

## HEAVY-DUTY ENGINE OIL CLASSIFICATION PANEL

OF

ASTM D02.B0.02

June 22, 2004

The Grand America Hotel – Salt Lake City, UT

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### ACTION ITEMS

- |           |  |                           |
|-----------|--|---------------------------|
| <b>1.</b> | <b>Request Sub-committee 3 to review D874 precision.</b> | <b>HDEOCP/J. McGeehan</b> |
| <b>2.</b> | <b>Resolve questions on voting weights.</b>              | <b>HDEOCP</b>             |
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### MINUTES

- 1.0 Call to Order
  - 1.1 Chairman Jim McGeehan called the June 22, 2004 meeting of the HDEOCP to order at 1:13 p.m. in the Imperial D ballroom of the Grand America Hotel in Salt Lake City, Utah. There were 19 members present or represented and there were approximately 53 guests present. The attendance list is shown as Attachment 2.
- 2.0 Agenda
  - 2.1 The published agenda (Attachment 1) was reviewed and Fred Girshick asked for an early slot to review work his task force had done on 90 cycle shear stability, at the request of the HDEOCP.
- 3.0 Minutes
  - 3.1 The minutes of the May 18, 2004 meeting were approved as distributed and posted on the TMC web site.
- 4.0 Membership
  - 4.1 There were no changes to the membership but Scott Harold of CIBA was recognized as the most recent member. See Attachment 3 for the membership list.
- 5.0 Shear Stability
  - 5.1 Fred Girshick reported on work his Section 7 task group had done to establish a 90 cycle shear stability method. His report is shown as Attachment 4 & 4a, along with Draft 5 of Work Item 2880 (Attachment 5) for the test method. They plan to expedite a ballot for the method in Sections 7 and Sub-Committee B by using preliminary precision estimates obtained during development. The precision estimates will be refined once a proposed round-robin is completed. Fred noted an inconsistency in kinematic viscosity measurements between the five labs participating in the method development work.
- 6.0 Chemical Limits

- 6.1 Rick Finn reported on his task force efforts to reach accord on chemical limits for PC-10 oils, see Attachment 6. The exit ballot on the proposed limits drew seven negatives. Five of those were resolved by the Task Force, but two remained.
  - 6.2 With regard to the DDC negative on the sulfur limit being too high, Pat Fetterman moved and Bill Runkle seconded that we move forward with 0.4% S maximum as a non-critical limit. Discussion focussed on the lack of data indicating sulfur's effect on aftertreatment devices. Rick Finn presented a slide (Attachment 7) on sulfur effects.
  - 6.3 The CIBA negative on phosphorus was withdrawn and CAT would change their negative on sulfur and phosphorus being too high if the matrix oils were blended with lower phosphorus. Pat Fetterman moved and Steve Kennedy seconded a motion stating that the matrix oils may be blended at lower SASH, P and S values than the proposed PC-10 limits. The motion passed with 18 for, 0 against and 0 abstain.
  - 6.4 As a carry over from task force discussions the day before, tiered limits for sulfated ash (SASH) were discussed. Jim Rutherford presented information on multiple test acceptance criteria (MTAC) and tiered limits, with some examples. See Attachment 8. Dave Stehouwer moved and Bill Runkle seconded that the tiered limits of 0.98 for one test, 1.0 for two tests and 1.01 for three tests as proposed by Jim Rutherford, be accepted as the limits for SASH. The motion passed with 10 for, 3 against and 5 abstains. Given the underwhelming positive response, discussion continued. Eventually, Pat Fetterman moved and Charlie Passut seconded that for determination of SASH by D874 against the non-critical PC-10 limits, we accept a value of 1.00% maximum for one test, 1.02 for two tests and 1.03 for three tests. These tiered limits are to be reflected in D4485. The motion passed with 18 for, 0 against, 0 abstain. Sub-Committee 3 is to be asked to review the precision of D874.
  - 6.5 Pat Fetterman moved and Steve Herzog seconded that we accept a non-critical phosphorus limit of 0.12 % maximum, by method D4951. The motion passed with 17 for, 0 against and 1 abstain.
  - 6.6 Jim McGeehan reviewed a slide (Attachment 9) showing the now adopted chemical limits box for PC-10.
- 7.0 Mack T-12
- 7.1 Greg Shank reviewed the T-12 development status. See Attachment 10.
- 8.0 Cummins ISB / ISM
- 8.1 Dave Stehouwer presented updates by Warren Totten on the ISB. See Attachment 11.
  - 8.2 Dave also reviewed the ISM situation...see Attachment 12. In a change to the 6.5% soot values shown on slide 9, they now plan to target 6.0% soot for the next phase of tests, using oils 1004, 830 and ISM(A). Greg Shank expressed support for the three oil approach.
- 9.0 Caterpillar C13
- 9.1 Abdul Cassim reviewed the C13 development status (see Attachment 13) and indicated they plan to have inspected parts to the labs by early August.
  - 9.2 Tom Franklin reported for the C13 Task Force and indicated a mini-matrix will use closed crankcase ventilation (CCV) and PC-10 fuel (<15 ppm S). They need a high discrimination oil. See Attachment 14.
- 10.0 New Category Development Team
- 10.1 Bill Runkle, chairman of the NCDT, reported that EMA has not reached a decision yet on the Caterpillar request to include the 1P in PC-10. See Attachment 15. The PC-10

timeline now shows a "Technology Demonstration" period in 2005 with limit setting and product qualification after that.

