Exeter CE440 Element Analyzer – Research Instrumentation Center

September, 2023, by Na Gou

Instrument Overview

Element Analyzer used thermal conductivity detection for measuring carbon, hydrogen, and nitrogen, after combustion and reduction. The instrument detects C, H, and N, all during the same analysis, from the same sample aliquot.

To detect these elements, the sample needs to be taken into the combustion tube and pass over suitable reagents to assure complete oxidation and removal of undesirable by-product such as sulfur, phosphorous, and halogen gas. Then in the reduction tube, oxides of nitrogen are converted to molecular nitrogen and residual oxygen is removed. In the mixing volume the sample gasses are thoroughly homogenized at a precise volume, temperature and pressure. This mixture is released through the sample volume into the thermal conductivity detector.

Between the first of three pairs of thermal conductivity cells an absorption trap removes water from the sample gas. The differential signal read before and after the trap reflects the water concentration and, therefore, the amount of hydrogen in the original sample. A similar measurement is made of the signal output of a second pair of thermal conductivity cells, between which a trap removes carbon dioxide, thus determining the carbon content. The remaining gas now consists only of helium and nitrogen. This gas passes through a thermal conductivity cell and the output signal is compared to a reference cell through which pure helium flows. This gives the nitrogen concentration.

The instrument need reconfiguration for O or S, we do not do so currently.

Appropriate Sample Types & Sample Preparation

- Solid samples need to be able to weight about 1-2mg.
- Semi-solids, liquids and air/extremely moisture-sensitive samples are considered ‘non-routine’ and are designated ‘drop-in’s’ as they are run immediately after weighing and are ‘dropped into’ the instrument.
- Duplicate runs of solid samples are the standard procedure, as long as enough sample can be recovered.
- Triplicate runs of liquid/semi-liquid samples are the standard procedure in case of lack of precision between only two runs.

Consumables

- Tin Capsule, smooth wall, 6x2.9mm, #6703-0418
- Nickle Sleeve, use with flat crimped Tin capsule, 7x5mm. #6703-0499

Weight sample with microbalance (optional)

- Solid samples need to be weight with tin capsules on microbalance.
- Make sure calibrate the microbalance with 2mg standard.
- Zero the microbalance with an empty tin capsule on the control side.
- When weighting, make sure door is closed and support is down.

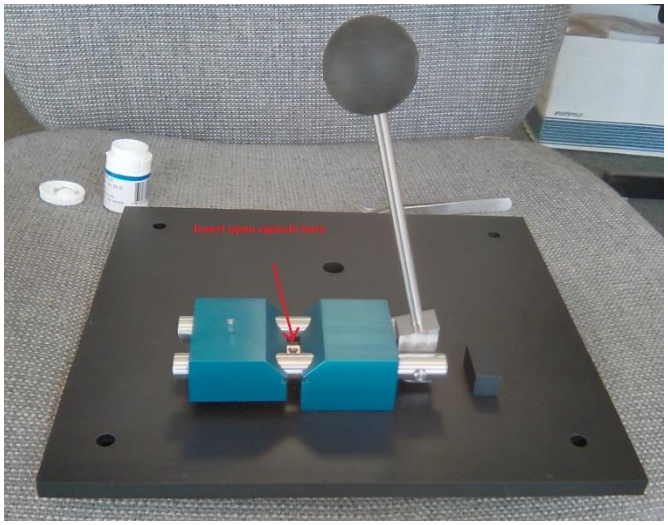
1. Turn on the main power switch at the back of the microbalance if it is not on, leave it on for at least 1 hours.
2. Make sure door is closed, turn the knob to lower the support then press “autotare” to zero the balance, waiting for 0 reading.
3. To calibrate the balance,
    a. make sure the support is up, and open the door, open the drawer at the bottom of the balance,
    b. use a tweezer pick the 2mg weight put on the left side, close the door, and lower the support,
    c. press “range” to select the “2”
    d. press “2”, then press “calib”
    e. wait for stable reading of 2mg
4. To weight sample
    a. make sure the support is up, and open the door, put 1 tin capsule on both sides, close door, lower support, press “autotare” to zero balance
    b. raise support, open door, and take the tin capsule on left side and fill with sample, put it back to left side, close door and lower the support
    c. wait for stable reading, that’s your weight. Better to be within the range of 1-2mg for element analyzer samplers.

Capsule sealer operation (optional)

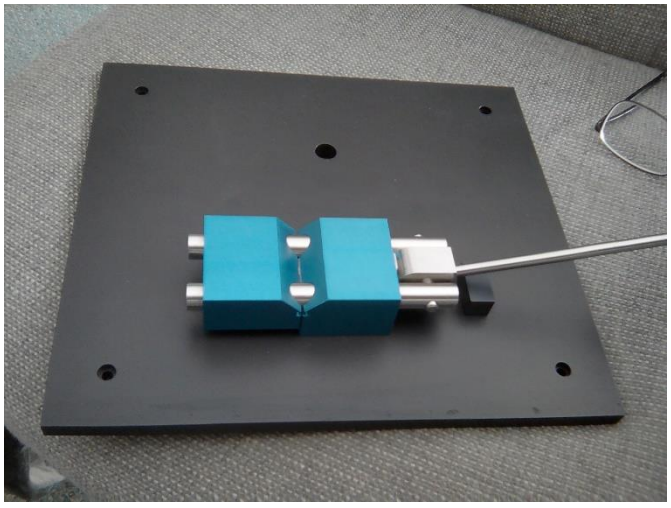
- Volatile liquid capsules require use of the "capsule sealer" device to ensure integrity of your sample.

