

proposing performance-based test procedures. Under the performance-based approach, a given test method could be approved for use in a specific laboratory or for field testing by meeting certain precision and accuracy criteria. Properly selected precision and accuracy values potentially would allow multiple methods and multiple commercially available instruments to be approved, thus providing greater flexibility in method and instrument selection while also encouraging the development and use of better methods and instrumentation in the future. For example, we are hopeful that with more time and effort a simpler test can be developed that can avoid the use of reagents and the generation of hazardous waste that is by product of the current commonly accepted method.

Under the performance criteria approach proposed today, methods developed by consensus bodies as well as methods not yet approved by a consensus body would qualify for approval provided they met the specified performance criteria as well as the recordkeeping and reporting requirements for quality control purposes. There would be no designated marker test method. We request comment on whether it would be more appropriate to adopt a designated marker test method. Such comments would be most useful if they include complete details on a suitable designated marker test method.

1. How Could a Given Marker Test Method Be Approved?

Under the proposed performance criteria approach, a given marker test method would be approved for use under today's program by meeting certain precision and accuracy criteria. Approval would apply on a laboratory/facility-specific basis. If a company chose to employ more than one laboratory for fuel marker testing purposes, then each laboratory would have to separately seek approval for each method it intends to use. Likewise, if a laboratory chose to use more than one marker test method, then each method would have to be approved separately. Separate approval would not be necessary for individual operators or laboratory instruments within a given laboratory facility. The method would be approved for use by that laboratory as long as appropriate quality control procedures were followed.

In developing the precision and accuracy criteria for the sulfur test method, EPA drew upon the results of an interlaboratory study conducted by the American Society for Testing and Materials (ASTM) to support ASTM's

standardization of the sulfur test method. Unfortunately, there has not been sufficient time for industry to standardize the test procedure used to measure the concentration of solvent yellow 124 in distillate fuels or to conduct an interlaboratory study regarding the variability of the method. Nevertheless, the European Union has been successful in implementing its marker requirement while relying on the marker test procedures which are currently available, as noted above. We are proposing to use this procedure to establish the precision and accuracy criteria on which a marker test procedure would be approved under the performance-based approach. We request comment on the suitability of the proposed reference marker test method, including whether standardized acceptability criteria exist regarding the visible spectrometer apparatus and associated measurement procedure used in performing the test.

There has been substantial experience in the use of the proposed reference marker test method since the August 2002 effective date of the European Union's marker requirement. However, EPA is aware of only limited summary data on the variability of the reference test method from a manufacturer of the visible spectrometer apparatus used in the testing.³³⁵ The stated resolution of the test method from the materials provided by this equipment manufacturer is 0.1 mg/L, with a repeatability of plus or minus 0.08 mg/L and a reproducibility of plus or minus 0.2 mg/L.³³⁶ In the lack of more extensive data, we propose to use these available data as the basis of our proposed precision and accuracy criteria as discussed below. We request that comments which suggest that these data are unsuitable for the intended use also include additional test data where possible to improve the derivation of precision and accuracy criteria.

Using a similar methodology to that employed in deriving the proposed

sulfur test procedure precision value results in a precision value for the marker test procedure of 0.043 mg/L.³³⁷ However, we are concerned that the use of this precision value, because it is based on very limited data, might preclude the acceptability of test procedures that would be adequate for the intended regulatory use. In addition, the lowest measurement of marker concentration that would have relevance under the regulations is 0.1 mg per liter. Consequently, we are proposing that the precision of a marker test procedure would need to be less than 0.1 mg/L for it to qualify. We request comment on this proposed precision level.

We are proposing that to demonstrate the accuracy of a given test method, a laboratory facility would be required to perform 10 repeat tests, the mean of which could not deviate from the Accepted Reference Value (ARV) of the standard by more than 0.05 mg/L. We believe that the proposed accuracy level is not overly restrictive, while being sufficiently protective considering that the lowest marker level of regulatory significance would be 0.1 mg/L. Ten tests would be required using each of two different marker standards, one in the range of 0.1 to 1 mg/L and the other in the range of 4 to 10 mg/L of solvent yellow 124. Therefore, a minimum of 20 total tests would be required for sufficient demonstration of accuracy for a given marker test method at a given laboratory facility. Finally, any known interferences for a given test method would have to be mitigated. We are proposing that these tests be performed using commercially available solvent yellow 124 standards. Since the European Union's marker requirement would have been in effect for over six years and we expect this requirement to continue indefinitely, we believe that such standards would be available by the implementation date for this proposed rule. We request comment on this assessment and on whether we should allow facilities that conduct the proposed tests to blend up their own marker standards using a pure supply of the fuel marker.

We request comment on the proposed precision and accuracy criteria described above. These requirements are not intended to be overly burdensome. To the contrary, we believe these requirements are equivalent to what a laboratory would do during the normal start up procedure for a given test

³³⁵ Technical Data on Fuel/Dye/Marker & Color Analyzers, as downloaded from the Petroleum Analyzer Company L.P. Web site at http://www.petroleum-analyzer.com/product/PetroSpec/lit_pspec/DTcolor.pdf.

³³⁶ Repeatability and reproducibility are terms related to test variability. ASTM defines repeatability as the difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials that would, in the long run, in the normal and correct operation of the test method be exceeded only in one case in 20. Reproducibility is defined by ASTM as the difference between two single and independent results obtained by different operators working in different laboratories on identical material that would, in the long run, be exceeded only in one case in twenty.

³³⁷ See Section VIII.D. of this proposal for a discussion of the methodology used in deriving the proposed precision and accuracy values for the sulfur test method.