



## Application Note 217

### CHN Analysis of High Concentration Fluorine Samples on the CE440

Most compounds containing fluorine can be run successfully on the Exeter Analytical CE440 under standard operating conditions. The magnesium oxide in the combustion tube reagent packings absorbs fluorine as HF, and releases the hydrogen. However, some difficult to combust, perfluorinated samples can give incorrect carbon and nitrogen data. This is believed to be due to the strength of the C-F bond. If not fully oxidized, CF<sub>2</sub>, passes through the combustion tube packings and is detected as nitrogen, yielding both low carbon and high nitrogen data.

For these high fluorine concentration samples, the method is modified as follows. The analysis is run at combustion temperatures greater than 1000°C, in tin capsules, to aid combustion. In addition, 10mg. of red lead oxide, Pb<sub>3</sub>O<sub>4</sub>, is added to the capsule before initial taring. After the sample is weighed in and the capsule is sealed, it is shaken to ensure good contact between the sample the Pb<sub>3</sub>O<sub>4</sub>. The analysis then proceeds as normal.

The Pb<sub>3</sub>O<sub>4</sub> acts as both an oxygen donor and as a fluorine scrubber. It should be noted that using Pb<sub>3</sub>O<sub>4</sub> may increase the carbon and hydrogen blanks. Therefore, the instrument must be calibrated with the lead oxide prior to running samples.

Using these modifications, we have been able to obtain the theoretical values of zero percent nitrogen and 22.10% carbon on a sample of perfluoro-N-octanoic acid. This compares to biased nitrogen values in the 1.5% range when analyzed under normal operating conditions. This modified method will help assure the operator of obtaining quality data on difficult to combust, highly fluorinated compounds.