The Model CE-440 Elemental Analyzer has been verified as an accurate technique for the determination of oxygen in organic compounds. The method involves a conversion, where the combustion and reduction tubes used for CHN analysis are replaced with pyrolysis and oxidation tubes, respectively.

During an analysis the sample is pyrolyzed at 1020-1060^{o}C. The oxygen pyrolysis products are converted to carbon monoxide in the presence of platinized carbon. Interfering by-products are scrubbed by the hot copper and the acid gas scrubber U-tube. The carbon monoxide then flows into the oxidation tube, where it is converted to carbon dioxide over copper oxide at 670^{o}C and eventually absorbed in the CO_2 trap.

To further improve performance and accuracy, Exeter Analytical has made several refinements to the technique.

The conversion from oxygen to carbon monoxide depends upon the catalyst to carbon ratio on the surface of the packing. To increase this ratio, as well as to alter the flow characteristics, a second platinum plug is now placed in the center of the platinized carbon area of the pyrolysis tube. Also, to prolong the life of the water trap, magnesium perchlorate has been added after the ascarite in the U-tube.

An oxygen scrubber (P/N 307-00039 has been added to the helium inlet line, to remove trace oxygen from the carrier stream. This stabilizes the capsule blank values to 250 + / - 10 microvolts, lending very good accuracy to low level oxygen determinations.

It is believed that trace oxygen in the helium combined with carbon in the sample or catalyst, forming carbon monoxide that was ultimately measured as oxygen. This erratic oxygen signal produced inconsistent blank values and poor precision on replicate samples. With the addition of the oxygen scrubber, capsule blanks are approximately half their historical value, and reproducibility is increased by a factor of three.

Blank values can still vary due to the pyrolytic by-products of the sample. The reaction taking place in the pyrolyis tube is: $CO_2 + C = C(O) + CO$, where C(O) denotes carbon - oxygen complexes that contain more carbon than oxygen atoms, (i.e., $C_xO_y$where x>>y). The pyrolytic by-products can decompose the C(O) products to CO and cause a high result. The quartz in the system may also yield high blanks by reacting with the carbon: $SiO_2 + C = CO + Si$). However, these actions are significant only at low levels. This is why a hydrocarbon blank should be used when the samples have a very low oxygen content.

This phenomenon of "hydrocarbon blank" products by samples composed only of carbon and hydrogen is still present, though the value above that of the capsule blank reading has been found to be weight-independent. This allows for easy correction on low-oxygen or no-oxygen samples. The hydrocarbon blank value for 1,000 micrograms of anthracene (0% oxygen) is the same as for 2,500 micrograms. This value has been found to be in the range of previously acceptable (<500 microvolts) capsule blank values.

The amount of conditioning required for a new pyrolysis tube and oxidation tube is minimal. Approximately five to ten runs should fully condition the tubes. Half of these runs should be with a high carbon content material, such as anthracene or paraffin oil, and the second half should be with a compound containing oxygen, such as acetanilide. One practice to avoid is the use of hydrocarbon conditioners with weights greater than 4,000 micrograms. Excessive sample weights have a tendency to contaminate the platinized carbon catalyst, resulting in a doubling of the oxygen "K" value to approximately 33. This is corrected by replacing the catalyst.

Once the packings have been conditioned, several blanks are run until the system is stable and the blanks agree to within + / - 10 microvolts. Acetanilide is recommended as a calibration standard, and the suggested start-up procedure is: conditioner, conditioner, blank, conditioner, standard, standard. The blank value that is entered into memory is the conditioned blank and the K values are calculated from the two standard runs. A good rule of thumb is to expect the oxygen K value to be 75% of the carbon K value when in CHN mode. However, if empty capsule blanks are used, the K value can be slightly higher. A good test to determine how well the system is running would be to run a sample of benzoic acid. If the results are low, a problem in pyrolysis is indicated. Raising the temperature should help bring the results into range.

The operating conditions found to give optimum results are as follows:

Pyrolysis Temp:	1020^{o}C
Oxidation Temp:	670^{o}C
Purge Time:	120 seconds
Combustion Time:	60 seconds
Fill Time:	180 seconds
Sample Capsules:	Silver

Aluminum capsules can be used for economic reasons, however, they tend to oxidize which causes low results. Tin capsules cannot be used.

This technique of oxygen analysis produces very good results for most compounds, with a few restrictions. Samples containing metals, phosphorus, silicon or boron usually yield low recoveries, due to the formation of refractory oxide complexes. High levels of halogens, especially fluorine, poison and catalyst, although samples containing up to 20% F can be run successfully.

As an additional feature, Exeter Analytical has developed an Oxygen Hot Changeover Kit (P/N 125-00011) to speed conversion from CHN to Oxygen mode and vice versa. Reduction of furnace temperature is avoided by the use of a helium purge transfer line to prevent combustion of the platinized carbon during changeover.