration of source materials. Analysts have a few choices for sample preparation, instrument settings, and measurement methodologies. Specific analytical methodology may be required based on the nature of certain sample materials. When this circumstance arises, laboratory specialists must authorize and document methodology deviations.

Apparatus, Reagents, & Materials

Before the cruise (during transit), confirm the existence and the condition of consumables, preparation tools, and chemicals for CHNS analysis on solid matrix.

Gases

- Helium (He) carrier gas: chromatographic grade
- Oxygen (O₂) for sample oxidation: minimum 999.995%

Laboratory Apparatus

Sample Preparation Tools

- Agate mortar and pestle: well washed, dried
- Chamber for freeze drier: well washed, dried
- Desiccator and silica gel: use well-dried silica gel
- Kimwipes
- Weighing paper
- Mill (for hard rock sample)
- Sample holder (2): stainless steel, well washed, dried
- Spatula (laboratory spoon, 2): stainless steel, well washed, dried
- Spatula (micro spatula, 2)
- Spring tweezers (2)
- Tin containers (set of 100): Thermo Electron PN 240 064 00
- Tin discs (30 mm) (set of 100): Thermo Electron PN 252 080 15, optional

Chemicals

Material Name	Description	Purpose
Aspartic acid (L-Aspartic acid)	C ₄ H ₇ NO ₄ : white fine crystals	Standard reference material
2.5-Bis(5-tert-butyl-benzoxazol-2-yl)thiophene (BBOT)	C ₂₆ H ₂₆ N ₂ O ₂ S: pale green crystals	Standard reference material
Copper oxide	Pre-packed	Filling material
Electrolytic copper	Pre-packed	Filling material
Ethyl alcohol (ethanol)	C ₂ H ₅ OH	Solvent for sample preparation
Quartz wool	SiO ₂	Filling material
Soil Reference Material	Light brown powder	Reference for nitrogen and carbon
Sulfanilamide	C ₆ H ₈ N ₂ SO ₂ : white odorless crystals	Calibration standard for CHNS/CNS

L-Cystine	$C_6H_{12}N_2O_4S_2$: white odorless crystals	Calibration standard
Vanadium pentoxide	V_2O_5 : yellow to red crystalline powder	Catalyst
Magnesium perchlorate	$Mg(ClO_4)_2$: white granulate	Filling material for water trap

Other Consumables and Tools

- Pre-packed reactor: Thermo Electron PN 468 020 15; also CE Elantech 061110
- Cleaning devices: Thermo Electron PN 205 006 25 and 276 060 10
- HPAR reactor extractor
- Crucible extractor

Advanced Apparatus

An expedition-specific science strategy or sample materials may require additional apparatus to handle liquid sample analysis.

- Automatic sampling system with injection assembly
- Water container and vials for solvent
- Syringe (0.5 μ L): PN 36504045
- Syringe (10 μ L): PN 36500525
- Syringe (250 μ L): PN 013680 (36504042)

Instrument Description

C, H, N, and S are analyzed on a Thermo Electron Corporation FlashEA 1112 CHNS elemental analyzer (Figure 1, below) with the following specifications:

- Supplier: Thermo Electron Corporation
- Model: FlashEA 1112
- Detector: Thermal conductivity detector (TCD)
- Instrument Control: *Eager 300* software for MS Windows
- Power Supply: 230 V AC; 60 Hz (US); 140 VA
- Dimensions: 500 mm H \times 590 mm W \times 558 mm D
- Weight: 65 kg
- Measurement Range: 100 ppm to 100% for C, N, H, and S
- Nominal Measurement Time: CNS: 8 min, CHNS: 10 min
- Sample Weight: 0.01–100 mg according to nature of the sample
- Temperature range: 15°–35°C Maximum rel. humidity: 30%–85%

The basic CHNS instrument configuration contains the following components.

