

**Perkin Elmer 2400 Elemental Analyzer Operating Instructions**  
**For CHN Analysis**  
(Rev III ; 07 October 08)

**Sample And Standard Preparation**

- \* Clean all weighing utensils and glass plate with water & acetone.
- \* Calibrate the microbalance before & after each weighing session following the instructions on flip chart on top of balance.

\*Weight guidelines for capsule samples:

- Acetanilide Standard = 2 mg +/- 0.2
- Organic Sample = 2 mg +/- 0.2
- Inorganic Sample = 15 mg
- Buffalo River Sediment (RM #8704) = 15 mg
- Apple Leaves (SRM #1515) = 2 mg

A. Preparing Capsule Samples and Standards

1. Working in 20 mg range, tare the weight of your capsule with another capsule as a counterbalance on reference (left) pan.
2. Place capsule in black holder.
3. Add appropriate amount of sample / standard to capsule.
4. Check weight; adjust if necessary.
5. When capsule contains correct amount of sample/std, fold as shown in figure 4-8 on p. 4-57 of Operator's Manual.
6. Re-weigh. Record weight on 'CHN Sample Prep and Run Sheet'.
7. Place sample/std in clean 96 well storage tray and record tray position.

Notes:

- \*Avoid cross contamination of samples / stds by cleaning all tools between groups
- \*Minimize exposure of standard to light and air

B. Preparing Filters

Either of the following two methods may be used for filter analysis:

Method 1

1. Weigh an ashed filter on 200 mg range using large stirrups. Record weight. Wrap in pre-cleaned aluminum foil and label.
2. Filter sample.
3. Dry filter at least overnight in 110°C drying oven.
4. Cool filters in dessicator.
5. Re-weigh and record final weight. Difference = sample weight.
6. Wrap filter in tin disk as compactly as possible. If sample is too long / fat it will lodge in injection port and will prevent subsequent samples from dropping. **All data for these will be lost!!**
7. Analyses are run on Single-Run, Non-Filter Mode (see below). Data are reported in terms of ug C/H/N on filter.

Method 2

1. Pass a known, recorded volume (measure accurately with TD grad cylinder) of sample thru ashed filter. Rinse down sides of filter funnel and grad cylinder to ensure all particulates are collected on filter.
2. Dry and cool filters as above on clean aluminum pan.
3. Follow step 6 above.
4. Analyses are run on Single-Run, Filter Mode (see below). Data are reported in terms of ug C/H/N per mL of sample filtered.

## Instrument Startup

1. You **MUST** provide an account number before you use the instrument! Open the helium (if closed) and oxygen cylinder valves.

*\*If instrument is already on and in **Gas Saver** mode, go to # 6.*

2. Turn on the power to the instrument and printer. **ENTER** time, date (dd mm yy), and name using keypad. Fill pressure should be set to 6.488 V; press **ENTER**.
3. Record run counter values on log (press **ENTER** to toggle thru).
4. Confirm combustion temp = 980 °C and reduction temp = 640 °C.
5. If pressure test is completed successfully, you will be prompted to purge with Helium & Oxygen. At this time answer **NO** to both. If pressure test fails, instrument will not proceed with warm-up. See Tom or Linda for troubleshooting.

6. Confirm that the following parameters are in their proper operating modes by pressing **PARAMETERS**, then the parameter number, then **ENTER**.

PARAMETER = 6	Operating mode: CHN
PARAMETER = 10	Lower temperatures: OFF
PARAMETER = 12	Furnace: ON
PARAMETER = 20	Oxygen valve: ON
PARAMETER = 21	Reduction valve: OFF
PARAMETER = 22	Gas save valve: OFF (This will need to be turned off if starting from <b>Gas Saver</b> mode)
PARAMETER = 32	NO; must be turned on before running samples if you are using filter method 2
PARAMETER = 34	NO

*If starting from **Gas Saver** mode:*

- Enter Operator ID ( PARAMETER 5)
- Check Run Counters (PARAMETER 4)
- SKIP # 7; Go directly to # 8.

Press **PARAMETERS** again to return to "STANDBY" mode.

7. Allow 2.5 hours for the instrument to equilibrate. Combustion and reduction temperatures should reach 980 °C and 640 °C, respectively. Normally, **MESSAGE 23** will appear during warm-up indicating an unstable detector signal; disregard.

8. Press **PURGE GAS** and **ENTER** the following values:  
Helium: 300 seconds  
Oxygen: NO

9. While purging, turn on computer (next to TOC-5000).

Open CHN Data Acquisition Excel file.

