

PE 2400 II CHN/S Operating Instructions

Revised 05/29/2015 S.V.

Location: 1240 Hach Hall
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Safety

All researchers working in 1240 Hach Hall must complete the EH&S course: “*Fire Safety and Extinguisher Training*”. Be aware that the CHN/S analyzer operates at very high temperatures. When weighing samples, very small excess quantities (typically microgram quantity spillage onto the weighing paper) may be disposed of in the waste container next to the instrument. It is your responsibility to remove the unweighed remainder of your samples from the lab. Samples left behind will be disposed. Personal protective equipment (safety glasses and gloves) is available in the lab, or in 1238A Hach Hall.



NOTE: *The guidelines in this document require that you understand the reason for each action. Quite often it will be necessary to deviate from the example protocol given below. Unless you understand the purpose of each action you will not be able to modify the actions intelligently.*

The Measurement Principle

The instrument may be operated in either CHN or CHNS mode. Small quantities of sample (typically about 2 milligrams) are accurately weighed into small tin capsules. At elevated temperatures, in the presence of excess oxygen, organic materials combust to form CO₂, H₂O, various N_xO_y compounds, and SO₂ if sulfur is present. These reactions are facilitated by solid catalysts packed in the combustion tube; other combustion products like HCl are generally removed by silver gauze and other solid materials packed in the tube. The N_xO_y combustion products are reduced by fine copper in the reduction tube to N₂. The excess oxygen used to support combustion is also scrubbed out in the copper reduction tube. The only gases collected for quantitative analysis are:

- CO₂ representative of carbon content,
- H₂O representative of hydrogen content,
- N₂ representative of nitrogen content
- SO₂ representative of sulfur content (only with the CHNS configuration)

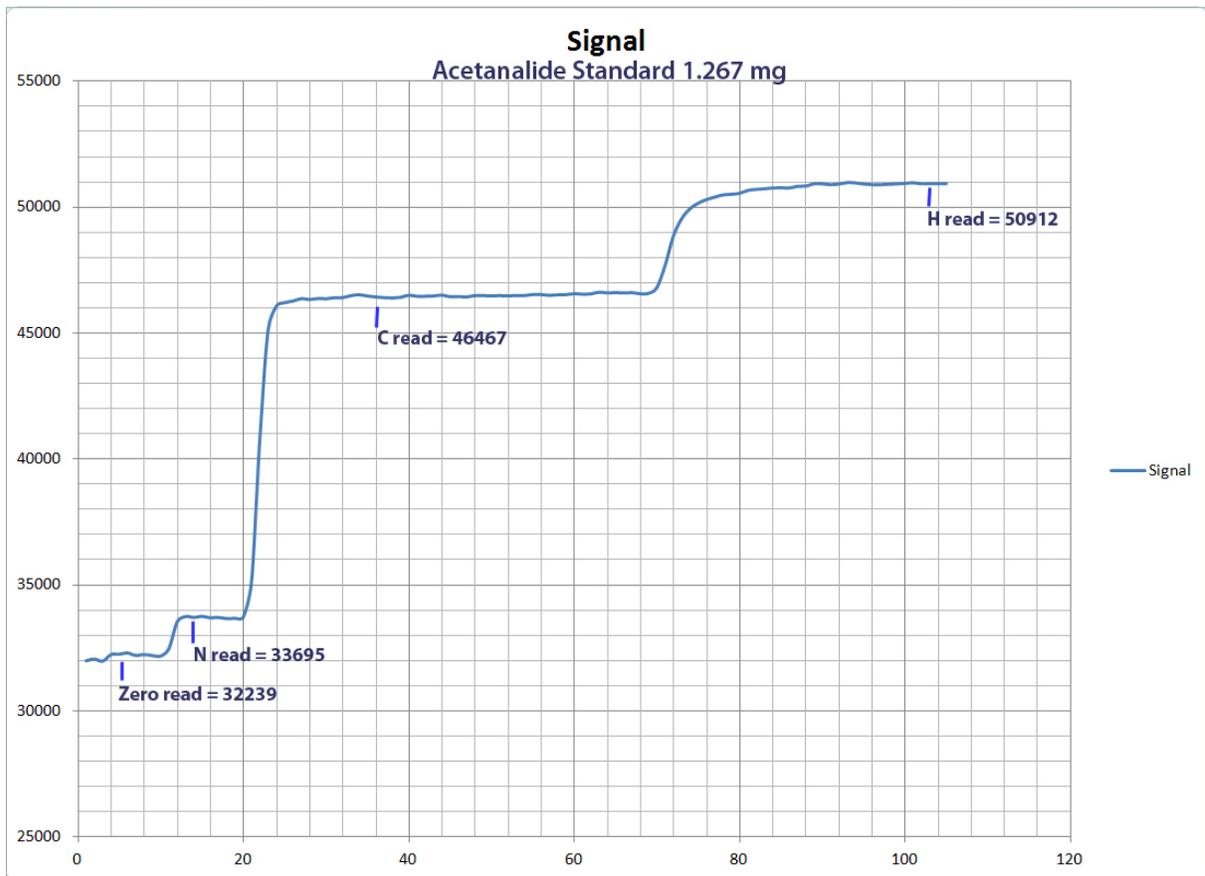
The catalysts in the combustion tube have a long life; however they can be poisoned over time. The effective lifetime of a combustion tube is about 750 runs. The protective quartz liner used in the combustion tube to collect the tin slag from the sample capsules should be cleaned or replaced every 100 runs. Because the copper in the reduction tube is converted to copper oxide as N_xO_y and O₂ are reduced, the reduction tube lifetime is finite. For CHN operation, there are separate combustion and reduction tubes. The reduction tube lifetime is about 120 runs. For CHNS operation, combustion and reduction functions occur in one tube. The CHNS combined-tube lifetime is therefore limited by the amount of copper in the tube; the lifetime is typically about 80 runs.