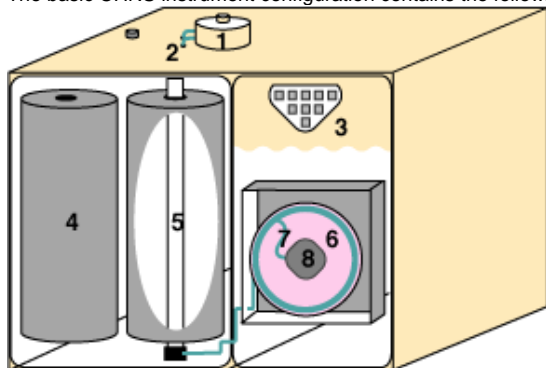


Figure 1. CHNS Components.

1. Autosampler (highlighted in red in Figure 2 and shown in detail in Figure 3)
2. Gas tubing for helium and oxygen
3. Synoptic panel
4. Furnace (furnace cover plate shown in Figure 4; opened plate in Figure 5)
5. Reactor
6. Oven
7. Water adsorption column (shown in Figure 6)
8. Gas chromatographic column (also in Figure 6)
8. TCD detector



Figure 2. Top and Front Instrument Panels. (Dry Sample Autosampler is located on top of furnace.)



Figure 3. Dry Sample Autosampler.



Figure 4. Furnace Compartment Access with Protection Plate.
WARNING! Do not open the furnace compartment during operation.

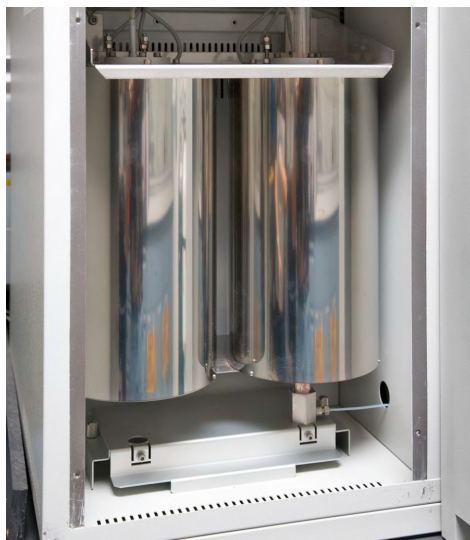


Figure 5. Interior Furnace Compartment.

WARNING: Do not touch either furnace when the furnace temperature is $>40^{\circ}\text{C}$. (Select **View > View Elemental Analyzer Status**).



Figure 6. Detector Access: Protection Plate, GC Column (inside), Adsorption Filter.

WARNING: The GC column may only be serviced by the shipboard technicians.

Sample Flow

Sample flow in the onboard laboratory normally proceeds as described in the following steps.

1. Collect samples from core (on the sample table).
 - Pack, seal, and label wet samples
 - Record sample information in the LIMS
1. Prepare sample for analysis (drying, powdering).
 - Freeze-dry the sample
 - Grind and homogenize sample
1. Weigh sample.
 - Wrap powder samples in tin film (cup), weigh, and place in sample holder
 - Save weight data in the LIMS

1. Output sample list from LIMS.
 - Select samples that are ready for analysis
 - Output analysis sample list from the LIMS
5. Upload sample list to *Eager 300* software.
6. Edit sequence table.
 - Manually add **Bypass** sample line to the sequence table
 - Add a blank line to stop the sequence at any time
7. Run test analysis and samples.
 - Confirm peak identification and integration parameters
 - Create calibration curve
 - Start analyzing unknown samples
8. Output data summary (Excel file).
9. Upload data summary and files to LIMS.

Starting the Instrument

Perform any required maintenance before starting the system (see *Instrument Maintenance* section).

Powering on the System

Proceed according to the following operating sequence. Early start up (6 hr before measurement) is recommended for this instrument.

1. Open gas line/bottles.
2. Power on the FlashEA 1112.
3. Power on the PC and monitor.

Creating Data Folders and Copying Method File

The sequence of data files must be saved in the folder with the method file. Create new folders in the following directory: *My Computer\Local Disk\Program files\Thermo Electron\Eager 300 for EA1112\Expedition No. & abbreviation\data 001-200*. If there are more than 200 expected samples, create two or more data folders (e.g., *data 201-400*).

