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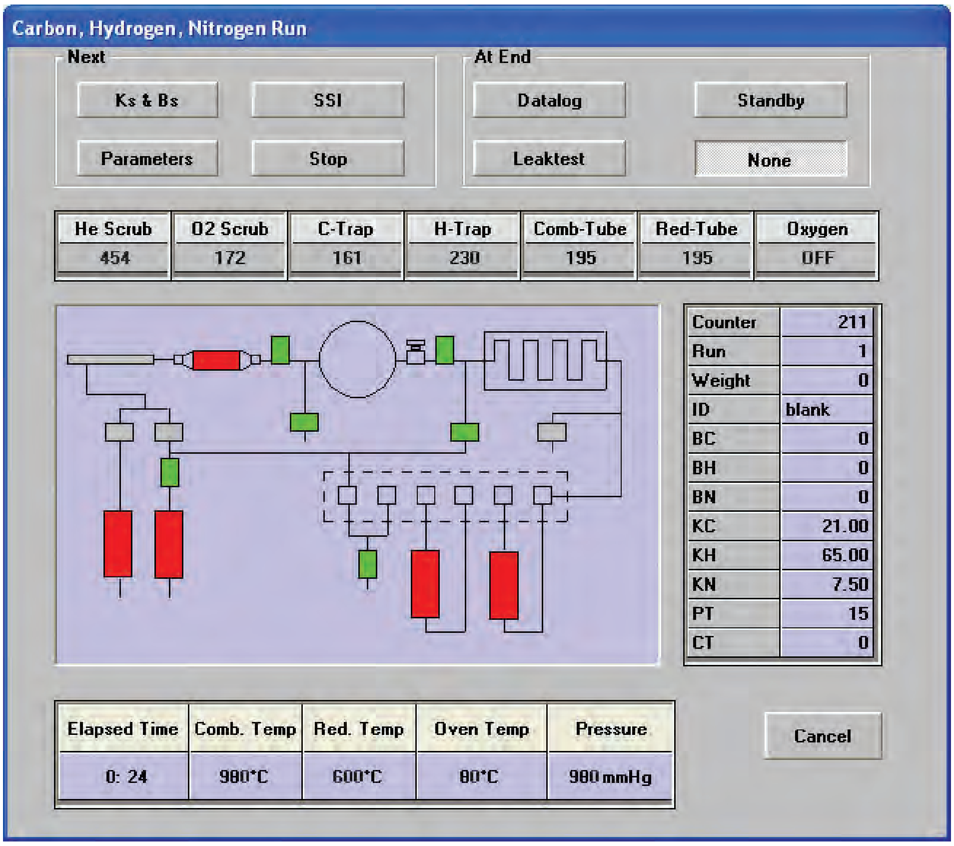
EXETER ANALYTICAL

incorporating WARWICK ANALYTICAL SERVICE

Application Note 232

Theory of Operations

In the 440 Analyser, 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 below in the CE440 flow diagram. 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 co-efficient of thermal conductivity.



Argon can be used with some CE440 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 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 monoxide, 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 SO2. 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.

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