Do not minimize Software Wedge.

Rename Excel template to new file name (eg. Date-name) Select File, "Save As"

Confirm that cursor is on cell A:2 before running instrument blanks.

10. Check the screen and make sure it says the instrument is in "STANDBY" mode.

11. Press the **DIAGNOSTICS** key and follow this order:

2	GAS	
2	VALVE	
4	VALVE 4	this selects valve "D"
1	ON	this turns on valve "D"
5	VALVE 5	this selects valve "E"
1	ON	this turns on valve "E"

wait for about fifteen seconds and then press the **DIAGNOSTICS** key again (this automatically closes the valves you just opened and also exits from the diagnostics mode)

12. Press **MONITOR** -> **PRINT LIST** -> **YES**

Confirm the following values:

<u>category</u>	<u>value</u>	<u>limit</u>
Combustion temp. (C)	925	+/- 2
Reduction temp. (C)	640	+/- 2
Detector oven (C)	82.6	+/- 2
Pressure (mmHg)	15	+/- 13 (after valve bleed)
Detector (counts)	4000	+/- 1000

If these are OK you may proceed; press **MONITOR** to return to Standby. If not seek assistance.

13. To stabilize the system run Instrument Blanks as follows:

- Press **SINGLE RUN**
- Press **1** for BK (blank)
- Enter 4** for Number of Runs -- the system will switch to the "**STANDBY**" mode
- Make sure the sample drop area is clean (blow out if necessary)
- Press **START**
- Record data on log sheet

14. Check for precision within the specifications listed below for blanks. If C, H, & N values of at least the last 2 blanks meet these criteria you should proceed. If not, continue to run instrument blanks one at a time until you achieve this precision. This should require no more than 5-6 runs at the maximum.

## Calibration (Start below at #1)

The procedure for running blanks (empty tin capsule) is as follows

- Press **SINGLE RUN**
- Press **1** for BK (blank)
- Enter 1** for Number of Runs -- the system will switch to the "STANDBY" mode
- Drop blank into funnel in sample drop opening at top of Auto-Injector; close lid
- Press **START**

The procedure for running K-Conditioning (standard as sample) is as follows:

- Press **SINGLE RUN**
- Press **3** for SAMP
- Enter "KASSx"** (no spaces) for ID using numbers corresponding to position of letters in alphabet (see chart on front of instrument)
- Enter the weight in mg
- Drop standard into funnel in sample drop opening at top of Auto-Injector; close lid
- Press **START**

The procedure for running K-Factors (standards) is as follows:

- Press **SINGLE RUN**
- Press **2** for KFACT
- Press **1** for Standard S1 (acetanilide)
- Enter the weight of the standard in mg
- Drop standard into funnel in sample drop opening at top of Auto-Injector; close lid
- Press **START**

1. Run the following calibration sequence using the **SINGLE RUN** mode. Record data on log sheet.

1. blank
2. K-Conditioning
3. blank
4. K-Factor
5. blank
6. K-Factor
7. blank
8. K-Factor

**\*NOTE:** Often, the PE 2400 will fail to average the Carbon values for K-factors. These values should be monitored to determine whether averaging is being performed during your calibration. If not, you must calculate the average of the Carbon values for the three K-factor runs and manually enter this as the "C" value in the instrument memory: (**PARAMETERS 2 → ENTER**)

2. If the precision criteria listed below are achieved continue to step 3. If not, continue with sequence alternating blanks & K-Factors. Seek assistance if sequence exceeds 10.

Minimum Precision Criteria for Blanks and K-Factors are as follows:

### **BLANKS:**

<u>category</u>	<u>value</u>	<u>reproducibility</u>
C	*	+/- 30
H	*	+/- 100
N	*	+/- 16
Fill time	30 sec.	+/- 10

\* blank values can be any positive or negative number in these categories

**K-FACTORS:**

<u>category</u>	<u>value</u>	<u>reproducibility</u>
weight (mg)	2.0	+/- 0.2 (optimal)
KC	*	+/- 0.15
KH	*	+/- 3.75
KN	*	+/- 0.16
Fill Time	24 sec.	+/- 5

\* Typical values are:

C	16.5 +/- 3.5
H	50.0 +/- 20.0
N	6.0 +/- 3.0

3. Run 1 K-Factor as Sample to check for recovery. If the % Bias for each element (use "QC Calculator") all fall within acceptance limits\*, proceed with analysis. If not, seek assistance.