Starting the Instrument Operating System

1. Double-click on the **Eager 300 for EA 1112** icon.
2. Select **CHNS** from the list and click **OK** to answers prompted step by step (*Figure 7*).

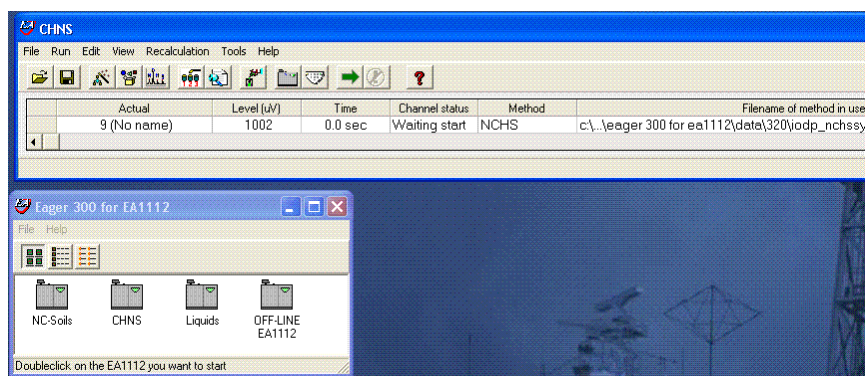


Figure 7. CHNS Screen

Checking for Leaks

1. Select **Edit > Edit Elemental Analyzer** parameter.
 2. Select the **Flow / Timing** tab (Figure 8).
 3. Select (check) the *Carrier*, *Oxygen*, and *Reference* parameters.
 4. Click **Send** to transfer the parameters.
 5. Click **OK** to close window.
 6. Click **NO** to exit without saving the method.
 7. Select **View > View Elemental Analyzer Status**.
 8. Select the **Special Functions** tab (Figure 9).
 9. Click **Leak Test** to open the *Leak Check* screen.
 10. Click **Start** to begin the operation.
 11. Click **Yes** for the Eager 300 to automatically test for leaks.
 12. After a few seconds, the reference gas flow must be 0.
- Note:** After ~560 sec, carrier flow will decrease. Carrier flow must be between 0 and 3 mL/min. Higher values indicate the system is not leak free. Leaks in the system are generally due to incorrect closure of the reactor and filter locking nut. Rarely, leaks may be due to the autosampler.

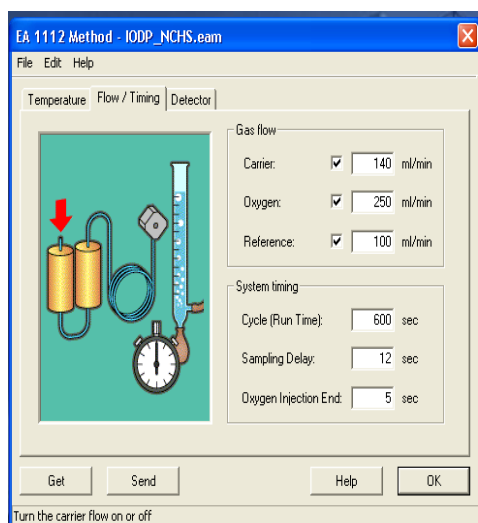


Figure 8. Flow/Timing Tab.

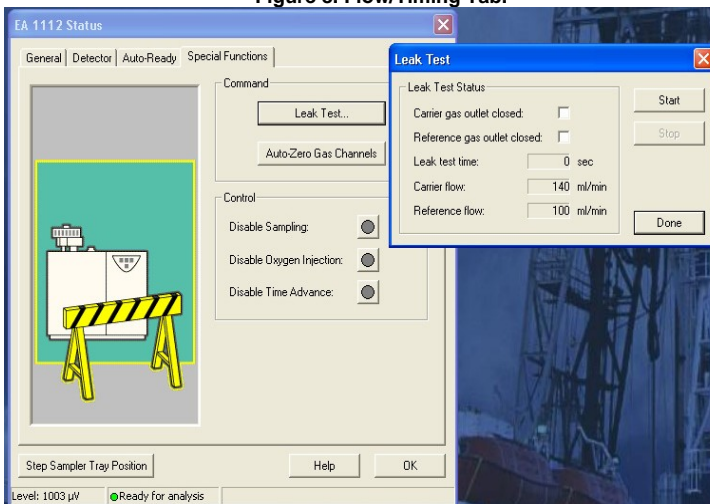


Figure 9. Special Functions Tab and Leak Test Screen.