Volatile samples will evaporate between weighing and analysis. Simply pinching the capsule with tweezers will not seal the sample. The capsule sealer is designed to seal tin capsules (6 x 2.9 mm), part # 6703-0418.

1. Here is a picture of the sealer. the capsule is inserted where indicated by the red arrow.

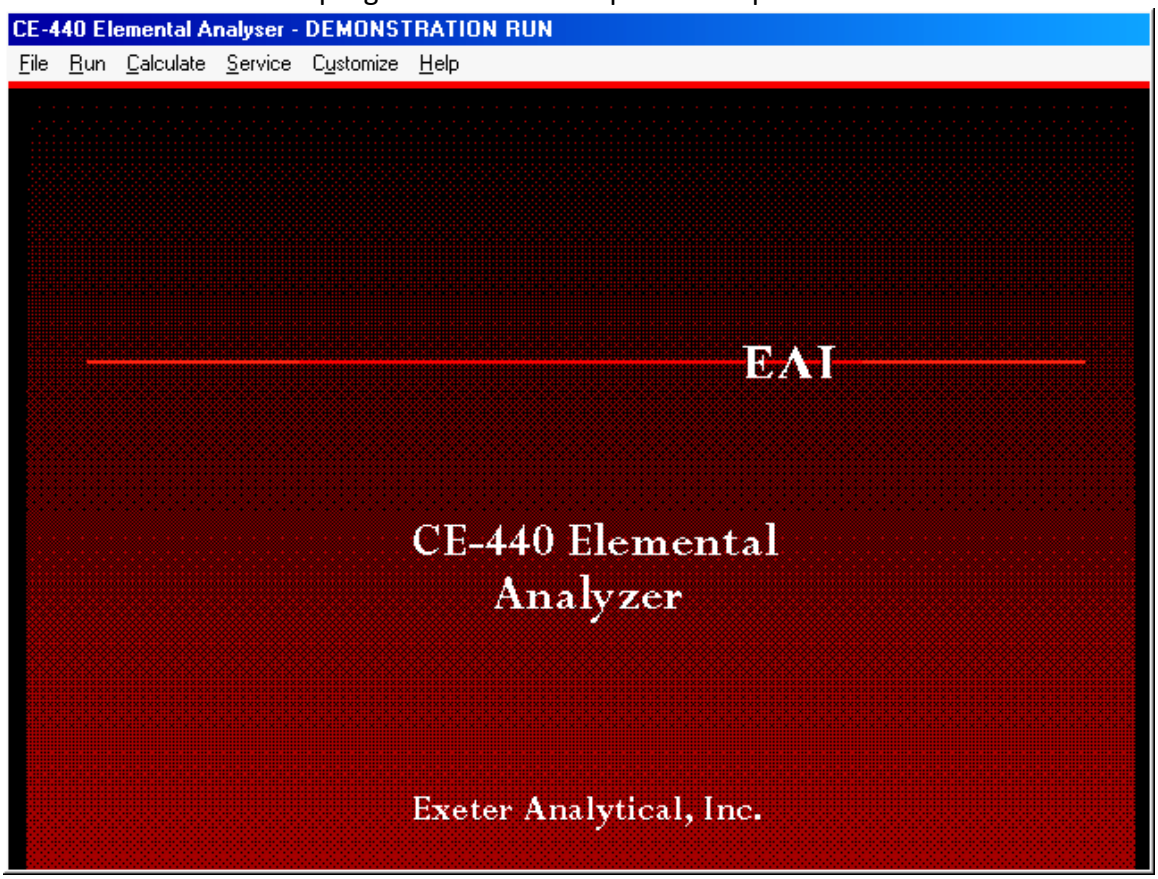


2. The lever is pressed firmly downwards and then pulled back upwards.



The 440 software program

You will view the CE440 program main menu upon start up



To access the menu, use the mouse to click one of the six menu at the top of the screen.
There is a username and password for the software
Username: exeter
Password: pass2005

