



Application Note 215

Enhanced Combustion for Elemental Analysis of Refractory and Steel Compounds on the CE440

Because of their physical properties, refractory compounds, such as carbides and nitrides, are finding ever increasing applications in the field of new technology. In one such field, advanced ceramics, the knowledge of the carbon and nitrogen content is of primary importance during the manufacture of materials for specific applications.

Difficulties in analyzing these compounds arise in their decomposition. This is particularly true for various nitrides and carbides which can have melting points in excess of 2000°C. Exeter Analytical has made significant advances in accurately analyzing refractory materials on the Exeter Analytical CE440 Elemental Analyzer, which employs a pure oxygen static combustion to fully combust samples. Similar instrumentation using limited oxygen with dynamic flow cannot insure complete combustion. A key element to successful analysis is the use of a special flux to completely break down the samples.

For a flux to be successful it must have three main functions. First, it must generate high temperatures to decompose the sample. Secondly, it must provide a catalytic effect in attacking carbon or nitrogen bonds, and thirdly, it must donate oxygen to fully oxidize the combustion by-products. One such flux, Hi-Temp, offered commercially by Exeter Analytical, performs all of these functions and tested better than Perkin-Elmer's Thermolite on the most difficult to combust samples. (see Table 1)

NBS Refractory SiC Standard Product Ref. #112b	Sample Theory	29.43	N/A	No Flux	20.11	34.76
		21.05	36.60	37.35		
Perkin-Elmer	29.32	39.36	RC			
Thermolite	29.67	39.11				
Exeter Analytical	29.37	40.85				
Hi Temp Flux	29.60	40.60	40.64			

Table 1: Comparison of % Carbon Results. REF: 215 REV. 1 PAGE 2

One problem arising from the thermal action of the flux is that it can devitrify both the ladle and the combustion tube quickly. To prevent this, the sample is placed in a protective alumina sleeve (P/N 0240-0810A). This sleeve is then placed on the modified sample ladle (P/N 0240-2061A) and the analysis is carried out using enhanced instrument conditions, which are summarized below:

Combustion Time: 185 sec. Combustion Temp: 1010°C Combustion Aid: Approx. 10 mg. Hi Temp Flux (Pre-dried at 100°C for 1 hour.) Sample Size: 1 to 1.5 mg. Sample Containment: Tin Capsule/ Nickel Sleeve/Ceramic Sleeve.

The increased combustion time allows for additional bursts of oxygen to reach the sample. The packing of the combustion tube is also altered under these conditions to optimize the accuracy and precision of the data. This is described in detail in the refractories kit instruction manual.

The sleeves, ladles, and flux along with standards and a refractories manual are all provided in the Exeter Analytical Refractories Kit (P/N 125-00018). The kit can also be used to analyze for carbon in steel, coke on catalysts, some organometallics and other composite materials which require rigorous conditions to extract the carbon and nitrogen.

The logo for Exeter Analytical, consisting of the letters 'EAI' in a large, bold, serif font. A horizontal red line passes through the middle of the letters.

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