Perkin Elmer 2400 Elemental Analyzer Operating Instructions
For CHN Analysis
(Rev VI; 15 December 2020)

Sample and Standard Preparation

* Clean all weighing utensils and glass plate with water followed by acetone.
*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. Tare the weight of your capsule on the microbalance.
2. Add appropriate amount of sample / standard to capsule.
3. Check weight; adjust if necessary.
4. When capsule contains correct amount of sample/std, fold as shown in figure 4-8 on p. 4-57 of Operator's Manual.
5. Re-weigh. Record weight on 'CHN Sample Prep and Run Sheet'.
6. 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. 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 desiccator.
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 through an 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 (from cold start-up)

1. Open the helium and oxygen cylinder valves. (if closed)

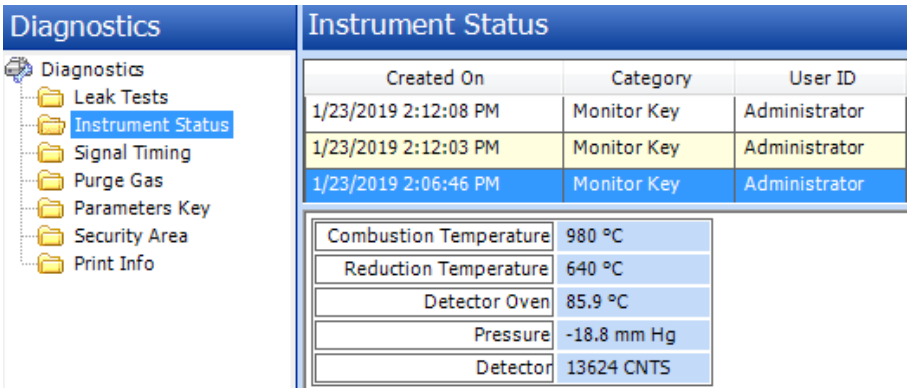
   If instrument is already on and in Gas Saver mode, go to # 6.

2. Turn on the power to the instrument. **ENTER** time, date (dd mm yy), and name using keypad. Fill pressure should be set to 6.488 V; press **ENTER**.

3. Confirm combustion temp = 980 °C and reduction temp = 640 °C.

4. 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 GEL staff for troubleshooting.

5. Confirm that the following parameters are in their proper operating modes and the instrument is connected to the software by pressing **MONITOR**, then **Yes** to print status. You should hear a beep and see a new line under "**Diagnostics**" in "**Instrument Status**" that displays today's date with the current parameters (similar to picture below). Make sure to scroll up to ensure today's date is there. If nothing appears and you didn't hear a beep reference step 5a below.



Confirm the following values:

Category		value	limit:
Combustion temp. (C)	980	+/- 2	
Reduction temp. (C)	640	+/- 2	
Detector oven (C)	82.6	+/- 3	
Pressure (mmHg)		15	+/- 13 (could be negative after valve bleed)
Detector (counts)		4000	+/- 1000

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

5a. Close the EA 2400 Data Manager software if it's already open. Re-open it. When prompted to enter,
the User ID is: Administrator
Password: seriesA

*The password may have changed, but check the computer monitor for most recent password.

Another message with a picture of the analyzer will appear. It will prompt you to press **PARAMETER 26** on the keypad of the instrument. You should hear a beep and see another screen detailing advanced settings. This means the instrument and Data Manager software are communicating and you may proceed.

Check that the below parameters are correct.

6. **PARAMETERS**, then the parameter number, then **ENTER**.
   PARAMETER = 12 Furnace: ON
   PARAMETER = 20 Oxygen valve: ON

PARAMETER = 22	Gas save valve: OFF (This will need to be turned off if starting from **Gas Saver** 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**) Initials and then press Enter**

**(This will put all your run data in your folder on the software. Otherwise it will end up in the folder of whoever ran before you)

-Check Run Counters (**PARAMETER 4**) note levels prior to analysis then press Enter

- 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. Check the screen and make sure it says the instrument is in "**STANDBY**" mode.
   If display does not say Standby at this point, instrument is in another mode which needs to be cleared by pressing button for that mode.