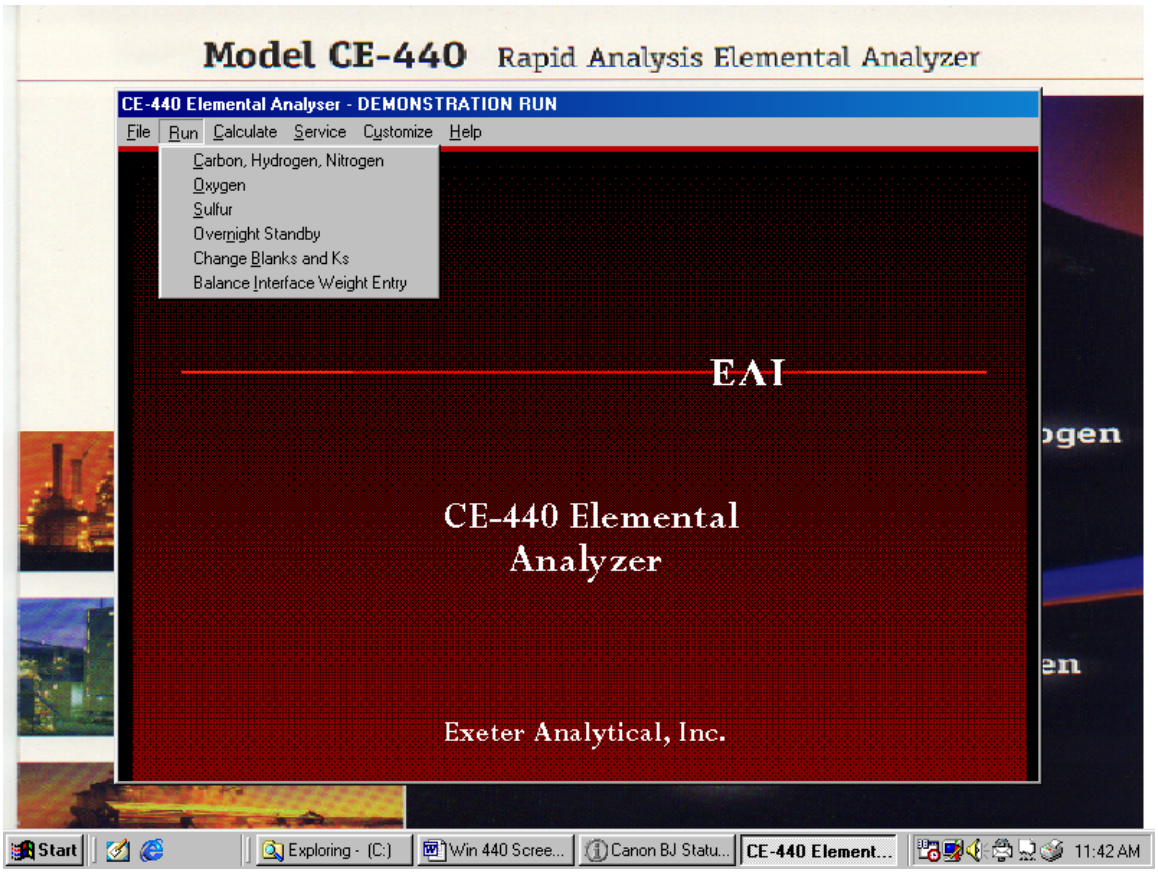
Starting the instrument

1. Check He and O2 pressure, helium ~ 16 psig and oxygen ~ 25 psig
2. Turn off overnight standby option via the menu options.
   "run"->"turn off overnight standby"

CHN analysis

FIRST MAKE SURE THAT THE B VALVE IS ON (Go to customizing and run parameters). This is the Oxygen valve.

This is the most commonly selected mode and is used for total Carbon, Hydrogen, and Nitrogen (and Oxygen by difference) determination of organic and combustible inorganic compounds. Select Carbon, Hydrogen, Nitrogen to proceed through the following sequence:

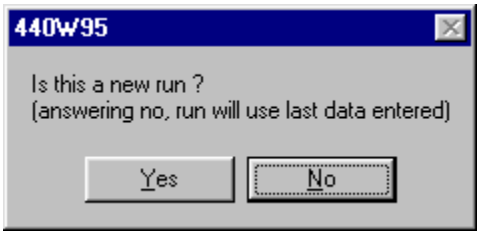


General Operating prompts

1. Use weight file for weight entry (Yes or No)?

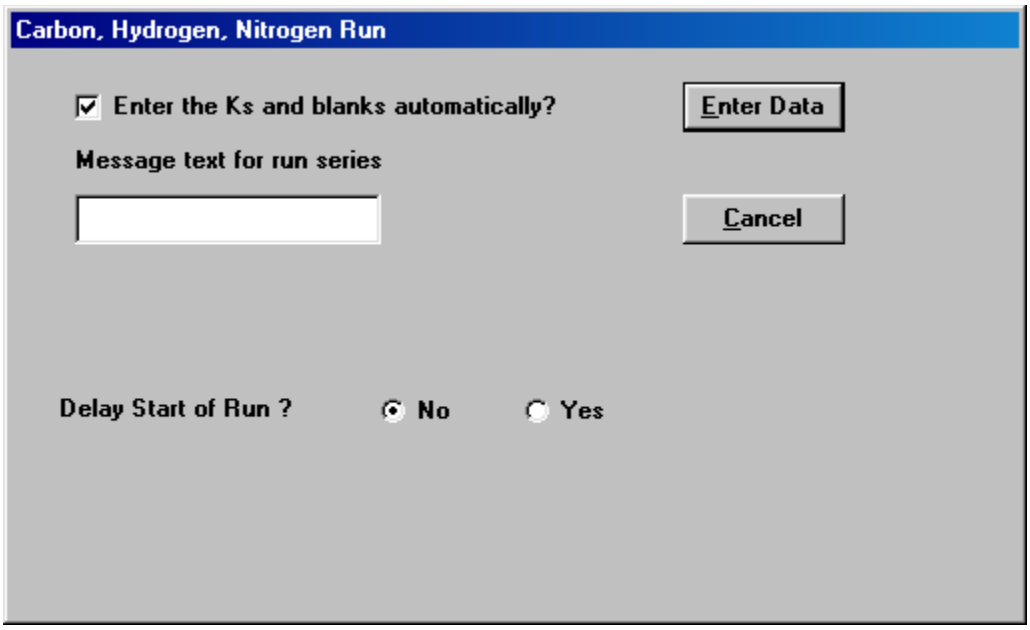
This prompt will only occur if there are weights stored in the weight file on disk. The weight file is automatically filled if the balance interface is used. Click [Yes] for automatic sequential use of the weights that are on the weight file.

2. Use existing run data from last run series (Yes or No)?



This prompt occurs only if an HA run series is aborted prematurely. Clicking [Yes] will allow the user the opportunity to use the sample information (weight and ID) still in memory. [No] allows the entry of new data.

3. Entering message text for this run series



Enter a useful message for the run series that is about to be run. Usually the operator name and sample type are entered here.