The combustion gases are collected automatically in a fixed-volume in the instrument, and then dosed over time onto a GC column designed specifically for the quantitative analysis of gases. The GC uses a thermal conductivity detector. Because the gases are dosed in over a period of time (as opposed to being instantly injected), discrete, well-separated peaks are not observed. Instead, "plateaus" are observed, and the steady-state values of each plateau are recorded by the instrument at pre-determined times. This is referred to as "frontal chromatography". The plateau values for each component are obtained by subtracting the value of the previous plateau. In the example shown below:

$$N_R = 33695 - 32239 = 1456$$

$$C_R = 46467 - 33695 = 12772$$

$$H_R = 50912 - 46467 = 4445$$



In order to convert the detector signals (baseline, nitrogen, carbon dioxide, water, sulfur dioxide) into %C, %H, %N, and %S, it is necessary to do the following. Each will be discussed in detail.

1. Establish steady state conditions in the instrument
2. Determine “blank” values
3. Run pure standards (known %C, %H, %N, %S) to establish “K-factors”
4. Run accurately weighed representative replicates of each sample.

1. Achieve steady-state conditions

Adsorption and desorption of gases on all glass and metal surfaces occurs continuously in the entire flow path of the instrument. These gases come from the combustion of samples, but also from impurities in the oxygen support gas and the helium carrier gas, and from small leaks at seals and valves that can introduce air into the system. Clearly, if the instrument has been unused for a period of time, desorption will exceed adsorption. The result of running a sample when the instrument is in a desorbed state will be arbitrarily low readings, since the combustion gases formed will first need to fill adsorption sites in the instrument before reaching the chromatograph detector. Similarly, the result of running a high-organic content sample after running a series of low-organic content samples (or blanks) could be low values. This is especially evident with

hydrogen content as the water adsorption fluctuates. To minimize these effects, the following should be done:

- Always use high-purity oxygen and high purity helium gases. Since the inherent impurity content is lower, the fluctuation from steady-state conditions will be less when blanks or low-organic content samples are run.
- Always run the leak diagnostic (diagnostics-2-1-2) when the columns have been changed, or you suspect there may be a leak in the system.
- Always condition the system by running several unweighed capsules of acetanilide (CHN) or cysteine (CHNS) at the beginning of the run sequence.
- Adjust your samples sizes based upon the expected organic content. Weigh more if the organic content is low. This is just one of several factors to consider when deciding the target weight of your samples.

2. Determine “blank” values

There will be a detector reading for zero, nitrogen, carbon, and hydrogen, even when an empty tin capsule is run. These are called the “blanks”, eg. Nbl, Cbl, Hbl. The instrument firmware keeps track of blank readings and uses these readings in a very specific manner, one that I do not totally agree with. When you run a blank, the immediate run values are given equal weight to the previous average blank values, and a new average blank value is calculated and used by the firmware calculations of %C, %H, %N, %S. To establish starting blank values independent of historical values, the firmware recognizes that when two blank runs occur in a row, the second one is to be considered correct and the values are not averaged with previous values; they are used as the new “average blank value” settings. Additional blanks need to be run throughout the run sequence so that trends in the blanks can be incorporated into the final recalculation of your results.

