CE-440 THEORY OF OPERATION

In the 440 Analyzer the carbon, hydrogen, and nitrogen content in organic and inorganic compounds can be determined. Combustion of the weighed sample (typically 1-3 mg; in certain cases up to 500 mg) occurs in pure oxygen under static conditions. With a conversion kit oxygen and sulfur can be analyzed. A kit for steel and refractories is also available.

The combustion train and analytical system are shown in Figure 1. Helium is used to carry the combustion products through the analytical system to atmosphere, as well as for purging the instrument. Helium was selected for this purpose because it is chemically inert relative to tube packing chemicals, and it has a very high coefficient of thermal conductivity. Argon can be used with some 440 modifications, but is not recommended since instrument performance deteriorates. Solenoid valves A through G control the gas flow through the system; valves H and I are used for automatic leak testing and other maintenance purposes.

The products of combustion are passed over suitable reagents in the combustion tube to assure complete oxidation and removal of undesirable by-products such as sulfur, phosphorous, and halogen gases. 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.

For oxygen analysis, the combustion tube is replaced by a pyrolysis tube containing platinized carbon. The reduction tube is replaced by an oxidation tube containing copper oxide. The sample is handled and run as before, but is now pyrolyzed in helium so that carbon monoxide is formed from oxygen in the sample. The CO is oxidized by the copper oxide to form carbon dioxide, which is detected and measured in the same manner as the carbon analysis.

For sulfur analysis, the combustion tube is replaced with one containing a tungstic oxide packing plus a dehydration reagent. The water trap is removed and replaced with silver oxide

to absorb SO.). The sample is handled and run as before, but the sulfur from the sample is oxidized to form SO2, and the water formed is removed. The SO2 is detected and measured in the same manner as the hydrogen analysis.

STANDARD RUN CYCLE (CHN)

The PC Compatible Computer controls the valve sequencing of the instrument as shown in the diagram on the front panel of the 440. To assist in maintenance and for trouble shooting it is helpful to understand the run cycle as described below.

At the start of each run the entire system is flushed with helium at a high flow rate while the sample is in the cool zone. With the HA automatic multi-sample injector, the injection box is automatically purged through the P valve. The combustion train is then filled with oxygen and the sample is injected. Shortly after sample injection, D valve closes to seal off the combustion train from the rest of the analytical system, which is still being flushed with helium. Combustion occurs under static conditions in an excess of oxygen at about 975 degrees Celsius.   During this time the mixing volume is being purged with E and F valves open. Then F closes to allow the pressure in the mixing volume to reach atmospheric pressure.

Near the end of the combustion period, a high-heat coil around the combustion tube vaporizes any condensates at the entrance of the combustion tube. These condensates may have been produced by diffusion of the sample during the initial stages of combustion. To further assure that complete combustion takes place, the ladle is retracted approx. 1 inch, a small amount of oxygen is added and the ladle is again fully injected. In addition, an extended combustion time can be selected with extra bursts of oxygen injected every 30 seconds. During high-heat, valve E closes, A and D reopen, and the combustion products are completely flushed from the combustion train into the mixing volume. When a pressure of 1500 mmHg (about 14.5 psig) has been reached, as monitored by the pressure transducer, valve D closes trapping the sample gas in the mixing volume. The time required to reach this pressure is called fill time and is recorded for diagnostic purposes. A fill time of 20-50 seconds is normal. The combustion train remains under positive pressure until the end of the complete cycle.

While the sample gases are mixing, pure helium flows from valve C through the sample volume and through the detectors. The signal from each detector bridge is read and stored in memory to provide a "zero" reading (base line) with no sample gas in the detector.

When the mixing is completed F and G valves open to allow the sample gas captured in the mixing   volume to expand through the sample volume to atmosphere. During this time valve C is closed and there is low flow through the detector.

When the sample gases are near atmospheric pressure, valves F and G close and C opens. The water, carbon dioxide, and nitrogen concentrations of the sample are measured by displacing

the sample gas through the detectors to atmosphere. The volume of sample gas in the system is large enough so that the helium flow used allows measurement of the contents of each detector in sequence, under steady state conditions. The sample gas passes through the detectors at a constant flow, pressure, and temperature to eliminate any variations in water vapor pressure or water vapor concentration due to changes in water adsorption on the walls of the pneumatic system.

While the sample gas is displaced through the detectors, the output signals are recorded. The difference in microvolts between each "read" signal and the "zero" level recorded earlier for the same detector is in direct proportion to the concentration of the sample gas measured.

At the end of the cycle, the exhaust valves are opened to allow the sample gases to escape to the atmosphere. The Computer then prints out the calculated results, puts the instrument into standby with C valve open, and awaits the next command. With the HA automatic injector the results are printed after completion of each run.

CE-440 FLOW SCHEMATIC