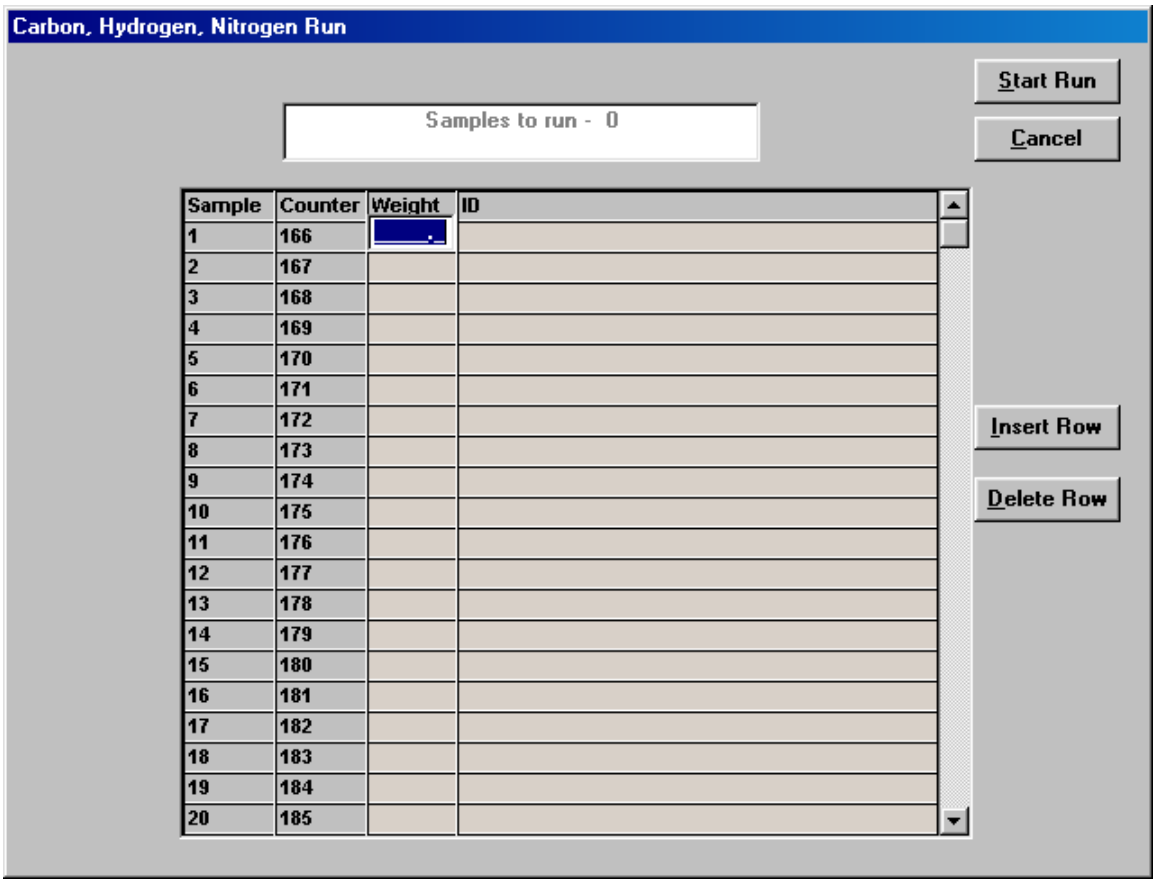
You may also select whether you require a delayed run. Enter the desired start time and date.

Checking “Enter Ks and Blanks entered automatically” will cause the software to automatically use Ks and Blanks found from calibration and blanks run on the analyzed data. Else, the system uses default data.

4. Weight and ID Entry

Fill in the Weight and ID entry table. You may delete or insert rows. “Sample” is the run number for this series of analysis. “Counter” is the corresponding infinite counter number which tracks all runs analyzed on the instrument (until reset).

When entering the weight of the sample, press TAB to use the present weight (if editing the table) or enter a new weight.



Sample ID's

- blank: If a weight of zero is entered then the ID is assumed to be a BLANK.
- std#: To calibrate, enter the sample ID as either STD1, STD2, STD3, STD4, STD5. If STD is entered as the first three letters then Ks will be calculated on the result report (calibration run). We use acetanilide as STD1. The following are commonly used standards and are pre-programmed into the CE-440 software. The system recognizes then as STD1, STD2 etc.

std	C%	H%	N%	O%	S%
1=acetanilide	71.09	6.71	10.36	11.84	---
2=benzoic acid	68.84	4.95	---	26.20	---
3=cyclohexanone	51.79	5.07	20.14	23.00	---
4=cysteine	29.99	5.03	11.66	26.63	26.68
5=chlorodinitrobenzene	35.58	1.49	13.83	31.60	---

- cond: Before running any sample or blank, it is necessary to run one or more conditioners. The purpose of conditioner runs is to saturate the walls of the system surfaces, especially the mixing and sample volumes with water vapor,

carbon dioxide and nitrogen which simulates actual sample running conditions. To simulate this condition as closely as possible, is advisable to use conditioners of approximately the same weight as in the samples to be run.

- Use any text as the sample ID for a sample and CHN results will be printed in percent by weight. Weight is in the unit of micrograms.

Sample Routine

1. Empty blank (nothing)
2. Empty blank (nothing)
3. Conditioner
4. Conditioner
5. Capsule blank (with capsule in sleeve)
6. Conditioner
7. Std#
8. Std#
9. Std#
10. Positive control with known standard
11. Positive control with known standard
12. Unkown sample