3. Establish “K-factors”

The equations that convert detector readings into %C, %H, %N, %S require a conversion factor for each element. This is established by running accurately weighed standards; normalizing the detector reading by weight; then dividing the normalized reading by the theoretical weight-percent of the C, H, N, S in the standard. These are called “K-factors”, i.e. K_{fC} , K_{fH} , K_{fN} , K_{fS} . Several duplicates of the standard are run at the beginning of the run sequence, spaced by blanks. Then additional duplicates of the standard (always preceded by a blank) are run throughout the run sequence. How often the standards are run depends upon several considerations, but generally a duplicate of standards should be run after the first 5-10 sample runs, then duplicates should be run after ever 10-15 sample runs. *NOTE: Although the firmware allows you to run these standards as K-factors, we ALWAYS acquire them as sample runs. It is then easier to use the results in the recalculation trend analysis.*

4a. Run representative sample replicates – sample composition

Properly prepared solid samples must be very dry, and pulverized into fine particles. Failure to do so may affect measurement precision due to incomplete combustion (insufficient surface area), or inhomogeneity of the sample. Without establishing measurement precision (repeatability) there can be no confidence in measurement accuracy, even when the instrument is well calibrated. The precision specification of the instrument is +/-0.4 weight-% for each element when at least triplicates are run. If your samples are properly prepared (dry, homogeneous mixture of fine particles, reasonable sample weight for the expected organic content) you might very well achieve measurement precisions better than this. A recent set of four ACE standards showed a measurement precision (one standard deviation) of 0.06 %C, 0.05 %H, and 0.15 %N.

4b. Run representative sample replicates – target sample weight

When establishing the target sample size to weigh, several factors apply. As you consider these, you will recognize that one target weight might not be optimal for all the information you are trying to get from your sample. Understanding these factors will allow you to make the best choices depending upon which element is most important to you.

- Is your sample 100% organic, with %C not exceeding 80%? Then your target weight should be 2.0 mg, +/- 0.5 mg.
- Is your sample 100% organic, with %C exceeding 80%? Your target weight should be much less, perhaps 1.5 mg +/-0.3 mg.
- Is your sample between 10-50% organic content? Your target weight should be 2.5 mg +/-0.5 mg.
- Is your sample less than 10% organic content? Your target weight should be 3.5 mg +/- 0.5 mg.
- Is your sample known to be difficult to combust? Your target weight should be 1.0 mg +/- 0.2 mg. Consider performing a combustion optimization run sequence.
- For CHNS, is sulfur greater than 5%? The previous suggestions all apply.
- For CHNS, is sulfur less than 5%, but not as important as the CHN results? The previous suggestions apply, but also run several “sulfur blanks”.
- For CHNS, is sulfur less than 5% and precise %S is most important? Multiply the weights suggested above by a factor of 1.5; be sure to run sulfur blanks; consider running low-sulfur content standards similar in nature to your compounds.
- Does your sample contain silicon? Adjust the target weights downward; be ready to accept less precise measurements; consider performing a combustion optimization run sequence.
- Does your sample contain halogens? The CHN combustion tube has chemicals to scrub out the halogens; performance depends upon the compound. The CHNS tube does not have compounds to scrub out halogens. In either case, use lower weights; be prepared for less precise results.
- In several of the above situations, running additional replicates (as many as four or five) might be necessary to achieve reasonable confidence in the results.

Acquiring Data

Instrument Preparation

The system should be checked for leaks whenever a tube is replaced; *diagnostic 2-1-2* is run. The system should be *purged* (He = 300 seconds; O₂ = 150 seconds) whenever a tube or gas cylinder is replaced, and just before a run sequence is initiated.

CHN Operation

The normal operating procedure includes running conditioners, blanks, calibrants, and standards throughout the run sequence. Acetanilide (ACE) is used for CHN studies. The default optimize-combustion settings for CHN measurements are 3,5,1,0. Check them before starting the run; often students change them to suit specific needs.

CHNS Operation

Cysteine (CYS) is used for CHNS studies. For CHNS measurement the optimize-combustion parameters are normally 1,1,1,0. A special sulfur conditioner (sulfonic acid - unweighed) is run prior to initiating the normal run sequence described above. You can run this as a *Single Run*, or as the first entry in the *Autorun* sequence. Cysteine (CYS) is used instead of acetanalide for all conditioning and K-factor adjustment runs.

