

## Application of GFAAS to Petrochemical Samples: Optimizing Ashing Temperatures

### “Easy” Samples:

Petrochemical samples can be very diverse. Most fuel and oil samples when dissolved in the proper solvent have very little background interference. Thus successful ashing can be achieved at low temperatures in a minimal amount of time. In such cases a variety of elements including relatively volatile species like arsenic, lead and cadmium can be easily determined.

### “Difficult” Samples:

For samples containing low volatility organic constituents and/or particulate matter dilution is necessary. Whenever this situation is encountered furnace conditions should be carefully optimized to obtain the maximum analyte signal and minimum background signal. Background signal should be decreased by an increase in the ashing temperature. When the ashing temperature is too high, however, analyte may be lost during ashing thus decreasing the analyte signal. The maximum ashing temperature has to be determined empirically, it can vary extensively depending on the form of the element and matrix.

Different types of samples have characteristic background absorbance profiles depending on the wavelength at which the background is determined and the nature of the background. Figure 1 shows the background profile vs ashing temperature for 30  $\mu$ l of a 1g engine oil sample dissolved in 100 ml of diisobutyketone (DIBK) and the maximum ashing temperature for each element determined in the dissolved sample.

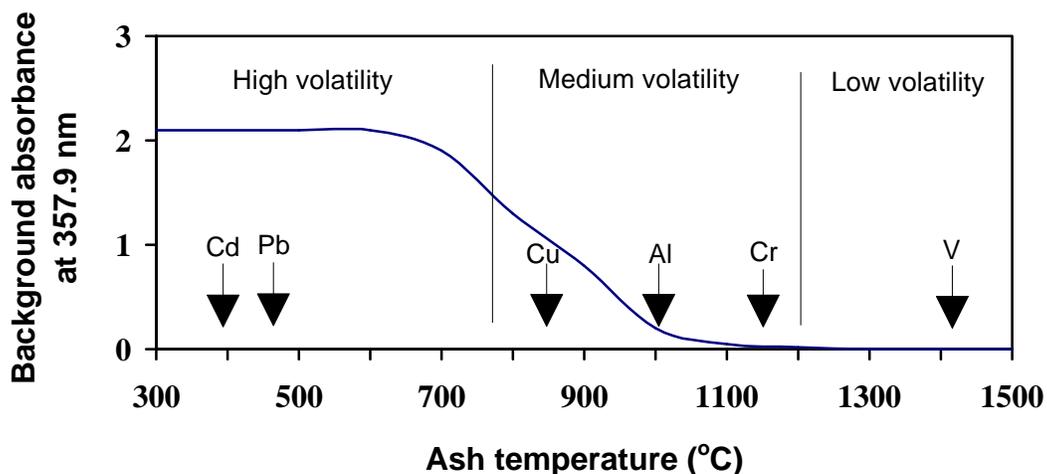


Figure 1. Background signal profile for used engine oil dissolved in DIBK

***Elements with high volatility:***

For highly volatile elements such as arsenic, lead and cadmium, ashing temperatures should be set below 500°C to prevent analyte loss. As a result of this low ashing temperature much of the sample matrix remains. Therefore, high background signal levels can be observed. There are three available choices for reducing this background signal:

- (a) use of an acid digestion method for sample preparation;
- (b) use of chemical modifiers such as diammonium hydrogen phosphate and palladium nitrate to allow higher ashing temperatures without analyte loss;
- (c) use of a separation method such as liquid-liquid extraction to transfer the analyte from the organic phase into an aqueous phase.

All of these methods permit the use of aqueous standard solutions for calibration.

***Elements with medium volatility:***

In region B of Figure 1, copper, aluminum and chromium can be determined from the diluted sample. Background correction should be used and a standard addition method is also recommended to compensate for any matrix or chemical interferences occurring during ashing and atomization. For samples where no chemical interferences are observed and all background effects are compensated for by background correction, normal calibration with oil soluble standards may be employed. Alternatively a sample preparation method where the sample is ashed at 550°C in a vycor crucible could be used thus allowing for the use of aqueous standard solutions for calibration.

***Elements with low volatility:***

For elements with low volatility such as vanadium, determinations can be easily done from a diluted sample. In this case very little background signal should be present. The use of aqueous standards is possible and normal calibration can be used.