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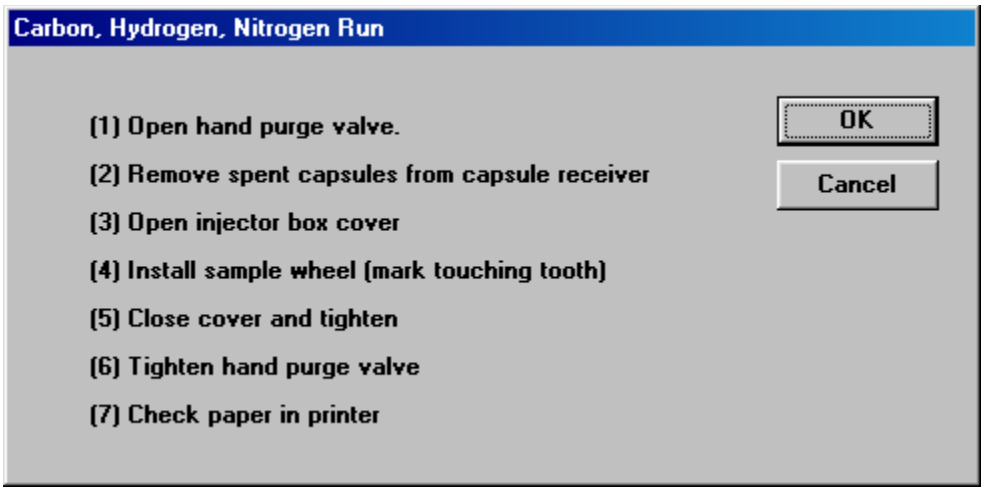
#: capsule blank (last run)

*Start the run early enough in the day so that the first standard result can be checked before leaving. If it is within acceptable limits let the run continue. If not, recalibrate, either by inserting standards via the SSI port or by restarting the run cycle.

5. Run prompt:

The following prompt will then appear if you have an HA system.



Please follow the instructions. You should insert the samples into the system at this point.

Loading the sample wheel with HA-64 sample automation

This mode opens the ADF and C valves allowing helium to enter the injection box and minimize air in the area while you install the sample wheel for the 64 sample automatic injector. It is recommended that the injector box be closed at all times, unless you are inserted the sample wheel.

Note: The injector housing should not be opened while pressurized. Vent the housing with the manual purge valve prior to opening the lid.

*make sure clean the capsule receiver before install sample wheel

To proceed with installation of the sample wheel:

1. Open the manual purge valve on the injector box (right side, behind the P valve) to relieve the internal pressure.

2. Loosen the 4 cover thumb screws and lift the lid. Remove the empty wheel if necessary.

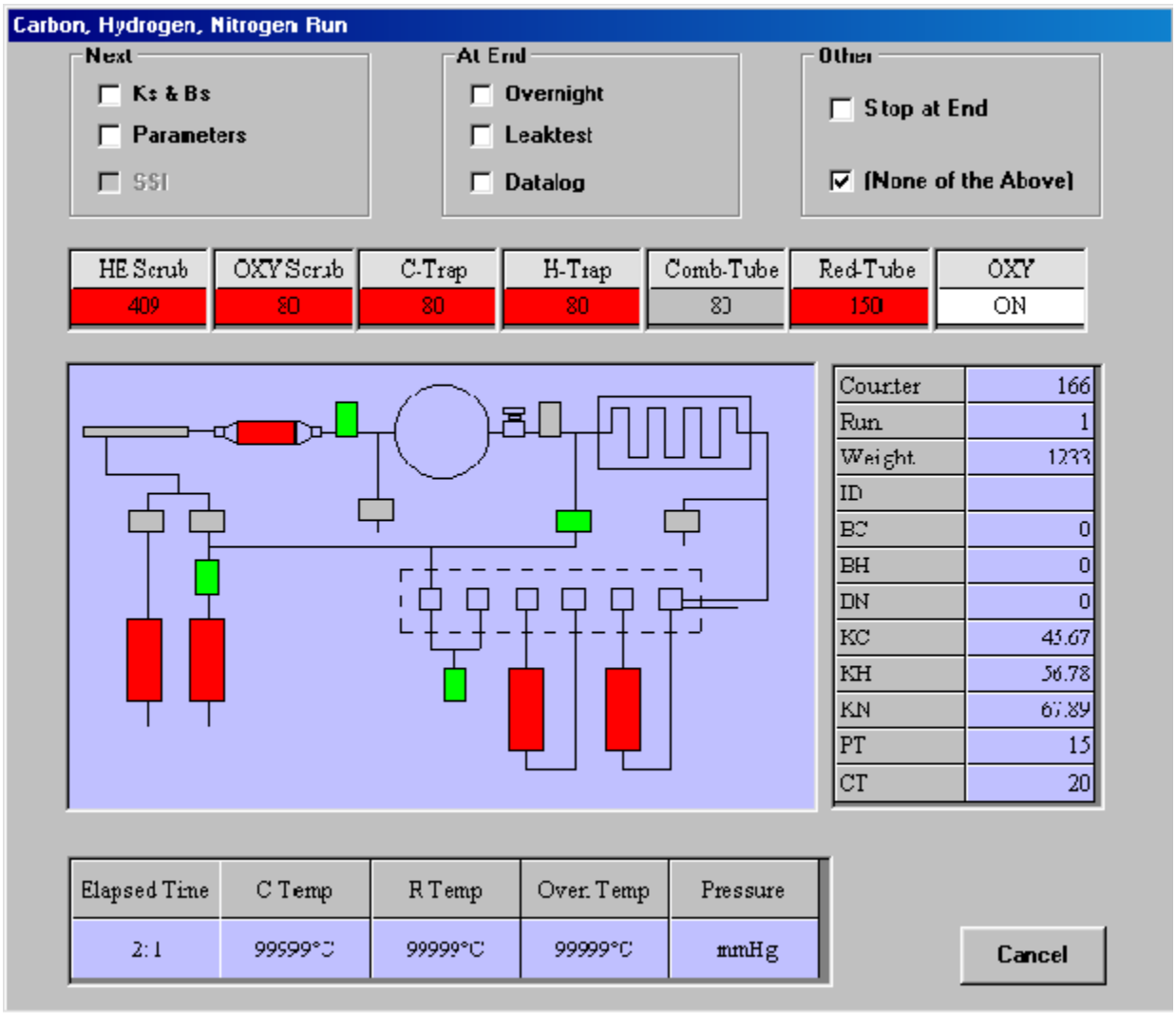
3. Insert the loaded sample wheel with the locking pin in place. Tilt the wheel slightly, line up the scribe mark on the wheel with the ratchet in the housing, lower the wheel and make sure that it is properly seated. Place the locking pin in the center hole. Check that the O-ring of the cover is clean and well seated in the groove.