Operational Procedure

Check gases

- He >200 psi in the tank; regulator set at 20 psi
- O₂ >200 psi in the tank; regulator set at 18 psi
- Air >80 psi

Verify Instrument Mode

PARAMETER 6 CHN mode or CHNS mode. If wrong mode for the runs you are planning, tubes must be changed.

Verify Run Capacity

PARAMETER 4 Run counters. All three must have values in excess of your expected total number of runs, including conditioners, blanks, calibrants, and samples. Because the run capacity is just an estimate, factor in a margin of safety. If more aggressive "optimize-combustion" parameters are used, the run capacity of the reduction tube decreases.

Check for Leaks (required after columns have been changed; otherwise optional)

DIAGNOSTIC 2-1-2. Resulting printout should "pass". If "failed", evaluate severity of failure. If pressure drops less than 20 mm Hg during the test, you may proceed if you wish as if the test had "passed". Report the small leak to me.

Optimize Combustion

PARAMETER 9 Optimize-combustion parameters. For CHN work, choose “constant”, unless you plan to burn some samples differently. Verify that the settings are 3,5,1,0 for CHN work. To use different values, choose “select” and set appropriate values in one of the four available programs. For CHNS, choose “constant” and verify that the values are 1,1,1,0.

Purge Gases

PURGE Purge helium for 300 seconds, and oxygen for 150 seconds.

Autorun Sequence

Move sample carousel clockwise to position #1.

AUTORUN If sample counter is not #1, choose *4RP -> 1 Reset -> Reset All Y*. This removes the existing data from the instrument memory. Be sure that the previous data has been fully processed before you do this.

AUTORUN #1,2,3 Choose 3S for each of these to run the unweighed ACE conditioners (CHN mode), or CYS conditioners (CHN/S mode).

AUTORUN #4,5 Choose 1B to run two blanks.

AUTORUN #6,7 Choose 3S to run weighed ACE (or CYS) calibrants.

AUTORUN #8 Choose 1B to run one blank

AUTORUN #9,10 Choose 3S to run weighed ACE (or CYS) calibrants.

AUTORUN #11 Choose 1B to run one blank

AUTORUN #12,13 Choose 3S to run weighed ACE (or CYS) calibrants.

AUTORUN #14->18 Choose 3S to run the first five samples

AUTORUN #19,20,21 Choose 1B,3S,3S to run a blank and two weighed ACE (CYS)

AUTORUN #22->31 Choose 3S to run the next ten samples

AUTORUN #32,33,34 1B,3S,3S to run a blank and two weighed ACE (CYS)

AUTORUN #35->44 Choose 3S to run the next ten samples

AUTORUN #xxx Finish by running a blank and two ACE (CYS)

In summary, a typical CHN run sequence would be:

Run #	Compound	Run as...	Purpose
1-3	ace (unweighed)	sample	condition the system
4-5	empty tin capsule	blank	set initial blank values
6-7	ace (1.5 - 2.5 mg)	sample	K-factor adjustment
8	empty tin capsule	blank	blank adjustment
9-10	ace (1.5 - 2.5 mg)	sample	K-factor adjustment
11	empty tin capsule	blank	blank adjustment
12-13	ace (1.5 - 2.5 mg)	sample	K-factor adjustment
14-18	sample runs 1-5	sample	unknown samples
19	empty tin capsule	blank	blank adjustment

20-21	ace (1.5 - 2.5 mg)	sample	K-factor adjustment
22-31	sample runs 6-15	sample	unknown samples
...continue samples in groups of 10

If this were a CHNS run sequence, #1 would be an unweighed sulfamic acid; runs 2,3 would be unweighed cysteine; cysteine would be used in all cases instead of acetanilide.

