Naphtha Analysis Procedure

SOMF-03

(For the Spectroil Family of Spectrometers)

Overview:
The naphtha analysis procedure is a sample preparation method that allows for the analysis of highly volatile fuel samples using the Spectroil M/F or Spectroil Model F Rotating Disk Electrode (RDE) Emission Spectrometers.

The essence of the procedure is that the naphtha sample is mixed with a small quantity of base oil and the mixture is then heated to drive off the volatile fraction. The contaminants in the naphtha remain with the heavier molecules of the base oil and the remaining liquid can be analyzed on the Spectroil M/F or Spectroil Model F Spectrometers following a standard procedure. In order for the procedure to be quantitatively accurate, an analytical balance is used to determine the weight of the naphtha at the beginning of the procedure and the weight remaining after heating. A simple calculation is then applied to determine the concentration of the contaminant elements in the naphtha fuel sample.

Background:
Highly volatile samples such as naphtha and gasoline normally cannot be analyzed with the RDE technique because the samples will ignite when subjected to the electrical arc necessary to excite the elements present in the sample. Some less volatile fuels such as No. 2 diesel and kerosene can be analyzed by utilizing a special aluminum "low flash point sample cover" which is placed on the sample holder and suppresses flame formation. However, naphtha and gasoline samples are so volatile, that even the low flash point cover cannot prevent ignition. This procedure eliminates the inaccuracies from the analysis that are due to sample ignition, plus it can also be used to concentrate the sample to improve detection limits.

Naphtha Analysis Kit:
The items required for the naphtha analysis sample preparation procedure are typically available in most chemical laboratories. As a convenience, a complete kit is available from Spectro Incorporated. The naphtha analysis kit includes the following:

- **Top Loading, 3-place Laboratory Balance** – to weigh the naphtha sample before and after heating on the hot plate.
- **Stainless Steel Sample Cup** – to hold the fuel sample during the preparation procedure. (10 are included in the kit).
- **Hot Plate** – to heat the sample to 340°C to drive off the volatile fraction.
- **Dial Temperature Surface Thermometer** – to measure the temperature on the surface of the hot plate.
- **Temperature Resistant Gloves** – to handle the stainless steel sample cup after heating to 340°C.

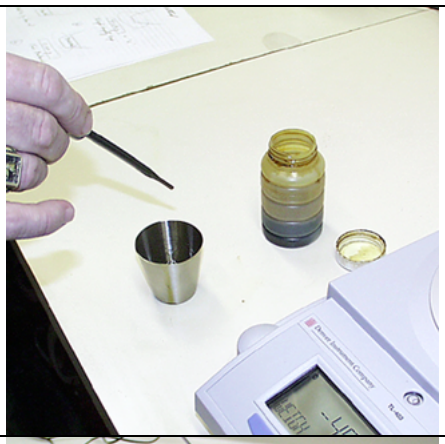
Step by Step Naphtha Analysis Procedure

The following is the step by step procedure to prepare highly volatile fuel samples for analysis with the Spectroil M/F or Spectroil Model F Spectrometers. The weights determined in the procedure are used in a simple equation to calculate the concentration of contaminants in the fuel sample.

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Step 1 Tare the top loading analytical balance to set the reading to "0000" *(To save time, turn on the hot plate, place the surface thermometer on it and preheat to approximately 360°C.)*	
Step 2 Weigh the empty stainless steel sample cup. Note the weight, it becomes variable **"C"** in the final calculation. *(In this example the weight of the empty cup is 37.336 gr.)*	
Step 3 Add approximately 2 grams of base oil to the stainless steel cup. *(Base oil is a highly refined mineral oil that is virtually contaminant free. The base oil used to standardize the Spectroil can be used for this purpose)*	
Step 4 Place the stainless steel sample cup containing the base oil on the balance. Tare the balance. *(This zeros the balance with the weight of the cup and base oil so that the quantity of fuel to be added in the next step can be determined)*	

Step 5 Add the fuel sample to the stainless steel cup containing the base oil. At least 2 times as much fuel sample as base oil should be added. *(The addition of at least 2 times as much fuel sample as base oil improves the detection limit of the analytical procedure. The quantity of fuel sample is not critical since the exact will be determined in the next step)*	
Step 6 Place the stainless steel cup containing the base oil and fuel sample on the balance. Note the weight, it becomes variable **"X"** in the final calculation. *(In this example the weight of the fuel sample is 6.804 gr.)*	
Step 7 Place the stainless steel cup containing the base oil and fuel sample on the hot plate after it has been preheated to approximately 360°C. Leave on the hot plate for at least 10 minutes. **CAUTION:** The hot plate must be placed under a fume hood so that the volatile fumes will be exhausted from the work area.	
Step 8 Put on a temperature resistant glove and remove the stainless steel cup from the hot plate. Allow to cool for 3 to 5 minutes. **CAUTION:** The cup is hot and must be handled accordingly.	

Step 9 Tare the top loading analytical balance to set the reading to "0000"	
Step 10 Weigh the stainless steel cup that contains the base oil and the residual from the fuel sample. Note the weight, it becomes variable **"Y"** in the final calculation. *(In this example the weight of the base oil and residual fuel sample is 41.843 gr.)*	
Step 11 Analyze the sample from the stainless steel cup on the Spectroil using the normal procedure for the analysis of fuel samples. Note the spectrometric analyses for each contaminant elements as they become variable "Z" in the equation. A "Z" value is required for each element of interest. In most instances, and at a minimum, the calculation is performed for sodium and potassium. *(In this example, assume the analytical result as reported by the Spectroil M/F is 1.1 ppm)*	

Plug the variables determined in the procedure and analysis into the following equation and solve for **each element**:

$$\textbf{Concentration of Element in Fuel (ppm)} = \frac{(Y - C)\,Z}{X}$$

Using data from above example, the calculation for sodium becomes:

$$\textit{Sodium Concentration} = \frac{(41.843 - 37.336)\ 1.1.ppm}{6.804} = 0.73\ ppm$$

Repeat the calculation for the other elements using their respective "Z" values as determined by the Spectroil M/F.