4. Close the cover, and tighten equally on all four thumb screws. This should be performed in an alternating sequence to achieve a uniform seal. Never over tighten or use any tools on the thumb screws.

5. Open and remove any spent capsules in the capsule receiver. Re-grease gasket and re-install the cover.

Now you are done and may proceed. Click OK.

6. Analysis will now commence and the run time graphic will then appear (please see below for detailed explanation of the run time graphic).



Once the run begins, the screen displays the following information:

The center of the display graphically illustrates the state of the CE-440 throughout the analysis of the sample. Green rectangles indicate “open” valves. White rectangles indicate “closed”

valves. The traps, scrubbers, reduction and combustion tubes will turn red when the number of runs exceeds the number allowed in the Maintenance table.

The infinite Counter, Run Number, Sample Weight and I.D., the operating K and B values, the preset Combustion and Purge times as shown on the right.

Elapsed time in minutes and seconds, combustion, reduction and oven temperatures are displayed on the bottom along with system pressure.

Run counters for the various tubes are displayed above the valve status diagram. The run counters will change from white to red when they approach 10% of the thresholds set by the user. A display also indicates whether your Oxygen valve is ON or OFF.

During the run the operator has various options available through the check off entries at the top of the page:

NEXT (after the current run, you can):

Ks & Bs - To access the Ks & Bs table at the end of the current run. This allows the operator to change the operating values.

PARAMETERS - Goes to parameters table at the end of the current run. You can modify these at this point.

SSI – To perform an SSI drop after the current run. The HA program will automatically resume after the SSI run (unless SSI is clicked again).

AT END (after all runs in the series are completed, that is, one run in the manual, SAI or SSI modes, or one wheel in the HA mode)

OVERNITE - The instrument will go into overnight standby at the end of the run cycle.

LEAKTEST - The leak test program is activated at the end of the run cycle.

DATALOG - A datalog is printed every half hour at the end of the run cycle. A, D, and F valves are turned on, as in the overnight standby mode.

OTHER

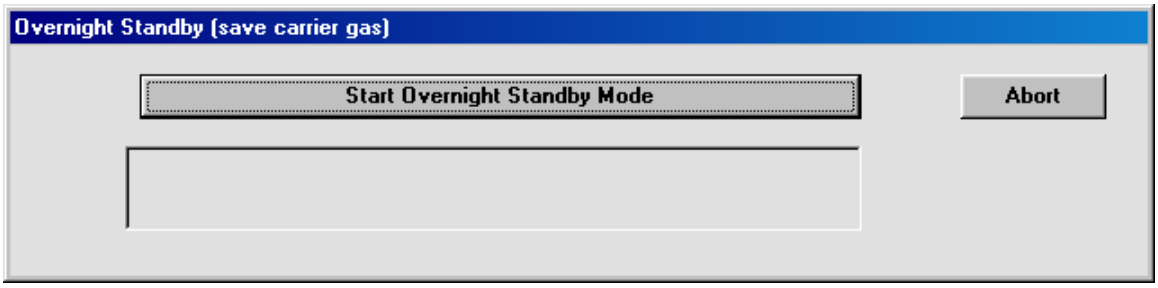
STOP AT END – The system will halt analysis of this series of samples and go to the main menu.

NONE OF THE ABOVE - Default. When no action is required.

A comprehensive report will be printed once a run has been completed (or shown on the screen if the printer option has been turned off). At this time any automatic reagent diagnostic warnings can be printed or displayed and the maintenance schedule will be updated as well as the K's and B's default values for the next run cycle. All data is transferred to the data file (440.DAT) at the end of each run.

Finish up

After collect your data, the instrument need to be put to standby mode to conserve gas and pressurize the detector during overnight or long term standby of the instrument, click ”run”->”overnight standby”, The following display will appear.

