

## **SetaAnalytics Technical Applications Note For IP 391, ASTM 6591 & EN 12916 Test Method for Aromatic Content of Distillate Fuels**

### **Background and Need**

In the early 1990's a number of regulatory bodies including the EU implemented a lower fixed volume percent limit on the aromatics content (mono/di/total aromatics) of "vehicular" diesel fuel as a means to achieve further reductions in diesel engine particulate matter (PM) and nitrous oxide (NOx) emissions. In most countries in the world vehicular diesel fuel is the fuel which must be used in both on-road vehicles and in non-road use (e.g. agriculture and construction equipment). Note: Locomotive and marine vehicles are not included in the definition of vehicular use.

Implementation of these volume percent aromatic limits, which is currently 11% max by wt in the EU is measured by using the HPLC test methods: IP 391 or EN 12196 or ASTM D6591.

The amount of Total aromatics is sometimes referred to as PAH (Poly Aromatics Hydrocarbon). Currently the EU are reviewing the max limit of PAH in road diesel in the EN 590 specification and this possibly may be further reduced in the future to further cut emissions from road vehicles especially trucks in 2010 or 2012.

The measurement of PAH or Total aromatics is mandated on all fuels refined or traded as EN 590. This European standard is now becoming widely adopted in many regions of the world such as the Middle East, Africa, Pacific Rim and Australia/New Zealand.

### **The Fuels**

There are a number of different distillate fuels that contain poly aromatic components, and these fuels range for distillate marine fuels such as F76 for military use, to the broad range of road and rail as well as stationary power fuels for turbine generators. The road transport fuels can vary in grade type depending on their application such as ASTM D975 has many different grades depending on the application, whether it takes in to account climatic operating issues, or the fuel is designated for off road use in the agricultural or mining/construction industries.

In the case of Europe there is the universal EN 590 on road diesel specification which has a fixed max value for PAH, but in many member states for other distillate fuel applications, i.e. Marine waterways, or rail/power train requirements, the specifications will often have a PAH range specified. In many parts of the world the ASTM D975 or EN 590 specifications are referenced or closely reflected in the National or Regional specification. In the case of Kero, such as Jet fuel, the aromatics content are specified and measured by the HPLC method IP 436 and this is discussed in more detail in the SetaAnalytics Aviation Aromatics Application note.

### **The Separation & Measurement Instrumentation**

The HPLC system comprises of 5 critical parts to enable quality values to be obtained.

- 1. Mobile Phase Degasser system**
- 2. Injection Valve**
- 3. Separation Column**
- 4. RI Detector**
- 5. Integrator**

#### **1. Degasser System.**

The mobile phase degasser is a very useful tool to ensure the mobile phase system is free of any dissolved air, which can lead to operability problems when the mobile phase pumped on pressure through the pre-packed column system. This can lead to poor separation and may also affect the detection of the aromatic components.

## 2. Injection Valve.

This highly engineered unit allows via the sample loop the delivery and insertion of the sample dissolved in a diluent, to be carried on down into the separation column as one discrete aliquot.

## 3. Separation Column.

This is the most crucial part of the separation system, and the quality of the absorbent material, in terms of chemical derivatisation, particle size and distribution have a major impact on the separation efficiency, the component resolution and the repeatability of the separation. As well as the reproducibility of the column performance for inter lab comparisons. Besides these critical areas, the longevity of the column is also determined by the quality of the packing material and the mechanical functionality of the columns construction.

## 4. RI Detector.

The sensitivity and base line stability of both the measurement source and the detector are very important to ensure good repeatable measurements at the lower levels that are to be determined.

## 5. Integrator

This is the final component of the system that enables the analyst to use either automatic or manual chromatographic determinations of the component peaks as they exit the column and are detected via the Refractive Index measurement. Not only does this system if set up correctly allow effective, repeatable measurements to be made, but will all automatic concentrations to be determined, if programmed with a suitable calibration data set for the components under evaluation. Also the system will provide an archiving capability to allow traceability, plus can be connected to a Laboratory Information Management System (LIMS).

An Example of typical separations is shown in the following example for a diesel meeting the EN 590 specification.

