

CHNS Quick Start Guide

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CHNS Analyzer: Quick Start Guide
v.362T, v.371T, v.378P

Introduction

The CHNS procedure measures nitrogen and total carbon (inorganic plus organic). Organic carbon content is then calculated by using the inorganic carbon value from coulometric analysis. Hydrogen and sulfur can also be analyzed on the CHNS.

General Safety

The user should be familiar with the hazards related to the use of this system before beginning work (c.f. CHNS User Guide for more information). High-pressure gases, high temperatures, and toxic chemicals are all present for CHNS work.

Sample Preparation

Samples are freeze-dried, crushed, and homogenized using a mortar and pestle or electric mill and weighed into a tin sample cup (crucible). If sulfur is being analyzed, vanadium pentoxide is also added, acting as a catalyst. The crucibles are then closed (referred to as "wrapping" the sample) for instrumental analysis.

The following amounts are used:

- Standards: 3, 5, 10, 15, and 20 mg
- Unknown samples: 12–15 mg
- Vanadium pentoxide (if used): ½ small spatula for blanks, standards, and unknowns.

Worklist Generator

Use the Worklist Generator application to identify the samples, weighed on the Cahn balance, to be run on the CHNS Analyzer. Export the sample list to the current Expedition's folder. This file will be imported in the CHNS software as the sequence table.

Instrument Operation

Run the program *Eager Xperience*. Select *Analyzer #1*.

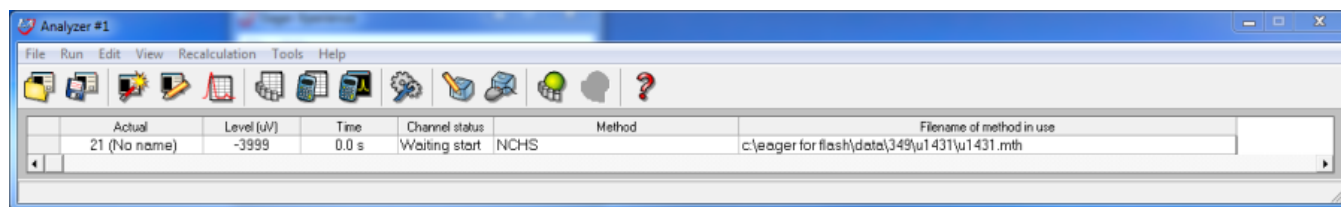


Figure 1: Eager Xperience main screen; the arrow points at the analyzer selection.

Instrument Parameters

- Helium pressure: 260 kPa
- Right furnace: 950°C

- TCD on: ~1400 µV
- Oxygen pressure: 300 kPa
- Oven: 60°C

Combustion Column

- A combustion column will last approximately 100–120 samples.
- To condition a new column, run the standard 2–3 times as a BYPASS.

Sulfur and Hydrogen

- If analyzing for sulfur, add vanadium pentoxide to the sample cups.
- If analyzing for hydrogen, remove the magnesium perchlorate trap. Run time will be around 1200 seconds.

Calibration

The instrument is calibrated at the beginning of each sequence. The calibration factors will first need to be reset:

Recalculation > Reset Calibration Factors

Immediately after the standards have been run, according to the sample table, it is a good idea to check the calibration:

View > View Calibration Curve (select the element of interest)

If the calibration is not acceptable, it is a good idea to immediately stop the sequencing, redo the standards, and start the sequence again, so no samples will have to be re-weighed.

Calibration is verified during the sequence by running the standard as an unknown.

Running Samples

1. Upload the LIMS sample table (Figure 2) to the instrument:

Edit > Sample Table

Edit Sample > Import sample table from LIMS (select the file exported from Worklist Generator)

A	Sample name	Filename	Type	Standard name	Weight (mg)
1	Act. BYPASS	BYPASS26	Bypass		
2	BLANK	BLANK6	Blank		
3	PwDR3472541	PwDR3472541_57791651_3	Std	thermo33840025	2.4801
4	PwDR3472541	PwDR3472541_57792011_4	Std	thermo33840025	5.1286
5	PwDR3472541	PwDR3472541_57794651_5	Std	thermo33840025	10.6416
6	PwDR3472541	PwDR3472541_57797101_6	Std	thermo33840025	15.4672
7	PwDR3472541	PwDR3472541_57792711_7	Std	thermo33840025	21.239
8	BYPASS	BYPASS27	Bypass		
9	SPCM5432911	SPCM5432911_57787161_9	Unk		16.5878
10	SPCM5433301	SPCM5433301_57787171_10	Unk		16.5341
11	SPCM5433271	SPCM5433271_57787181_11	Unk		16.7648
12	SPCM5433711	SPCM5433711_57787191_12	Unk		15.2452
13	SPCM5431811	SPCM5431811_57788461_13	Unk		14.0044
14	SPCM5433241	SPCM5433241_5778841_14	Unk		15.1217
15	SPCM5431321	SPCM5431321_57788551_15	Unk		16.2183
16	SPCM5431591	SPCM5431591_57789481_16	Unk		15.5839
17	SPCM5433041	SPCM5433041_57790111_17	Unk		14.5626
18	SPCM5433151	SPCM5433151_57791341_18	Unk		15.9234
19	PwDR3472541	PwDR3472541_57793671_19	Unk		12.1409
20	BYPASS	BYPASS28	Bypass		
21					
22					
23					

Figure 2. Sample table

The sample/sequence table should look similar to the one above.