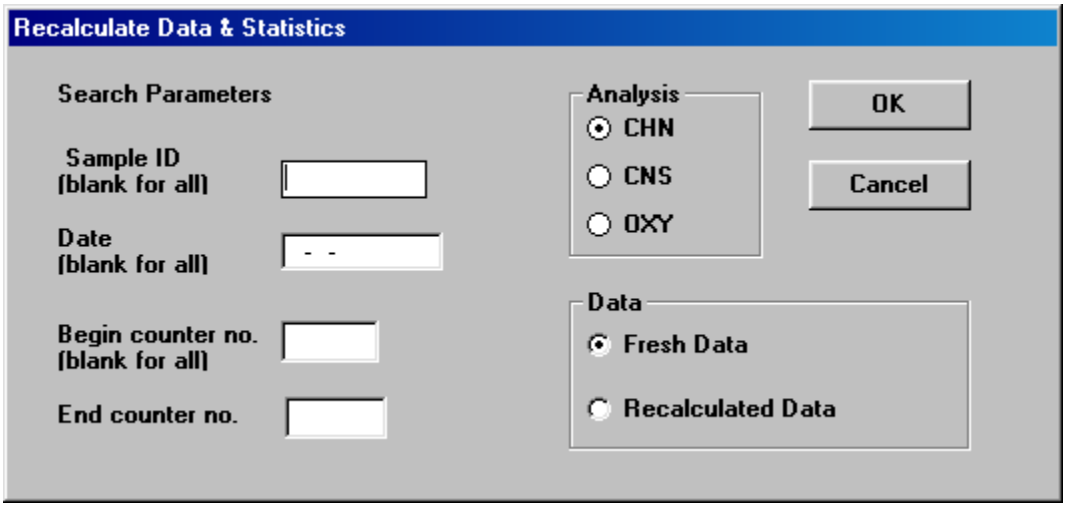


After-analysis recalculation

This calculation is useful for adjusting the values of the data due to explained or unexpected changes in the blank or calibration (K) factor during an analytical run cycle. Blanks can change due to sample handling changes, different capsule, sleeve, or combustion aid types, or a small but precise leak, or due to contamination. K-factors should remain stable but can drift due to flow changes caused by variable pressure drops in the traps or helium scrubber, or by changing delivery pressure at the helium regulator, or by operator change of the combustion time.

It may also be necessary to modify blank and/or K factors due to the failure of the software to automatically update them when required or requested. This would happen if the difference between the new blanks and/or news K’s and those in memory is too great.

You will be presented with the following search menu after selecting the Recalculate Mode by click “calculate”->”recalculate data & statistics”



* Sample ID:

Enter the specific sample ID that is to be searched for. If all of the samples and blanks run during a time period and with a specific ID are to be used, leave blank.

* Date (i.e.-06-03-2003)

Enter the date of the sample runs to be recalculated or blank to signify all dates.

* Begin Counter #

Enter the infinite run counter number of the first run that is to be recalculated. If you wish to search the entire data file, leave blank.

* End Counter #

Enter the infinite run counter number of the last run that is to be recalculated. If searching the entire data file, this entry will be ignored.

* Type of Analysis (CHN, CNS OXY)

Click the type of analysis that was performed on the samples. CHN is the default.

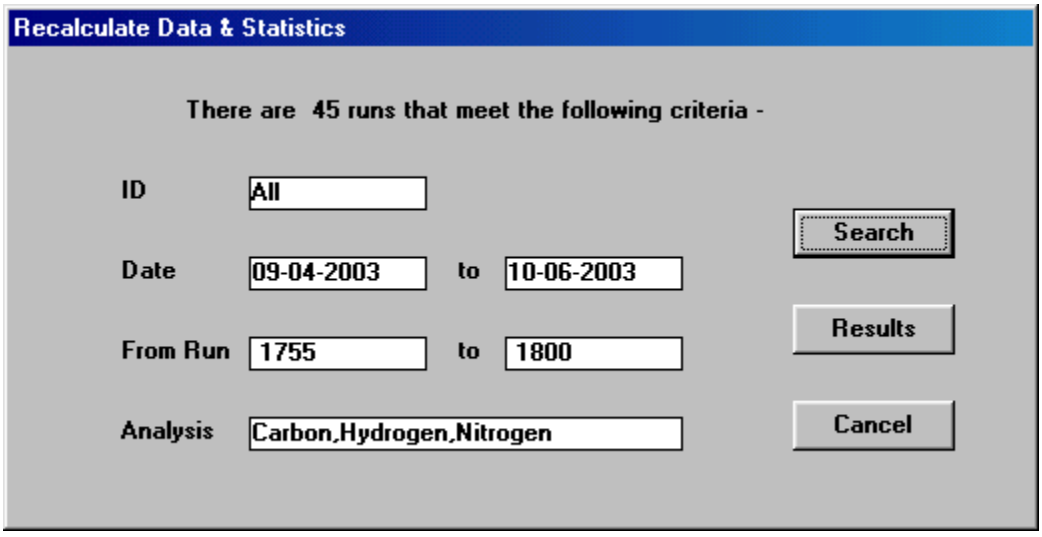
* Data

Click whether to search "fresh" data or data that has already been recalculated. Please note that the "440.dat" always contains the original data as found when the sample was run. "Recalulated" data is stored in another data file [C:\440w95\dat\440.tmp] if you request that the data be stored.

When done selecting search criteria, click "OK". Click "Cancel" to back out immediately.

The computer will now search your data file (440.DAT). This may take a few minutes if you have a large data file with the results of several thousand samples.