REPROCESSING DATA

The instrument prints out the results from each run as it is acquired. The data is also stored in the microprocessor memory, as long as it was acquired using the AUTORUN program. Data acquired as SINGLE RUN is printed, but not stored in memory. A typical printout of "blank" data will look like the results below. Note that since AVERAGE RESULTS are the same as the measured values, this must be the second of two consecutive blank runs. Recall that when blanks are run consecutively, the firmware assumes that you wish to assign the current blank measurement as the historical average.

```

DATE 01 05 15          TIME 12 57 08          OPERATOR ID SV
RUN 8  BLANK RUN

                                SIGNALS          AVERAGE RESULTS
                                ZR 32137
                                NR 32192          CARBON 9
                                CR 32183          HYDROGEN 153
                                HR 32366          NITROGEN 55

```

A typical printout of an ACE standard run as a sample will look like:

```

DATE 01 05 15          TIME 13 07 17          OPERATOR ID SV
RUN 10  ID ACE          WEIGHT 1.870

                                SIGNALS
                                ZR 32050
                                NR 33048
                                CR 51807
                                HR 56305

CARBON 71.46%
HYDROGEN 6.49%
NITROGEN 10.09%

BLANKS -9 153 55
KFACTORS 14.045 35.818 5.000

OXFILL  COMB  BOOST1  BOOST2
 3       5     1       0

FILL TIME 47 SECONDS

```

Using nitrogen as an example, the equation used by the firmware to calculate the %N is:

$$\begin{aligned}
 \%N &= ((NR-ZR-Nbl)*100) / (wt\ ugms*Kf_N) \\
 &= (33048-32050-55)*100 / (1870*5.000) \\
 &= 10.09
 \end{aligned}$$

Similarly the equation used for this same data to recalculate the Kf_N , knowing that the theoretical weight-% for acetanilide is 10.36, would be:

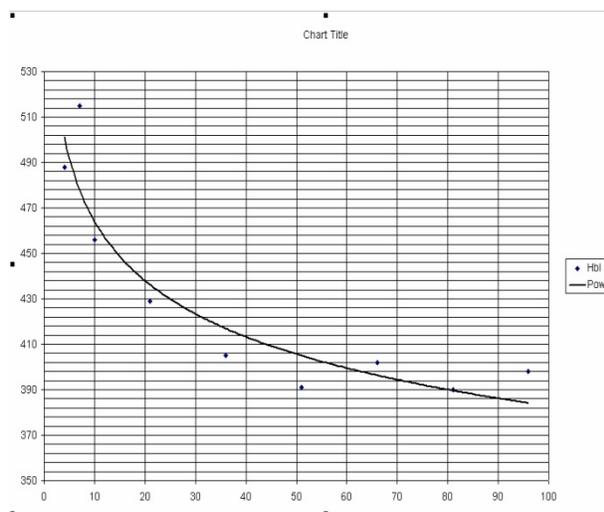
$$\begin{aligned}
 Kf_N &= ((NR-ZR-Nbl)*100) / (wt\ ugms*10.36) \\
 &= (33048-32050-55)*100 / (1870*10.36) \\
 &= 4.868
 \end{aligned}$$

Clearly, as long as we have a printed copy of the original measurements for each run in the run sequence, we can use Excel to recalculate results from scratch. However, it is laborious to type in all of the original weights and measurements into Excel. We normally adopt a hybrid approach which requires some work in Excel, followed by some use of the instrument firmware recalculation capabilities.

Hybrid recalculation approach

The measured blank values are transferred to Excel. When trends are not discernible, blank corrections are established by taking the average of all readings obtained throughout the run, or by allowing the system-calculated running average values to remain. Either method will usually suffice for the C, N, and S readings. Hydrogen blank values normally require adjustment based upon a curve. For example:

Run#	Cbl	Hbl	Nbl
3	33	707	59
4	14	488	55
7	24	515	47
10	32	456	63
21	18	429	52
36	24	405	48
51	15	391	49
66	24	402	48
81	44	390	49
96	25	398	46
avg	25.3	see chart	51.6
stdev	9.1		5.7



NOTE: The procedure to enter the reprocessing part of the firmware is a bit complicated, and is presented in the Appendix.

The corrected blank values are then entered into the "reprocessing" function of the analyzer firmware, and a blank-corrected printout is obtained for all runs.

From the blank-corrected printout, the measured values for all acetanilide runs (cysteine if it is a CHNS sequence) are transferred to Excel. The average %C, %H, %N, (and %S) values are obtained for the ACE (CYS) standards. Using simple ratio and proportion, corrected K-values are obtained.

