# **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.

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5	🗊 🗭 🦻		🗊 🗊	🖗 📎 🎜	l 🔐 🛑 🦻		
	Actual	Level (uV)	Time	Channel status	Method	Filename of method in use	
	21 (No name)	-3999	0.0 s	Waiting start NO	CHS	c:\eager for flash\data\349\u1431\u1431.mth	
4							•

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)

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File Ed	it sam	ple Verify chrom. file Ba	lance Help	1				
	A	Sample name	Filename		Туре	Standard name	Weight (mg)	ĺ ▲
1	Act.	BYPASS	BYPASS26		Bypass			
2		BLANK	BLANK6		Blank			
3		PWDR3472541	PWDR3472541	_57791651_3	Std	thermo33840025	2.4801	T
4		PWDR3472541	PWDR3472541	_57792011_4	Std	thermo33840025	5.1286	T
5		PWDR3472541	PWDR3472541	_57794651_5	Std	thermo33840025	10.6416	T
6		PWDR3472541	PWDR3472541	_57797101_6	Std	thermo33840025	15.4672	T
7		PWDR3472541	PWDR3472541	_57792711_7	Std	thermo33840025	21.239	Τ
8		BYPASS	BYPASS27		Bypass			T
9		SPCM5432911	SPCM5432911	_57787161_9	Unk		16.5878	Т
10		SPCM5433301	SPCM5433301	_57787171_10	Unk		16.5341	T
11		SPCM5433271	SPCM5433271	_57787181_11	Unk		16.7648	Τ
12		SPCM5433711	SPCM5433711	_57787191_12	Unk		15.2452	T
13		SPCM5431811	SPCM5431811	_57788461_13	Unk		14.0044	Τ
14		SPCM5433241	SPCM5433241	_57787841_14	Unk		15.1217	
15		SPCM5431321	SPCM5431321	_57788551_15	Unk		16.2183	Τ
16		SPCM5431591	SPCM5431591	_57789481_16	Unk		15.5839	T
17		SPCM5433041	SPCM5433041	_57790111_17	Unk		14.5626	Τ
18		SPCM5433151	SPCM5433151	_57791341_18	Unk		15.9234	
19		PWDR3472541	PWDR3472541	_57793671_19	Unk		12.1409	Τ
20		BYPASS	BYPASS28		Bypass			
21								
22								
23								-
								•
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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:

🤣 Ana	alyzer #1	A	Spectrum .				
File	Run Edit View Reca	alculation Too	ls Help				
5	📮 🗭 🦻		<b>a</b> 🔊	🖗 📎 🕹	🔉 😪 🌒 🤶		
	Actual 21 (No name)	Level (uV) -3999	Time 0.0 s	Channel status Waiting start	Method NCHS	Filename of method in use c:\eager for flash\data\349\u1 431\u1 431.mth	

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

a. Click the green arrow toolbar icon.

b. Monitor acquisition status at View > View Sample Being Acquired.

### Analyzing Data

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

Selected	TextID	SampleType	Mass	Carbon	Nitrogen
	BYPASS	By-Pass		0	0
1	BLANK	Blank		0	0
<b>V</b>	PWDR3472541	STD	2.1675	2.289999961853	0.20999993443
1	PWDR3472541	STD	5.0787	2.289999961853	0.20999993443
<b>V</b>	PWDR3472541	STD	11.4655	2.289999961853	0.20999993443
<b>V</b>	PWDR3472541	STD	14.2071	2.289999961853	0.209999993443
<b>V</b>	PWDR3472541	STD	21.1557	2.289999961853	0.20999993443
	BYPASS	By-Pass		0	0
<b>V</b>	CAKE5450121	UNK	10.3017	1.096609354019	0.067886158823
<b>V</b>	CAKE5453131	UNK	10.9448	1.153383851051	0.073644801974
<b>V</b>	CAKE5455211	UNK	10.6909	0.936060786247	0.090922147035
<b>V</b>	CAKE5450731	UNK	11.9027	1.561592221260	0.066816955804
<b>V</b>	CAKE5452771	UNK	11.3386	1.076490402221	0.085271388292
<b>V</b>	CAKE5452311	UNK	10.8252	0.685598492622	0.090011924505
<b>V</b>	CAKE5451351	UNK	9.6649	2.222162961959	0.105388417840
<b>V</b>	CAKE5450611	UNK	11.3063	1.402155280113	0.060080733150
<b>V</b>	PWDR3472541	UNK	10.1588	2.324612140655	0.214610502123
	BYPASS	By-Pass		0	0
[TTM]	CALCE ADDRES	LINUZ	10.0005	0.004404000040	0.070400740047

#### 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  $\mathbf{OK}$  button will then be made active. Click on it.