

Tier III Gasoline Regulations on the Horizon

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On February 19, 2015, the EPA published Tier 3 Annex VI Implementation; Direct Final Rule. This codifies the deadlines and procedure for verifying compliance with the new gasoline and marine fuel requirements. It is now official that the new analytical procedures for Tier 3 gasoline must be in place by January 1, 2016. The methodology selected was Performance Based Testing with specific Statistical Quality Control Requirements. This testing is site-specific and must be performed by the operator at the local site and the records retained there for five years for possible examination by an EPA auditor.

These requirements are a bit complicated and require close examination. The basic concern of the regulation is to reduce environmental pollution from vehicles by lowering the permitted level of sulphur from 30 mg/kg down to 10 mg/kg effective January 1, 2017. In order for this to happen, time has been allotted to purchase and validate the instrumentation which will be used to release this fuel. The one-year period has been set aside for this process to be completed. Thus, in the next six months, all effected parties including refiners, importers and bio-fuel blenders must prepare for these requirements. The EPA has been as flexible as possible in establishing the requirements for the Performance Based Testing as defined in 40 CFR Part 81, April 28, 2014.

The measurement of sulphur in fuel has been defined as an absolute fuel parameter meaning that a gravimetric standard may be prepared. This distinguishes it from consensus named fuels which derive their accepted values from analyses in a series of laboratories. The remainder of the Tier 3 Requirements is driven by this distinguishing feature of gravimetric standards. With the establishment of gravimetric standards the requirements for precision and accuracy are defined for two different concentration ranges. The minimum acceptable values are also established by examining the methods available through a voluntary consensus-based standards body, ASTM.

The process was begun by examining the applicable methods and examining both the accuracy and repeatability of typical samples. For the accuracy tests it is broken into two ranges of 1-10 PPM and 10-20 PPM. The requirement is to perform 10 tests on a commercially available gravimetric standard. The arithmetic average of the results in the range 1-10 PPM may not differ from the accepted reference value by more than 0.71 PPM. In the case of the 10-20 PPM gravimetric sulphur standard may not differ from the accepted reference value by more than 1.0 PPM. This testing protocol is very routine and is often included in some form as a part of an SQC (standard quality control) program.

These tests can be performed quite readily after the receipt of a new analyser. It is a matter of preparing ten samples of the gravimetric and running them on the instrument and recording or saving the results. The results can then be averaged together in a simple spreadsheet or with a calculator. The output result is then subtracted from the accepted reference value. If the value is less than that given for the established concentration range given above, the test has been passed. Since each of these series of tests should take about one hour, it is preferable to do this test first. If for some reason you are not successful, additional work must be done prior to moving on.

After these two testing protocols have been successfully completed it is time to do the precision testing. This test is quite a bit more involved as it requires 20 days to complete. In this case it involves the testing of a particular sample; in this case commercially available gasoline with a concentration of sulphur very close to 10 PPM. There are some requirements also relevant (seven or fewer tests per week and two or fewer tests per day). From this data set the standard deviation is then computed. This result is then related back to the ASTM Method with the highest repeatability "r" value, D7039.

The "r" for a 10 PPM sample is 1.75 PPM from the method itself. This value is first multiplied by 1.5 and then divided by 2.77. In the case of a 10 PPM sample this works out to be 0.95



PPM. The result from the 20 Day Test must be less than or equal to this value. This data comparison again results in a pass fail type of answer. The addition of the factor of 1.5 to the calculation provides a good "safety cushion" to permit methods to pass this testing. In the example of the previous ultra-low sulphur diesel (ULSD) performance based testing, all laboratories were able to successfully qualify for running these samples. Similar results are to be expected for ULSG.

This testing represents the first step in the qualification process for the EPA. The next phase is the establishment of an acceptable SQC program. These requirements are spelled out in great detail and are drawn up based upon ASTM Method D6299. These programs are spelled out in detail on pages 23637 and 23638. The relevant paragraphs will probably only make sense if you have a current copy of D6299 at your disposal. Although these requirements are only required after the commissioning of the instrument, they must be planned out well in advance. With January 1, 2016 not that far in the future, now is the time to develop the strategy to meet these requirements. There are many resources available to you through your local instrument supplier.

Gas Chromatograph for Hydrocarbon Measurement

Baseline (USA), a **MOCON** company, announces the release of the PetroAlert Series 9100 Gas Chromatograph for mud logging during oil and gas exploration and other hydrocarbon measurement applications. Utilising a FID (flame ionisation detector) provides exceptional detection capabilities of both light and heavy hydrocarbons. Measurement of C1 to C5 (methane, ethane, propane, butanes and pentanes) can be accomplished in as little 30 seconds.

The PetroAlert not only provides a fast measurement, but a precise one as well. A measurement range from low ppm to high percent concentration levels allow for both quantitative and qualitative analysis. During the mud logging process, the speed and accuracy provided by the PetroAlert Series 9100 while drilling enables the geologist to make timely decisions regarding the well's potential, creating a more efficient process.

Baseline's mission is to provide turnkey, high-quality solutions to our customers. The expansion of the Series 9100 product line to include the PetroAlert is key in keeping with Baseline's customer expectations.

The Series 9100 GC is relatively new to the market and offers an advanced level of technological innovation. It is fully integrated with powerful GC software eliminating the need for an external PC. The programmable touchscreen and USB interfaces offer a great deal of user flexibility.

Other key features include remote access and control, automatic calibration, internal data storage allowing for unattended operation. Detector choices include FID (flame ionisation detector), TCD (thermal conductivity detector), PID (photoionisation detector) and HS PID (high sensitivity photoionisation detector).



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