Theory =	71.09	6.71	10.36
Run#	%C	%H	%N
5	71.61	6.49	10.42
6	71.51	6.57	10.34
8	71.66	6.07	10.27
9	71.61	6.31	10.28
11	71.63	6.47	10.53
22	71.54	6.02	10.36
23	71.52	6.36	10.38
37	71.51	5.92	10.38
38	71.75	6.28	10.38
52	71.47	5.58	10.29
53	71.59	5.85	10.32
67	71.55	5.55	10.34
68	71.63	6.58	10.38
82	71.4	5.77	10.38
83	71.66	6.2	10.42
97	71.3	5.2	10.36
98	71.4	6.37	10.41
average=	71.55	6.32	10.37
std dev=	0.11	0.23	0.06
oldK	14.190	30.110	5.050
newK	14.282	28.338	5.053

$$\begin{aligned} \text{newK}_C &= \text{oldK} / \text{Theory\%} \times \text{Average} \\ &= (14.190 / 71.09) \times 71.55 \\ &= 14.282 \end{aligned}$$

$$\begin{aligned} \text{newK}_H &= \text{oldK} / \text{Theory\%} \times \text{Average} \\ &= (30.110 / 6.71) \times 6.32 \\ &= 28.338 \end{aligned}$$

$$\begin{aligned} \text{newK}_N &= \text{oldK} / \text{Theory\%} \times \text{Average} \\ &= (5.050 / 10.36) \times 10.37 \\ &= 5.053 \end{aligned}$$

Note that measurements not used in the averages should be crossed out. In this example, there were no obvious outliers to exclude from the %C or %N averages, but each of the first of the duplicate %H measurements was low. This is a result of water desorbing from the system during the immediately preceding "blank" run. While somewhat troublesome, this is normal. The effect can be minimized by using very pure helium carrier gas so that the change from adsorbed water steady-state conditions is small, but it is still necessary to correct based upon a curve. The standard deviations for the ACE measurements are reasonable; well below the instrument specification of +/- 0.4%. The new K value to be used in the final reprocessing of the data is calculated (using K_{fC} as an example) from:

The corrected Kf values are then entered into the "reprocessing" function of the analyzer firmware, and a final printout using the best possible blank and K-factor values is obtained for all runs.

APPENDIX

Precision and Accuracy

The instrument specification is $\pm 0.3\%$ for each element as the standard deviation of four replicates. So, what does it mean if the precision of the measurements on your sample is not as good as the values for the standard? What does it mean if the accuracy of the measurements (based upon your expected formula) is not as good as the values for the standard?

First of all, accuracy can never be better than precision. Precision should ideally be established with at least four replicates, but most students just do two replicates. If and when you notice that two measurements differ by more than 0.8% (the standard deviation would then be 0.6%), you should run a third replicate if sample is available. If the values for the third replicate group well with the higher valued of the first two runs, you can exclude the low outlier. There are several reasons why we can rationalize an outlier being low, but we cannot rationalize an outlier being high. Some of the factors you can control that affect precision are sample homogeneity and particle size. A factor you may not be able to control is the inherent combustibility of your sample. If that is an issue, a full combustion study should be done to optimize combustion parameters, and possibly combustion additives like vanadium pentoxide, or thermolite. Rule out homogeneity and particle size first as the cause of poor precision.

If good measurement precision is achieved but the values are not as you expected, be aware that accuracy will depend upon factors such as the purity, dryness, and trapped solvent molecules in your sample. This last effect can be determined by running an NMR of your sample and quantifying the nature and percent of solvent in your sample. The measured %C, %H, %N values can then be corrected.

Combustion and Reduction Tubes

- Tubes are packed in-house using reagents purchased from Perkin Elmer or from other third-party vendors.
- Depleted reduction tubes are regenerated using a special furnace we have constructed; a 6% mixture of hydrogen in argon gas (Praxair Hydrostar H-6) is used.
- Quartz tubes are reused whenever possible; new tubes are purchased from Perkin Elmer, from other third-party vendors, or manufactured by our glassblower.
- Ultra-clean tin capsules (6mm x 4mm) are used.
- UHP Plus grade oxygen is used to support combustion
- UHP Plus grade helium is used as the carrier gas
- All standards are from Perkin Elmer or from other third-party vendors.

CHN/S Microprocessor

The instrument is controlled from the soft keys on the front panel. These interface with the embedded microprocessor and memory. Each soft key should beep when pressed. Note that there are no alpha characters. Alpha characters are entered by pressing the DOT key followed by a two-digit numeric code associated with each letter.

Generally, each program function is entered or exited by pressing the program key. In cases where the program has multiple levels, exiting may require pressing the function key multiple times to roll back through each program level. If you mistype something several levels deep in a program, first try the CE (clear entry) key; otherwise simply exit the program and start over.