Copying and Loading Method

1. To copy and load a previous method, select **File > Copy method from...** (Figure 10).
2. Uncheck the **Sample table** check box.
3. Click the button to the right of **Method File...** to select the method.
4. Select the method file **IODP_NCHSsystem** from the folder that has the previous expedition number and click **Open**.
5. Select **Edit > Edit Elemental Analyzer parameters**.
6. Click **File** and select the folder that has last expedition number.
7. Open file *IODP_NCHS.eam*.
8. Click **File > Save as** to save instrument settings into the created folder. **Note:** Your created folder now contains three files with extensions MTH, EAM, and CC.
9. Select **File > Save method**.

10. Use same method file that you copied in an earlier step and select the folder that you created.
11. Click **Save** to save the copied method in the new folder.
12. Select **File > Load method > Load New Method**.
13. Select the folder that contains the copied method and click **Open** to load method.
14. Click **Send Parameters to Analyzer** to send the method to the instrument.
Warning: When the furnaces begin to heat, helium flows in the circuit and the Furnace, Oven, and TCD lights illuminate on the synoptic panel.
15. Select **View > View Elemental Analyzer Status** to confirm status of instrument.

If the analytical configuration changes from CHNS to CNS, select **File > Instrument Configuration** on the main screen before editing the method (see instructions in the FlashEA Operating Manual, p. 217–221).

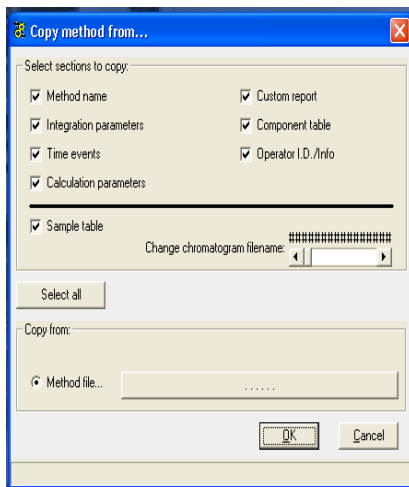


Figure 10. Copy Method from Screen.

Checking Maintenance Status

If a reactor is changed, the maintenance program must be reset. The maintenance program shown in Figure 11 gives an indication of reactor life. Reactor deterioration can also be detected on chromatograms.

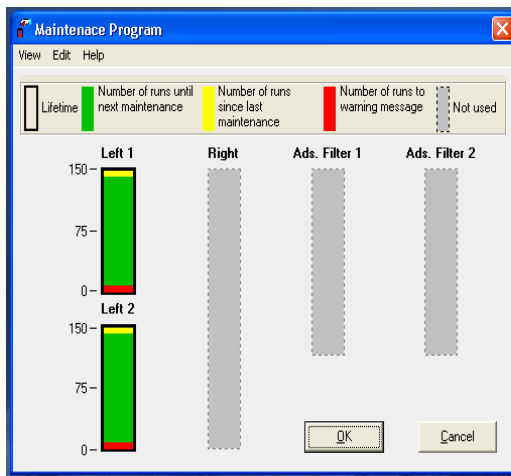


Figure 11. Maintenance Program Screen

Reactor life is also influenced by the nature of samples, especially water content. So you may have to check for anomalies on the chromatogram on a regular basis, as follows:

