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.

There are two main schools of thought concerning reasons for problems with highly fluorinated compounds.

1) The first is thought to be due to the strength of the C-F bond. If perfluorinated compounds are not fully oxidized, residual CF2 passes through the combustion tube packings and is detected along with nitrogen yielding both low carbon and high nitrogen data.

2) The second theory proposes that fluorine reacts with the silica from the combustion tube and forms Sift4, which is volatile and is detected along with nitrogen causing similar problems to above.

For these high fluorine concentration samples, the method is modified as follows. The analysis is run at combustion temperatures greater than 1000^{o}C, in tin capsules, to aid combustion. In addition, 10 mg. of red lead oxide, Pb_3O_4, is added to the capsule before initial tarring. After the sample is weighed in and the capsule is sealed, it is shaken to ensure good contact between the sample the Pb_3O_4. The analysis then proceeds as normal.

The Pb_3O_4 acts as both an oxygen donor and as a fluorine scrubber. It should be noted that using Pb_3O_4 might increase the carbon and hydrogen blanks. Therefore, the instrument must be calibrated with the lead oxide prior to running samples i.e. blanks should be established.

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.