4. Run 2 External Source QC's (RM # 8704 for soils/sediments, or SRM # 1515 for tissues). If the %CV and % Bias (use "QC Calculator") fall within 10 %, proceed with analysis. If not, seek assistance.

\*See Acceptance Limit Chart.

**Theoretical Values**

	% C	%H	%N
<i>Acetanilide Standard</i>	71.09 ± 0.40	6.71 ± 0.40	10.36 ± 0.40
<i>Buffalo River Sediment # 8704</i>	3.351 ± 0.017	Not Determined	0.20 ± 0.04
<i>Apple Leaves #1515</i>	Not Determined	Not Determined	2.25 ± 0.19

## Capsule Sample Analysis

Capsule samples can be analyzed in either the Single Run OR Auto Run modes. Procedures for running samples in each of these modes are outlined below.

### Single Run Mode:

1. Press the **SINGLE RUN** key
2. Press **3** for Sample
3. Key in Sample ID up to 12 characters, NO spaces; **ENTER**
4. Key in Sample Weight in mg; **ENTER**
5. Drop sample into funnel in sample drop opening at top of Auto-Injector; close lid
6. Press **START**
7. Repeat 1-6 for remaining samples / standards / blanks

### Auto Run Mode:

1. Press **AUTO RUN**
2. Press **4**
3. Press **1** to clear old run sequence from memory and reset run sequence to # 1
4. Press **3** for Sample; for blank or standard recoveries between groups of samples choose **1** or **2**, respectively
5. Enter ID and sample weight in mg (no weight req'd for blanks)
6. Place sample in appropriate position on clean Autosampler carousel; Dust off first if necessary
7. Record Auto Run # and Autosampler position of sample on data sheet
8. Remove funnel from sample port if installed.
9. Attach carousel to instrument and rotate until sample drops into injection port
10. Press **START**
11. While the first sample is running, repeat steps 4 - 7 for remaining samples / standards / blanks to be run in sequence. Sequence data (ID's & weights) can be printed out (**AUTORUN** → **4** → **2**) to check for entry errors.

To edit an entry in Auto Run Mode (i.e. weight, ID, run type):

Change number to run# to be edited.

Press **Parameters** key and make changes.

### **\*\*IMPORTANT\*\***

In both modes, a series of  $\leq 12$  samples is followed by a K-Factor run and then a blank run. Data for these should be recorded on the back of log sheet under "Recovery". Proceed with your next series of samples only if these data meet the above precision criteria when compared to the previous K-Factor and blank. If not, it may be necessary to run more K-Factors and blanks to re-calibrate the instrument. You should seek assistance if precision is not achieved within 3 additional runs of each.

**\*\*See additional NOTES below\*\***

## Filter Sample Analysis

1. Turn on Filter mode if using filter method 2 (**PARAMETERS → 32 → ENTER → ON**). Enter information at prompts; press **PARAMETERS** again to return to **STANDBY**.
2. Run the following sequence. Record all blank & K-factor data on log sheet.
  - A. 3 **FILTER** blanks (filter + tin disk)
    - Plug averages of the C, H, & N, values for the 2<sup>nd</sup> and 3<sup>rd</sup> blanks into instrument memory (**PARAMETERS 1 → ENTER..**) These values will be subtracted from filter samples to correct for filter background.
  - B. Set of samples not to exceed 10 (**SEE NOTES BELOW**)
  - C. **CAPSULE** blank. Compare to last CAPSULE blank
    - Plug in C, H, N values as described in A.
  - D. K-factor; compare to last K-factor.
  - E. **FILTER** blank; Plug in C, H, N values as described in A.
  - F. Repeat steps B - E for all sets of samples except last. For final set, run only B - D.

### **\*\*IMPORTANT\*\***

Proceed with your next series of filter samples only if these data meet the above precision criteria when compared to the previous K-Factor and blank. If not, it may be necessary to run more K-Factors and blanks to re-calibrate the instrument. You should seek assistance if precision is not achieved within 3 additional runs of each.

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### **NOTES (For ALL Analyses):**

\*Data generated before a K-Factor and/or blank that is out of tolerance should be considered questionable. Therefore, toward the end of the life of a reduction tube (~ 50 runs left on counter) it is wise to reduce the number of samples run between recovery checks, **ESPECIALLY FILTERS**.

\*Watch for increasing N blanks and abnormally high N for samples toward the end of a reduction tube's life. These indicate an exhausted reduction tube. If you see this happening discontinue your analysis and make a note of with which run the trend began. Further runs will only waste samples and reagents. If reduction tube is exhausted, furnace must be cooled to room temperature before replacement. See **Miscellaneous Operating Procedures (sec. E)** for instructions on shutting down furnace.