1. Select **View > View Maintenance**. The *Maintenance Program* screen shows the reactor status (Figure 11). To reset the maintenance program, select **Edit > Reset Maintenance Program**.
2. Select **View > Maintenance Status** to see the current settings. The program can be changed in two ways.
3. Click **Edit > Default**. Original settings for **Left 1** and **Left 2** were

- Lifetime: **200**
- Number of runs to warning message: **10**

4. Click **Edit > Manual**. Based on onshore tests, change the settings to

- Lifetime: **170**
- Number of runs to warning message: **10**

5. Click **OK** to save changes.

Editing the Method

Select **Edit > Edit Element Analyzer parameters**. The following parameters will display:

–Temperature tab:

- *Left Furnace*: generally not checked
- *Right Furnace*: checked, **900°C** default (keep value)
- *Oven*: checked, **65°C** default (keep value)

–Flow / Timing tab:

- *Carrier*: checked, **140** mL/min (This can be changed to achieve peak separation).
- *Oxygen*: checked, **250** mL/min (default)
- *Reference*: checked, **100** mL/min (default)
- *Cycle (Run Time)*: **600** sec (default)
- *Sampling Delay*: **12** sec (default)
- *Oxygen Injection End*: **5** sec (default)

–Detector tab:

- *Filament On*: checked
- *Time Event*: not checked

Note: Peak retention times are confirmed based on the chromatogram of the **Bypass** sample obtained using standard materials, which is then used to validate the calibration curve.

Editing the Component Table

1. On the *CHNS* screen, select **Edit > Sample table** (*Figure 12, below*). Clear the sample table and create two new rows.
2. Fill in the sample table as follows:

–Row 1:

- *Sample name* = **Blank test**
- *Filename* = **Blank_test_ddmmyy**
- *Type* = **blank**

–Row 2: Insert one blank line to stop measurement automatically after the blank.

–Row 3:

- *Sample name* = **Bypass test**
- *Filename* = **Bypass_test_ddmmyy**
- *Type* = **Bypass**

1. Prepare **Blank** (tin cup with V2O5) and **Bypass** using a standard material (–0.6–0.7 mg of sulphanilamide or BBOT with V2O5 and tin cup). Place the **Blank** in the sample tray of the MAS 200 R autosampler for the FlashEA 1112.
2. **Note:** Acquire a chromatogram that contains all the components to be identified.
3. Click **OK** to close the sample table.
4. Select **File > Save method** to save the sample table.
5. On the *CHNS* screen, click the green arrow toolbar icon to start running the samples.
6. Select **View > View sample being acquired** to confirm run status.

Note: Confirm chromatogram from the **Blank** to check the condition of the elemental analyzer. If the chromatogram has unexpected peaks and/or an unacceptable (disturbed) baseline, identify the cause of problem before running the **Bypass** sample. If the **Blank** is fine, continue measurement of **Bypass**.

1. After the **Bypass** sample is done, select **Edit > Component table** to identify the analysis peaks (*Figure 13, below*). Instructions for editing the component table are given in the Eager 300 Software Operating Manual, p. 101–115.
2. Select **File > Load Chromatogram**, and select **bypass test-ddmmyy** (last sample run).

10. Click **Open** to view the chromatogram.

11. Select **Time** of any component on the table.

12. Move the cursor near the red-gray arrow on the screen and right-click.

13. Adjust the red line on the screen to intersect the top of the peak.

14. Repeat Steps 11–13 for each peak on the chromatogram, and click **OK** to save changes.

Warning: If peak retention times have changed a lot, measure an additional **Bypass** sample to confirm the new peak positions. Possible causes of peak retention time shifts include (1) gas leaks (include outside of the instrument), (2) gas flow changes, (3) atmospheric pressure changes, and (4) others.

A	Sample name	Filename	Type	Standard name	Weight	Protein F.	Category	Sampler
4	ST_OTHR469401	ST_OTHR469401_4499731_3	Std	Sulfanilamide	6.295			
5	ST_OTHR469411	ST_OTHR469411_4499761_4	Std	Sulfanilamide	7.1297			
6	ST_OTHR469421	ST_OTHR469421_4499791_5	Std	Sulfanilamide	9.9814			
7	ST_OTHR469431	ST_OTHR469431_4499821_6	Std	Sulfanilamide	10.7989			
8	ST_OTHR469371	ST_OTHR469371_4499641_8	Std	Sulfanilamide	4.0533			
9	Act.		Bypass					

Figure 12. Sample Table.