- Column A: Act signifies which is the current sample
- Sample name:
 - BYPASS
 - BLANK
 - TEXT ID

- Filename: A unique name. Worklist Generator creates the names for the samples
- Type:
 - Bypass (empty slot in autosampler)
 - Blank (empty tin capsule)
 - Std (calibration standard)
 - Unk (sample or standard)
- Standard name: only the calibration standards will have this field filled in.
- Weight: Values from Worklist Generator are automatically filled in for standards and samples.

2. Load the autosampler tray with the standards and samples; leave empty slots for BYPASSes. Make sure that the lid is on the tray and put on the autosampler. Advance the zero slot to slot one.

3. Run samples:

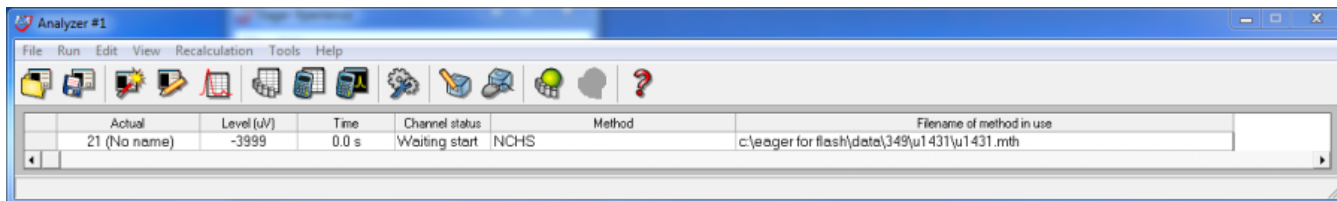


Figure 3. Eager Xperience screen; the arrow points at the run samples button

- Click the green arrow toolbar icon.
- Monitor acquisition status at View > View Sample Being Acquired.

Analyzing Data

- Go to **Recalculation > Summarize results**.
- Go to **File > Export to Excel File**. This creates the results file that the MUT will upload into the LIMS.
- Copy the file created in Step 2 to the MUT upload directory.

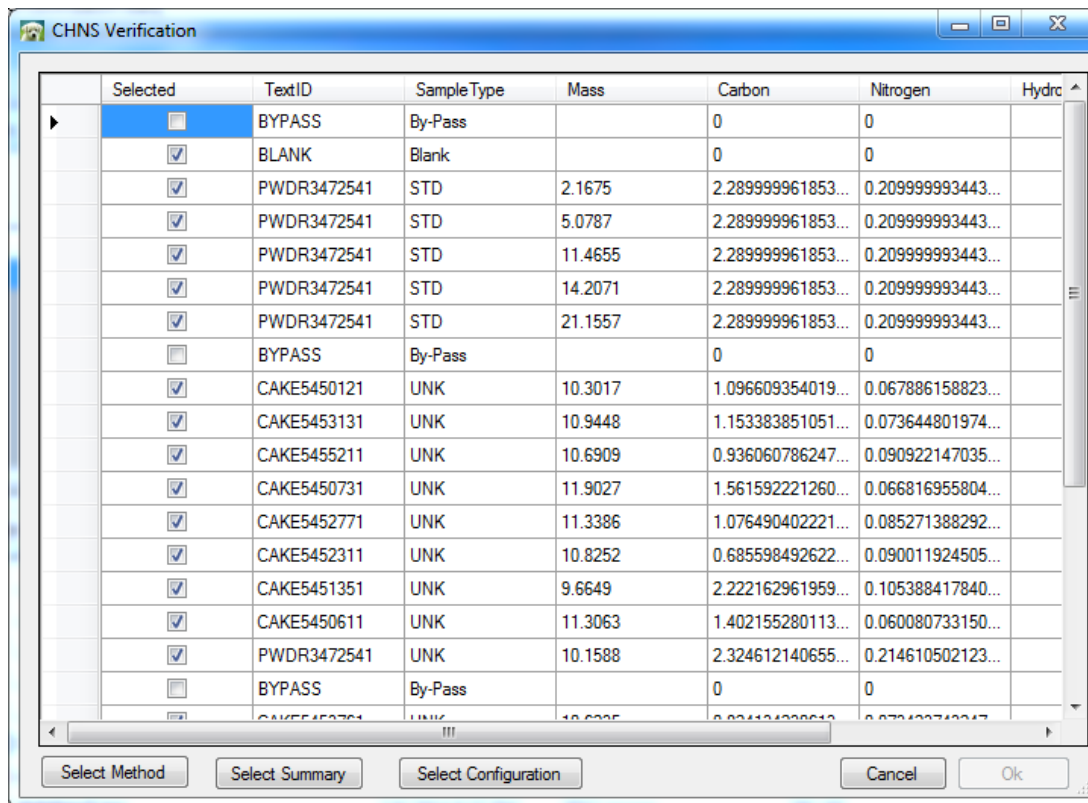


Figure 4. CHNS verification screen

4. In MUT, select **Upload** to open a window as shown above in Figure 4. Click on :

- a. Select Method: Choose your EA method file.
- b. Select Summary: Choose the summary file from the sequence just run.
- c. Select Configuration: Choose your method configuration file
- d. The **OK** button will then be made active. Click on it.