\*If an extremely low value is obtained during a filter run, suspect a sample drop problem (jam or miss)

## Shutdown

1. Save Excel data file.

In GEL Database, enter information in red fields after run is completed.

QC Type: Buffalo River Sediment for soil samples  
Apple Leaves for plant samples

Use QC Calculator to enter results of QC runs.

Enter number of capsules and filters.

Accept data.

Enter billing information and accept.

2. Bleed valves D (4) and E (5) as outlined below.

Press the **DIAGNOSTICS** key and follow this order:

2	GAS	
2	VALVE	
4	VALVE 4	this selects valve "D"
1	ON	this turns on valve "D"
5	VALVE 5	this selects valve "E"
1	ON	this turns on valve "E"

wait for about fifteen seconds and then press the **DIAGNOSTICS** key again (this automatically closes the valves you just opened and also exits from the diagnostics mode)

3. Put the system in Gas Saver Mode:

Follow these steps:

- A. press the **PARAMETER** key and enter the following:

22	Gas saver valve
1	Select ON

- B. Enter today's date (dd/mm/yy format) and time (24 hour format) for gas saver mode to turn on. A time about 2 minutes past the current time is suggested.

- C. Enter the date and time for the gas saver to turn off. This date should be a few years from now, just in case.

- D. Press the **PARAMETER** key to exit to the standby mode

## Miscellaneous Operating Procedures

### A. Aborting An Analysis In Mid-Run:

On occasion it is necessary to abort a run, for whatever reason. Although this does not damage the instrument, a few things need to be done to return it to good operating condition. In addition, the proper way to abort is less intuitive than other, improper, ways. To abort a run properly and recover the instrument afterwards, follow these steps:

1. Enter Standby mode from Auto Run by pressing **AUTO RUN**
2. Press the **SINGLE RUN** key twice.

**Note: Do not press the Standby key!**

To recover the instrument, repeat the bleed procedure as outlined below. This is to bleed the columns and the mixing volume of pressure build up. The system will not stabilize if this step is skipped.

press the **DIAGNOSTICS** key and follow this order:

- |   |         |                         |
|---|---------|-------------------------|
| 2 | GAS     |                         |
| 2 | VALVE   |                         |
| 4 | VALVE 4 | this selects valve "D"  |
| 1 | ON      | this turns on valve "D" |
| 5 | VALVE 5 | this selects valve "E"  |
| 1 | ON      | this turns on valve "E" |

wait for about fifteen seconds and then press the **DIAGNOSTICS** key again (this automatically closes the valves you just opened and also exits from the diagnostics mode)

### B. Pausing An Auto Run Sequence After Completion of Sample In Progress:

Press the **SINGLE RUN** key once; Auto Run sequence will pause before next sample.

### C. Putting The System in Gas Saver Mode:

Whenever the instrument is not going to be used for over an hour (extended break) it should be put in Gas Saver mode. This is to preserve the high purity (and expensive) gasses used by it. Follow these steps:

1. press the **PARAMETER** key and enter the following:

22	Gas saver valve
1	Select ON
2. Enter today's date (dd/mm/yy format) and time (24 hour format) for gas saver mode to turn on. A time about 2 minutes past the current time is suggested.
3. Enter the date and time for the gas saver to turn off. This date should be a few years from now, just in case.
4. Press the **PARAMETER** key to exit to the standby mode

### D. Clearing A Jammed Sample From Injection Port

1. Press **DIAGNOSTICS**
2. **2** - Valve
3. **11** - Valve 11
4. **1, ON** - Opens sample injector slide
5. Clear jammed sample(s)
6. Press **DIAGNOSTICS**; slide will close
7. Purge system with He for 300 seconds; **PURGE GAS** key
8. Bleed valves D (4) and E (5) as outlined above in section A
9. Resume run

## E. Shutting Down Furnace

**\*You must bleed the columns of pressure to avoid breaking combustion tube during cool down.**

press the **DIAGNOSTICS** key and follow this order:

2	GAS	
2	VALVE	
4	VALVE 4	this selects valve "D"
1	ON	this turns on valve "D"
5	VALVE 5	this selects valve "E"
1	ON	this turns on valve "E"

wait for about fifteen seconds and then press the **DIAGNOSTICS** key again  
(this automatically closes the valves you just opened and also exits from the  
diagnostics mode)

press the **PARAMETERS** key and follow this order:

12	Furnace
2	Off