The two-line LCD display may show current state, selection choices, results, or keypad entries.



The microprocessor programs include:

- **Diagnostics:** A suite of tests, including a leak test you must be familiar with.
- **Monitor:** Allows viewing of various transducers, including furnace temperature.
- **Purge Gas:** Both the Helium and Oxygen lines should be purged prior to a run sequence.
- **Parameters:** Allows viewing and selection of all instrument parameters.
- **Autorun:** Used to set up the run sequence and also reprocess data stored in memory
- **Single Run:** Not recommended; data is printed but not stored in memory
- **Start:** Used to start or continue execution of the Autorun sequence or Single Run.
- **Standby:** Not recommended; this key is more for emergency use.
- **<- and ->:** Move the LCD flashing cursor position left or right.
- **Yes and No:** Response to Y/N selection questions.

CHARACTER	KEYPAD ENTRY
(Space)	.00
A	.01
B	.02
C	.03
D	.04
E	.05
F	.06
G	.07
H	.08
I	.09
J	.10
K	.11
L	.12
M	.13
N	.14
O	.15
P	.16
Q	.17
R	.18
S	.19
T	.20
U	.21
V	.22
W	.23
X	.24
Y	.25
Z	.26

Alpha Chart

1. Blanks
2. K Factors
3. Theories
4. Run Counters
5. Operator ID
6. Operating Mode
7. Combustion Furnace Temperature
8. Reduction Furnace Temperature
9. Optimize Combustion
10. Lower Temperatures
11. Wake-up Option
12. Furnace On/Off
13. Blanks Criteria
14. K Factor Criteria
15. Sample Drop Criterion
16. Keyboard Beeper On/Off
17. Date
18. Time
19. Stabilization Criteria
20. Oxygen Valve On/Off
21. Reduction Valve I On/Off
22. Gas Saver Valve G On/Off

Parameters Menu (1)

23. Reset Parameters to Default Values
 24. Security Area
 25. Run Display Mode
 26. ROM Identification
 27. Print Code Lists
 28. Separation Timing Intervals
 29. Sample Run Significant Places S
 30. Automatic Signal Detection
 31. Linear Regression
 32. Filter
 33. Polymers
 34. Element Ratios
 35. Mixtures
 36. Simplest Empirical Formula
 37. Heating Value
 38. Solvent of Crystallization
 39. Results Calculated on a Dry Moist
 40. Print Blanks and K Factors and/o
 41. Print Fill Time
 42. Blank Selection
- DIAGNOSTICS: 2-1-2

Parameters Menu (2)



Normal instrument state when no programs are running.



42 different parameter settings are used to define the instrument configuration and elements of the run sequence and data processing.

Parameters you will likely review and/or set include:

- 4. Run Counters



Set to 120 for new CHN reduction tube; set to 90 for CHNS

-



Don't reset. Notify me when it drops below 100.

-



Set to 120 for CHN; set to 90 for CHNS

Used to set countdown warnings for the combustion/reduction tubes and vial receptacle.

- 6. Operating Mode.



Used to select between CHN and CHN/S operation. We do not have the OXY option.

- 9. Optimize Combustion.



1.->



2.->



Choosing "constant" prompts you to set static values for Oxyfill, Comb, OxyB1, and OxyB2 that will be used automatically in the *Autorun* program. Choosing "select" prompts you to establish four

different sets of values (Opt 1, 2, 3, 4) each with different Oxyfill, Comb, OxyB1, and OxyB2 settings; *Autorun* will then ask for a selection each time you add a run to the run sequence.

- 12. Furnace ON/OFF



Use to start furnace cool-down so that a column can be replaced.

- 42. Blanks Selection

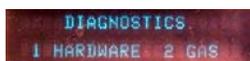


Normal setting is OFF. ON results in Autorun presenting a choice.

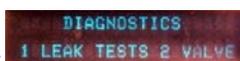
Normally *Autorun* establishes CHN/S blank values based upon running empty tins, but it is known empirically that the Sbl reading is slightly higher when a CHN standard, containing no sulfur, is run. For low-content sulfur samples, a more precise sulfur reading can be obtained by using a Sbl value derived from this special sulfur-blank method. Setting this parameter to ON results in *Autorun* presenting a selection choice when a blank run is requested.



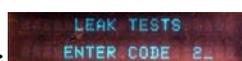
A range of instrument diagnostics can be selected from a multi-layer menu. Students should only run "2-1-2", the combustion leak program.