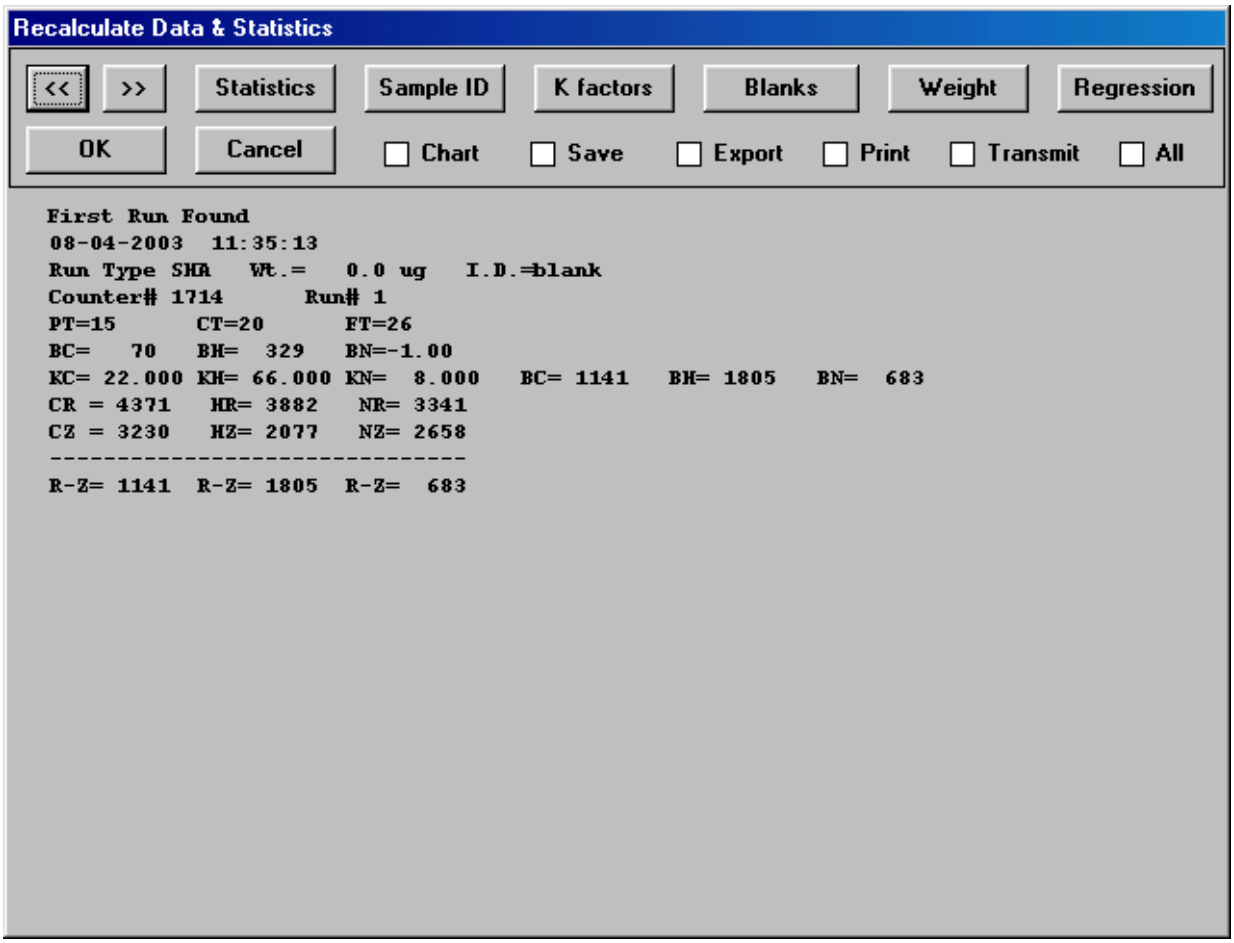
Upon completion of the search, results are displayed as follows:



Click "Search" to launch a new search.

Click "Cancel" to exit.

Click "Results" to view the results of your search. A typical "Results" display looks as follows:



The data may be manipulated once displayed. You will see the results of the first run found during the search. You can use the “>>” and “<<” buttons to navigate through the requested results.

* Check off boxes

Check “Chart” to chart the results.

Check “Save” and then click OK to save the selected results to a file.

Check “Export” and then click OK to save the selected results as a comma delimited TXT file.

Check “Print” and then click OK to print out the selected results at any time.

Check “Transmit” and then click OK to transmit the data over the user configured serial port.

Check “ALL” if you wish to apply selected actions to all the data found in the search. Else, the actions will be applied to only the displayed event. For example, print would print all the results found.

The other buttons offer the following:

* Statistics: This selection will display the selected data results in a significantly truncated format. The report can be saved or printed.

* Sample ID: Allows you to modify the Sample ID.

* K Factors: Allows you to modify the K factors or gains of the resultant search data. You may enter new values for any of the K's and decide which runs the new data is applied to.

* Blanks: Allows you to modify the Blanks of the resultant search data. You may enter new values for any of the K's and decide which runs the new data is applied to.

* Weights: Allows you to modify the Blanks of the resultant search data. After entering a new weight for the sample that is presently displayed on the screen, the run will be recalculated and redisplayed.

* Regression: This allows you to automatically perform linear regression on the Ks & blanks. The program will linearize the data between blanks and standards brought into the search buffer.

NOTES ON AUTOMATIC ENTRY OF K'S AND BLANKS

Any time BLANK (i.e., no entry) is used for a sample I.D. the values from that run are entered as the operating blank values. Any time a STD# is entered as sample I.D. the computer calculates and enters a new set of operating Ks based on a weighted formula using the last three sets of Ks in memory. This occurs only if all three Ks fall within the following windows:

New KC = KC in memory +/- 1.0

New KH = KH in memory +/- 2.0

New KN = KN in memory +/- 0.5

Thus it is important that the Ks in memory are close to expected values or new Ks generated will not be within the window and therefore will not be accepted for automatic insertion. The weighted formula is:

K = (K1) + (0.5 * K2) + (0.25 * K3) / 1.75

where K1 = K found this run

K2 = Next K in memory

K3 = Last K in memory

If this is the first K found, then K1=K2=K3=K then for each K (found) thereafter the stack is pushed up 1.

The blanks will only be accepted if all three blanks fall within the following:

New BC < 500

New BH < 3000

New BN < 250

NOTE: If incorrect insertion occurs, the data can easily be corrected by using the "Recalculate Data" mode in the calculation Menu.

Loading Sample with Manual SSI

The following prompt will then appear if you select a SSI injection:

Remove the Entrance Plug before clicking CONTINUE. After clicking continue, a high flow of Helium will prevent air from entering the combustion tube. DO NOT touch the ladle as this will contaminate it. Use gloves. Take out the spent capsule and residue from the previous run and load the pre-weighed sample. Put the ladle back into the combustion tube by slowly swinging the magnet down until it "grabs" the ladle. Do not insert the ladle into the furnace at this time. Replace the entrance plug and click continue to start the run.

Analysis will now commence and the run time graphic will then appear.