

This document is for ASTM Committee use only. It shall not be reproduced, circulated, or quoted, in whole or in part, outside of ASTM Committee activities, except with the approval of the Chairman of the Committee with jurisdiction, or the President of the Society.

**7B.8, Task Group on Ninety-Cycle Shear Stability (NCSS)
Report to Heavy Duty Engine Oil Classification Panel
22 June 2004**

Executive Summary

The Task Group hopes to have an approved test method by October of this year. The test procedure has been finalized, as Work Item WK 2880, draft 5, and a preliminary precision statement is available. Repeatability and reproducibility of 90-cycle viscosity loss are, respectively, 1.8% and 2.9% (absolute, not percent of the result). Repeatability and reproducibility of 30-cycle viscosity loss are, respectively, 1.4% and 2.5%, which are statistically equivalent to the precision for ASTM D 6278, 1.05% and 2.68%.

The preliminary precision statement is based on 49 results from five laboratories for a single industry reference oil, TMC 820-2. The Task Group will begin a full Round Robin, comprising 10 oils and nine labs, within two weeks. The Task Group expects to revise the test method with the new precision statement before the December 2004 meeting.

The test method (Draft 5) includes a 30-cycle intermediate result in addition to the 90-cycle final result. The initial volume of test oil is increased from 170 mL to 200 mL to accommodate the intermediate sample removal. The number of pump strokes per cycle is increased for the initial 30 cycles to maintain constant shearing severity for all 90 cycles. The Task Group also considered a variation wherein the initial volume of oil is kept the same as D 6278 (170 mL), and the volume for the second phase of shearing is reduced to 140 mL following removal of the 30-cycle sheared sample. Although both variations have advantages and disadvantages, the Task Group had no clear preference; the deciding factor was the availability of a precision statement for the Increased Initial Volume method.

Test Method WK2880, Draft 5 is attached.

History

The Task Group on Ninety-Cycle Shear Stability was formed by Section D02.07.B on 17 June 2003, to develop a modification of Test Method D 6278, Standard Test Method for Shear Stability of Polymer Containing Fluids Using a European Diesel Injector Apparatus, that will shear oils for 90 cycles instead of 30 cycles. At that time, the method was proposed for 90-cycles only. In a series of conference call meetings, the Task Group developed Draft 1 of the new method, and made plans for a Round Robin.

On 7 and 12 August 2003, Section B.2 suggested a 30-cycle intermediate result is not required, and requested HTHS measurements on the sheared oils. They emphasized the key requirement is to develop a test method in time for inclusion in the API CI-4+

specification, and acknowledged including a 30-cycle intermediate result will add complexity and could delay the method development.

On 17 September, the draft method was registered as Work Item WK2880.

On 8 October 2003, Subcommittee D02.B issued a formal written request to Subcommittee 7 to develop a test method for 90-cycle shear stability that includes a 30-cycle intermediate result, and that uses a single oil charge. Precision at both 30- and 90-cycles was requested. The Task Group considered 30-cycle sample with replacement and without replacement. Replacement maintains the total oil volume and the relationship between stroke number and shear cycles, but contaminates the sample with unsheared oil. Sampling without replacement maintains the integrity of the sample, but requires re-setting the stroke counter. Sampling without replacement was chosen. Draft 2 was developed to include a 30-cycle intermediate result without replacement and circulated on 25 November 2003.

At its 7 December 2003 meeting, the Task Group considered the conflicting requests from HDEOCP to 1) develop a 90-cycle method with a 30-cycle intermediate result, and 2) choose whatever method will be fastest to develop. Several options to obtain a 30-cycle intermediate result were considered and the Task Group chose the “Delta Volume” method, wherein the stroke counter is reset in proportion to the change in sample volume following removal of the intermediate 30-cycle sample. The Task Group recommended, by a vote of 7-0, selecting a 90-cycle only method. At the HDEOCP meeting, this recommendation was rejected, and the requirement for a 30-cycle intermediate result was confirmed.

The design, plan, and timing for a Round Robin were finalized. During the next few months, the Round Robin oils were shipped by the donors to the Chair. Two labs joined the Round Robin, and the final list of participating labs was confirmed.

Draft 3, which includes a 30-cycle intermediate result, was circulated on 7 April 2004. After review of Draft 3, and expressing some degree of frustration, the Task Group instructed the Chair to re-visit the issue of 30-cycle intermediate result with HDEOCP or Section B.2. In conversations on 27 April and 10 May 2004, Section B.2 re-confirmed the need for a 30-cycle intermediate result. The Chair declared it a closed issue.