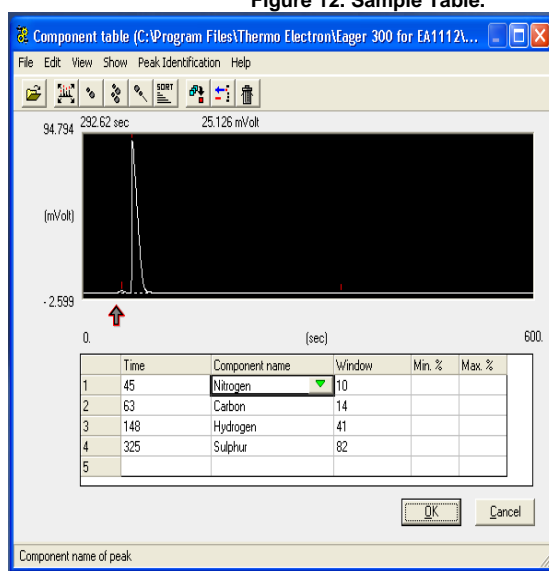


Figure 13. Component Table.

Editing the Integration Parameters

1. After finishing peak identification in the previous section, select **Edit > Edit Method** on the *CHMS* screen. Method editing instructions are shown in the Eager 300 Software Operating Manual, p. 74–76.
2. Select the **Integration parameters 3** tab and select **Test Integration** (Figure 14).
3. Select **File > Load Chromatogram**.
4. Load the chromatogram for the last test result (**Bypass**).
5. Select **Detect > Peak Integration**.
6. Four default integration parameters will be indicated. To customize detailed settings, click **Time events**, then **Peak Detect**. (Figure 15).
7. To edit the integration parameter table, select any peak time.
8. Move the cursor near the red-gray arrow. The cursor will convert to a narrow white- black arrow. Left-click to move the arrow.
9. Click **Type of Event** in the table, and then click the green inverted triangle.
10. Choose a type of event and click **OK** to close the window.
11. Click **OK** to save the modification.
12. Click **OK** on the *Peak detect* screen and then close the *Chromatogram* screen.

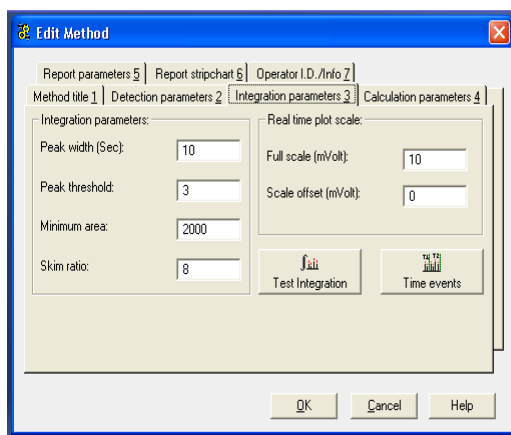


Figure 14. Edit Method Screen.

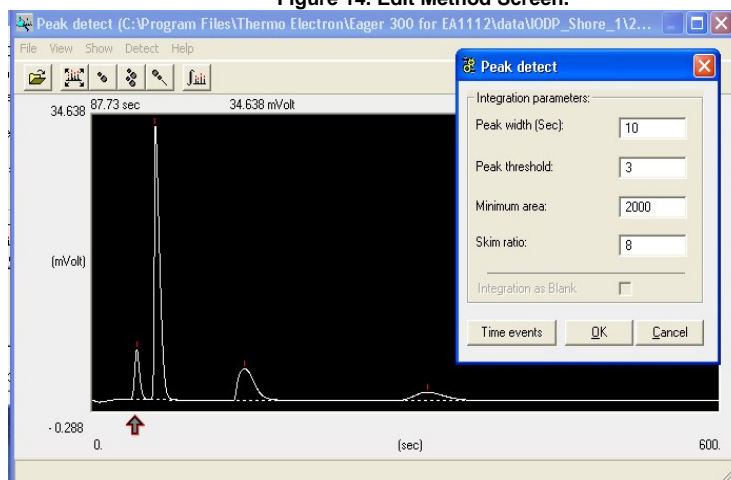


Figure 15. Peak Detect Screen.