9a. Press the **DIAGNOSTICS** key and follow this order:

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

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)

10. To stabilize the system run Instrument Blanks as follows:
   -Turn off Oxygen Valve : PARAMETER = 20 Oxygen valve: OFF
   -Press **SINGLE RUN**
   -Press **1** for BK (blank)
   -Enter 4 (minimum) 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**
   -When Instrument Blanks are done running, Turn on Oxygen Valve : PARAMETER = 20 Oxygen valve: ON. NOTE: Oxygen Valve should remain turned on until next use of instrument

*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 folded 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 (K-factor 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 (should be to four decimal places)
-Drop standard into funnel in sample drop opening at top of Auto-Injector; close lid
-Press **START**

The procedure for running K-Factors (Acetanilide standard) 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. Entire sequence for instrument startup should be 4 air blanks; blank tin; KASSX; blank tin and a minimum of 3 K-factors. Once to the blanks and K-factors these can be run using the Auto-sampler if desired.

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

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:

Category		value	reproducibility
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:

Category	value	reproducibility
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 (KASSX) to check for recovery. If the % Bias for each element fall within 10% acceptance limits*, proceed with analysis. If not, repeat KASSX run, if values do not recover within 3 attempts then seek assistance.

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

***See Acceptance Limit Chart**.

QC & K-Factor Theoretical Values

	% C	%H	%N
Acetanilide Standard (K-Factor)	71.09 ± 0.40 or 67.155 to 75.064	6.71 ± 0.40 or 5.99 to 7.465	10.36 ± 0.40 or 9.462 to 10.898
Buffalo River Sediment # 8704	3.351 ± 0.17 or 3.167 to 3.519	Not Determined	0.20 ± 0.04 or 0.192 to 0.252
Apple Leaves #1515	Not Determined	Not Determined	2.25 ± 0.19 1.957 to 2.562
Bituminous Coal	76.88 ± 0.15 72.893 to 80.881	5.10 ± 0.05 4.797 to 5.407	1.59 ± 0.01 1.501 to 1.680
Algae	47.2 ± 2.36 44.84 to 49.56	6.929 ± 0.3465 6.583 to 7.276	10.72 ± 0.536 10.184 to 11.256

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 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. Adjust carousel clockwise to line up space 1 with the sample drop port.
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
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.

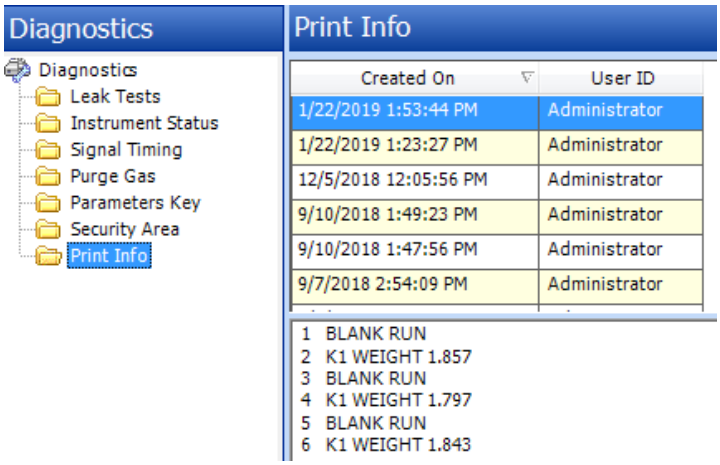
To edit an entry in Auto Run Mode (i.e. weight, ID, run type):
Change number to run# to be edited.
Press **Parameters** key and make changes.

****IMPORTANT****

In both modes, a series of $\leq$ 12 samples is followed by a K-Factor run and then a blank run. 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**

If you forget what you entered or want to reference the list of weights and samples you've already entered, press **AUTO RUN, then **4**, then **2** to Print List. The list will appear under "Diagnostics", "Print Info" (see picture below).



Filter Sample Analysis

1. Filter samples are packed in foil discs NOT tins/capsules; so all blanks, standards and QC samples should also be weighed into foil discs. Repeat same blank; KASSX; blank; K-factor sequence as listed for tins/capsules to achieve instrument calibration.

2. Run the following sequence of filter blanks.
   A. 3 **FILTER** blanks (filter + tin disk)
      - Plug averages of the C, H, & N, values for the 2nd and 3rd 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. **Foil** blank. Compare to last Foil 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.

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 which run the trend began. Further runs will only waste samples and reagents. If reduction tube is exhausted, furnace must be cooled by ramping down temperature to around 100-200 deg C 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. 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)

2. 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.

Press the **PARAMETER** key to exit to the standby mode

To Export Data

Check the box on the far left next to the samples/standards/blanks you would like to export. Click **File**, then **Export**. It will automatically export as an excel file. Save your file to a USB or in the Data folder.

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 <u>not</u> 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. Press **PURGE GAS** key to purge system with He for 300 seconds; as done at startup of instrument.
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

Please seek staff assistance for any confusion or issues.