On 21 May 2004, Draft 4 was circulated. This version, provided by Perkin-Elmer, reflects current practice of the Mack T-11 test development group. On 10 June 2004, “mini-Round Robin” data for this method was made available by TMC, via Perkin-Elmer. These data comprise 49 results from five labs on a single industry reference oil, TMC 820-2.

At the 20 June 2004 Task Group meeting, the “mini-Round Robin” results were analyzed using the methods in ASTM E691, resulting in a preliminary precision statement for the Draft 4 procedure. Repeatability and reproducibility for 90-cycle viscosity loss are, respectively, 1.8% and 2.9% (absolute, not percent of the result). Repeatability and

reproducibility of 30-cycle viscosity loss are, respectively, 1.4% and 2.5%, which are statistically equivalent to the precision for ASTM D 6278, 1.05% and 2.68%.

Repeatability and reproducibility for initial oil kinematic viscosity were calculated, and found to be, respectively, 1.4% and 2.4% of the mean (relative percent). These values compare unfavorably with the published D 445 precision statement for fully formulated oils, of 0.26% and 0.76%. The most recent laboratory cross-check data for D 445 for fully formulated oils has a reproducibility of 1.38%, which is still significantly better than that obtained in this “mini-Round Robin.” The cause for the discrepancy is unknown, but may be due to labs using the Mack T-8 Appendix viscosity procedure instead of D 445. WK 2880 requires using D 445 for kinematic viscosity measurements of unsheared and sheared oil samples.

Draft 4 was reviewed for editorial, grammatical, and typographical issues, resulting in Draft 5. Draft 5 will be used for the Round Robin. Data submission sheets for the Round Robin were designed.

Timeline

The Round Robin will be launched immediately following June 2004 meeting week. It is expected the samples will arrive at the participating labs by the beginning of July, and it will take at least two months for the required 19 runs (beginning September). It will take one or two weeks to analyze the Round Robin and write a Research Report. Under ideal circumstances, this may be in time for the 04-04 Subcommittee ballot, which issues 1 October 2004. Failing that, it is possible Headquarters will allow an extra Subcommittee ballot before the December 2004 meeting week. The Task Group expects to resolve the Subcommittee ballot at the December 2004 meeting, and proceed to Committee ballot 05-01, whose return deadline will be around the end of March 2005.

To expedite publication of the method, the Task Group will go to ballot immediately with Draft 5 including the preliminary precision statement. This should satisfy the Form and Style Manual requirements for a Precision Statement (sections A21.2.2 and A21.2.3). The method will indicate a more formal precision statement is being prepared by a full Interlaboratory Study. The ballot will be conducted in Subcommittee 7 with a courtesy ballot to Subcommittee B.

Membership, Attendance, and Round Robin Participants

<u>Name</u>	<u>Company</u>	<u>Status</u>	<u>Last Attended</u>	<u>Round Robin</u>
Fred Girshick	Infineum	Chair	Jun 2004	Participant
Reid Patterson	Lubrizol	Secretary	Jun 2004	Participant
Ernst Bielmeier	RohMax			Participant
Mike Birke	SWRI	Mailing List		
Jeff Clark	TMC	Member	Jun 2004	
Mike Covitch	Lubrizol	Oil supplier		
Mark Devlin	Afton Chemical		Dec 2003	Participant
David Dragert	Petro-Canada		Jun 2004	
Alan Flamberg	RohMax	Member	Jun 2004	Participant
Joe Franklin	Perkin Elmer	Member	Jun 2004	Participant
David George	Chevron Oronite	Member	Dec 2003	
Herman George	Lubrizol	Member	Dec 2003	
Dhanesh Goberdhan	Infineum UK	CEC Liaison		
Becky Grinfield	SWRI	Member	Jun 2004	Participant
Tom Hitchner	Exxon Mobil			Participant
Mark Kelley	BP-Castrol	Member	Jun 2004	Participant
Steve Kennedy	Exxon Mobil	Member		
Jorge Klisans	PDVSA		Jun 2004	
Jim McGeehan	HDEOCP	Ex officio		
Helmut Melchior	RohMax			Participant
Greg Müller	Tannas			Participant
Jerome Obiols	TOTAL France		Jun 2004	
Chris Onyeso	Ethyl Corporation		Jun 2004	
Charlie Passut	Ethyl	Member		
Greg Shank	Mack Trucks	Member		
Marilu Stea	PDVSA		Jun 2004	
Dave Stehouwer	Section B.2	Ex officio		
Fanny Uejias	PDVSA		Jun 2004	

Respectfully submitted,

Fred W. Girshick
 Chair, Task Group on Ninety-Cycle Shear Stability
 Section B, Subcommittee 7
 908-474-3247
Fred.Girshick@Infineum.com