Editing Other Parameters

Select the **Calculation parameters 4** tab on the *Edit Method* screen to select the calibration method. *Eager 300* has three calibration methods, details of which are described in the FlashEA 1112 Operating Manual, p. 232–234. Generally, we use **Linear fit** for sediment samples. **K-Factor** is sometimes useful for samples that are characterized by low component concentrations.

Running Samples

Proceed to measuring samples, see the *CHNS Analyzer User Guide*.

Shutting Down the Instrument

At the end of the cruise, if needed, shut down the system following the procedure below:

1. Select **Edit > Edit Element Analyzer** parameters.
2. Select **File > System Defined Method Folder** in the Eager300 for EA1112 folder.
3. Select **Shutdown.eam**, and then click **Open** to open shutdown setting.
4. Click **Send** to send opened setting to the EA 1112.
5. Monitor temperature change until oven temperature reaches <200°C.

Maintenance and Troubleshooting

Installing the Reactor in the Furnaces

Warning: The reactor must be installed with the furnaces at room temperature or <50°C with gloves. Do NOT use any mechanical tools to screw or unscrew the fixing nut.



Figure 16. Autosampler Removal.

See the FlashEA 1112 Operating Manual, p. 126–129 and 134. Pages 306–308 are also useful when replacing the O-ring of the coupling unions.

1. Loosen fixing nut and remove autosampler (*Figure 16, above*).
2. Delicately remove the used reactor (*Figure 17, below, 2*) from the right furnace.
3. Detach O-ring from the used reactor. If O-ring is undamaged, you can use it again.
4. Gently cleanup coupling union (*Figure 17, 1*) and O-ring. Wipe metal with alcohol if needed, but **do NOT** apply alcohol to O-ring. If using alcohol, apply a small amount of Vaseline to the O-ring to fix the cleaned coupling union.
5. Put a kimwipe or aluminium foil on the coupling union (*Figure 17, 3*) to protect cleaned coupling union from dust.
6. Delicately introduce the reactor into the furnace, ensuring that the tube conical end is turned downward toward the base (*Figure 17, 4*).
7. Gently press the edge of the reactor until introduction is complete.
8. Slip on the O-ring with its conical section turned upward.
9. Manually screw the fixing nut.

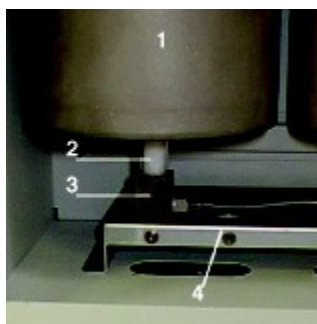


Figure 17. Furnace Reactor.

Cleaning the Autosampler

The autosampler does not normally require maintenance except for the sample tray. However, when the instrument is extensively used, it is a good practice to occasionally check the shaft housed in the autosampler.

Cleaning the Sample Tray

Review the FlashEA 1112 Operating Manual, p. 309–313. Once per week or whenever the autosampler is dirty, clean the sample tray as follows:

1. Detach the sample tray from the autosampler.
2. Using a Kimwipe and ethanol, wipe the holders and protective cover of tray.
3. Place the holder with protective cover over the autosampler.

Cleaning the Shaft of the MAS 200 R Autosampler

If you detect a gas leak in the autosampler (see "Test for Leaks"), clean the shaft of the MAS 200 R. The cleaning method is shown in the FlashEA 1112 Operating Manual, p. 314–318.