2.->



1.->



2.->

Runs comb. Tube leak test.

Note that when the 2-1-2 diagnostic starts, the tube should pressurize to about 760 torr within 15-30 seconds, and then should stay relatively constant at that value, dropping no more than 5-10 torr over the duration of the test. The diagnostic will then report Pass or Fail. If the test fails catastrophically, you must consult with me and you should not begin your measurements. NOTE: The furnace must be completely warmed up and stable at the operating temperature before you begin the diagnostic.



When cooling or heating the furnace, the temperature can be monitored. Choose NO to the Print List question, then select Monitor Code 1.



NO ->



Both the helium and the oxygen lines should be purged before a run sequence is started. Typically 300 seconds and 150 seconds are used.



YES ->



YES ->



All runs should be programmed as part of an *Autorun* sequence. Of the three acquisition types, blanks, K-factor and sample runs, we only use

(1)Blanks, and (3) Samples. This has been discussed previously. The 4RP selection is used to reprocess data stored in memory after it has been acquired, and will be discussed in the next section.

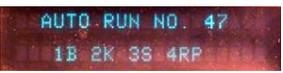
1. Blanks  Offers a choice only if Parameter 42 is set to “ON”.

 Offers a choice if Parameter 9 is set to “SELECT”.

3. Samples  Use the numeric keypad to enter the coded alpha characters and/or the numeric characters of the sample ID. Pressing ENTER prompts you to enter the weight. Note that the weight can then be transferred directly from the balance by pressing ENTER, or it can be typed in, followed by pressing ENTER. If Parameter 9 is set to SELECT, you will be presented with the choice of optimize combustion programs 1-4.

NOTE: Be aware that after the final ENTER is pressed, the entry is added to the queue and the system assumes there is now a sample (or blank) physically in the assigned position in the autosampler carousel. If an Autorun is in progress, this sample (or blank) will be run without further operator intervention.

Reprocessing Data

When you enter Autorun , selecting RP allows you to reset and begin a new run sequence, print sample information, print sample results, or recalculate the data. 

(1) RESET ->  is used only when you are setting up a new run sequence to begin at position/sample 1. This will erase all of the results from memory, so be sure that data has already been reprocessed. The system will ask if you wish to RESET ALL runs; select YES.

(2) PRINT INF This is used to print a summary list of all the runs entered in the run sequence. You must provide this list to me at the end of your session so that proper billing can occur.

(3) PR RESLTS This choice can be used to reprint the entire sample information, analysis conditions, and results for each entry in the run sequence. It will duplicate the report printed as each run finishes during the analysis.

(4) RECALC To recalculate data, first enter the AUTORUN program. The display will show the next position in the queue available to be programmed:  In this example it is

position 47. To access earlier data in the queue, use the left arrow key  to move the flashing cursor so it is under the “4” in 47. Then press the CE (clear entry) key , and type in the first run number you

wish to reprocess, followed by “ENTER”. In this example, it is run number 37 .



Press the Parameters key ! You will be presented with the RP display . Select

(4) RECALC. You will be presented with the recalculation choices dialog box . You can recalculate any single run based upon a different weight. This is helpful if you notice a transcription error in the weight recorded on your sample sheet (or in the logbook) and the weight you actually entered. You can also recalculate a series of runs based upon new blank values or new K-factor values.

As discussed earlier, when reprocessing a series of data, we first correct the C, N and S blanks based upon the average of all of the blanks acquired during the run sequence. The H blank is taken from the plotted curve of Hbl vs run# and applied sequentially to smaller groups of runs. Select (1) BLANKS. You will be presented sequentially with the current C, H, N, and S blank values, and given the opportunity to enter new values for each one.



In each case, the value is changed by moving the cursor that is flashing until it is under one of the digits, pressing CE, and typing in the new value. You will then be asked how many runs the new blank values should be applied to, beginning from (in this example) run 37. The recalculated results will print.

After reprocessing the run sequence with more correct blank values, the data needs to be reprocessed again with more correct K-factors. The protocol for establishing new K-factors was discussed previously. After entering the calculation program at the first run to be reprocessed, select (2) KFACTORS. You will be presented sequentially with the current C, H, N, and S K-factors, and in each case given the opportunity to enter new values.



The value is changed by pressing CE, and typing in a new value. You will then be asked how many runs the new K-factor values should be applied to, beginning from (in this example) run 37. The recalculated results will print.