Note: If you replace a shaft, you must send the old shaft back to vendor, CE Elantech, Inc., to maintain a shaft.

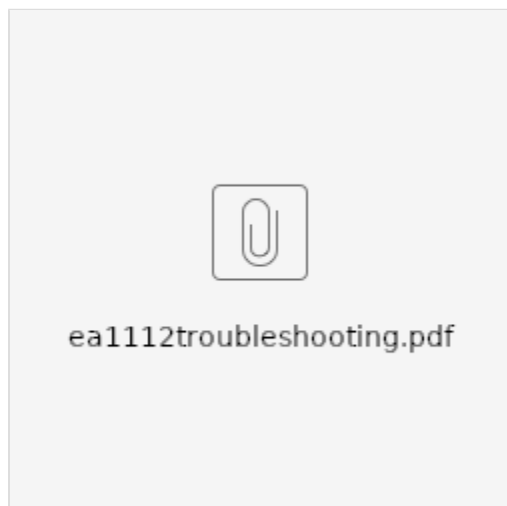
Connecting the Water Trap (Adsorption Filter)

Warning: Wear gloves and face protector when preparing the adsorption filter. Work with quartz wool under the draft chamber because quartz wool is an inhalation hazard.

1. Introduce into either of the tube ends a sufficient amount of quartz wool to form a layer ~1 cm (1/3 in.) thick.
2. Plug the tube mouth with your hand and press the quartz wool gently using a cleaned stainless steel spatula.
3. Screw the nut completely with its seal onto the thread mouth.
4. Pour the filling material (magnesium perchlorate) into the adsorption filter.

5. At the top, add a 1 cm (1/3 in.) layer of quartz wool.
6. Screw the second nut completely with its seal.

Common issues and Troubleshooting



Environment, Health, & Safety

Laboratory Safety and General Operation Training

Offshore laboratory specialists must complete all mandatory training before sailing. In addition to the general laboratory safety training, offshore laboratory specialists must complete general user training at the onshore facility. The CHNS User Guide has more information on safety related to this system.

High-Pressure Gas Safety

High-pressure gas lines, particularly the O₂ line, can be hazardous if there is leak. Check for gas leaks at gas fittings at the instrument regularly.

Other Safety Issues

The FlashEA 1112 has a safety cut-off function to prevent hazards. When an alarm condition is detected, this device cuts off the power to the heating elements of the oxidation and reduction furnaces. Instrument malfunctioning, because of component failure or abnormal operating conditions, is identified by the red safety cut-off LED on the synoptic panel. Reference: FlashEA1112 Operating Manual, p. 23–24.

Material Safety

Wear gloves and face protector when working with quartz wool. Also work under the draft chamber because quartz wool is an inhalation hazard. Vanadium pentoxide and magnesium persulfate are also significant hazards; be sure to read the material safety data sheet (MSDS) for each and every reagent and chemical, including the standards, used in this procedure!

Software User Guide

The *CHNS* screen of the *Eager 300* software application is the starting point to enter all functions.

–**File:** instrument operation functions:

- Set language...
- Color set
- Instrument name and config
- System administration
- Installation qualification
- Load method
- Load system defined method
- Save method
- Copy method from...
- Printer setup
- Print method
- Exit Eager 300

-Run: start, stop, or abort sequences:

- Start sequence of samples
- Stop running sequence
- Start single sample data acquisition
- Stop data acquisition
- Abort data acquisition
- Run macro

-Edit: instrument setup and analytical parameters:

- Edit Method
- Component table
- Sample table
- Wizard method development
- Edit Elemental Analyzer parameters

-View: analysis and chromatogram functions

- View sample being acquired
- Last sample calculated results
- View Calibration curve
- View Chromatograms
- Overlay Chromatograms
- Operate on Chromatograms
- Compare Chromatograms
- View Elemental Analyzer Status
- View Maintenance

-Recalculation: cancel or recalculate calibration or results

- Reset calibration factor
- Recalculation
- Summarize results

-Tools: reactor maintenance functions

- Ashes removal
- Reactor replacement

-Help