10.2 Jim McGeehan reviewed the slide (Attachment 16) of PC-10 requirements.

10.3 Greg Shank remarked he is looking for HD oil data from the Seq. IIIG.

#### 11.0 PC-10 Matrix Design and Funding

11.1 Steve Kennedy presented a report on the matrix design and funding activities (see Attachment 17). Concern arose from the floor that the ISM may need matrix testing and Larry Kuntschick of ILMA expressed concern about the proposed reduced time to qualify oils for license.

#### 12.0 Other Business

12.1 Jim McGeehan showed his slide on PC-10 progress thus far...Attachment 18.

12.2 The EMA (Greg Shank) questioned the 75% majority to move forward and how much weighting, if any, EMA votes would receive. This item is to be put on the next meeting agenda.

12.3 Pat Fetterman requested an endorsement of an aromatics range change for PC-10 fuel. The current range is 28 to 33.5% and the proposed new range is 26 to 31.5%. Pat moved acceptance of this range change and Lew Williams seconded the motion, which passed by unanimous voice vote.

12.4 Since time was running late, the 1N liner issue was skipped, with a request that it be presented to "B" the next day. See attachments 19 & 20 for what was to have been presented.

12.5 Volunteers for the "PC-10 Fuel Supplier Selection Task Force", solicited by Jim Wells, thus far are: Tom Franklin, Pat Fetterman and Mesfin Belay. Please contact Jim if you are interested in participating.

#### 13.0 Next Meeting

13.1 The next meeting is planned for September 29<sup>th</sup> in Chicago, at the Rosemont DoubleTree.

#### 14.0 Adjournment

14.1 The meeting was adjourned at 4:30 p.m.

#### 15.0 Report to "B"

15.1 Jim McGeehan's report to B.02 is shown as Attachment 21.

Submitted by:

Jim Wells  
Secretary to the HDEOCP

**Tentative Agenda**  
**ASTMSECTION D.02.BO.02**

ATTACHMENT 1

**HEAVY-DUTY ENGINE OIL CLASSIFICATION PANELS**

**Grand America Hotel, Salt Lake City, UT**  
**June 22, 2004**  
**1:00-5:00 pm**

**Chairman/ Secretary:** Jim Mc Geehan/Jim Wells  
**Purpose:** PC-10  
**Desired Outcomes:** PC-10 timing, tests, chemical limits.

TOPIC	PROCESS	WHO	TIME
Agenda Review	<ul style="list-style-type: none"> <li>• Desired Outcomes &amp; Agenda</li> </ul>	Group	1:00-1:05
Minutes Approval	<ul style="list-style-type: none"> <li>• May 18<sup>th</sup>, 2004</li> </ul>	Group	1:05-1:10
Membership	<ul style="list-style-type: none"> <li>• Changes: Additions</li> <li>• Comments</li> </ul>	Jim Mc Geehan	1:10-1:20
Chemical Limits	<ul style="list-style-type: none"> <li>• Exit Criteria ballot results</li> <li>• Discussion</li> <li>• Vote</li> </ul>	Rick Finn	1:20-2:20
PC-10 Test Development report	<ul style="list-style-type: none"> <li>• Mack T-12</li> <li>• Cummins ISB</li> <li>• Cummins ISM</li> <li>• Caterpillar C13</li> <li>• Caterpillar IP replacing 1N</li> <li>• Review all the tests in category</li> <li>• Exit-Criteria ballot date on PC-10 engine tests mid November.</li> </ul>	Greg Shanks Dave Stehouwer Abdul Cassim  Jim Mc Geehan	2:20-3:20
Task-Force for Matrix	<ul style="list-style-type: none"> <li>• Team selection</li> <li>• Matrix oils</li> <li>• Timing of availability of oils</li> </ul>	Steve Kennedy	3:20-3:45
Funding	<ul style="list-style-type: none"> <li>• Status of funding</li> </ul>	Steve Kennedy	3:45-4:00
NCDT Time-line	<ul style="list-style-type: none"> <li>• NCDT decision on PC-10 time-line</li> </ul>	Bill Runkle	4:00-4:15
HDEOCP Motions	<ul style="list-style-type: none"> <li>• Completed to date on PC-10</li> </ul>	Jim Mc Geehan	4:15-4:30
Cat 1N	<ul style="list-style-type: none"> <li>• Correction to TLHC on with the new 1Y3998 liner</li> <li>• Vote</li> </ul>	Jim Mc Cord	4:30-4:50
New or old business			4:50-5:00

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3	Matthew Urbanak - Shell	Mesfin Belay - Detroit Diesel
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13		
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# **Ninety-Cycle Shear Stability (NCSS) Task Force,**

**Section B on Non-Newtonian High Temperature Viscosity  
Subcommittee 7 on Flow Properties**

## **Report to the HDEOCP**

**F. W. Girshick, Chair  
R. Patterson, Secretary  
22 June 2004**

# CONCLUSIONS

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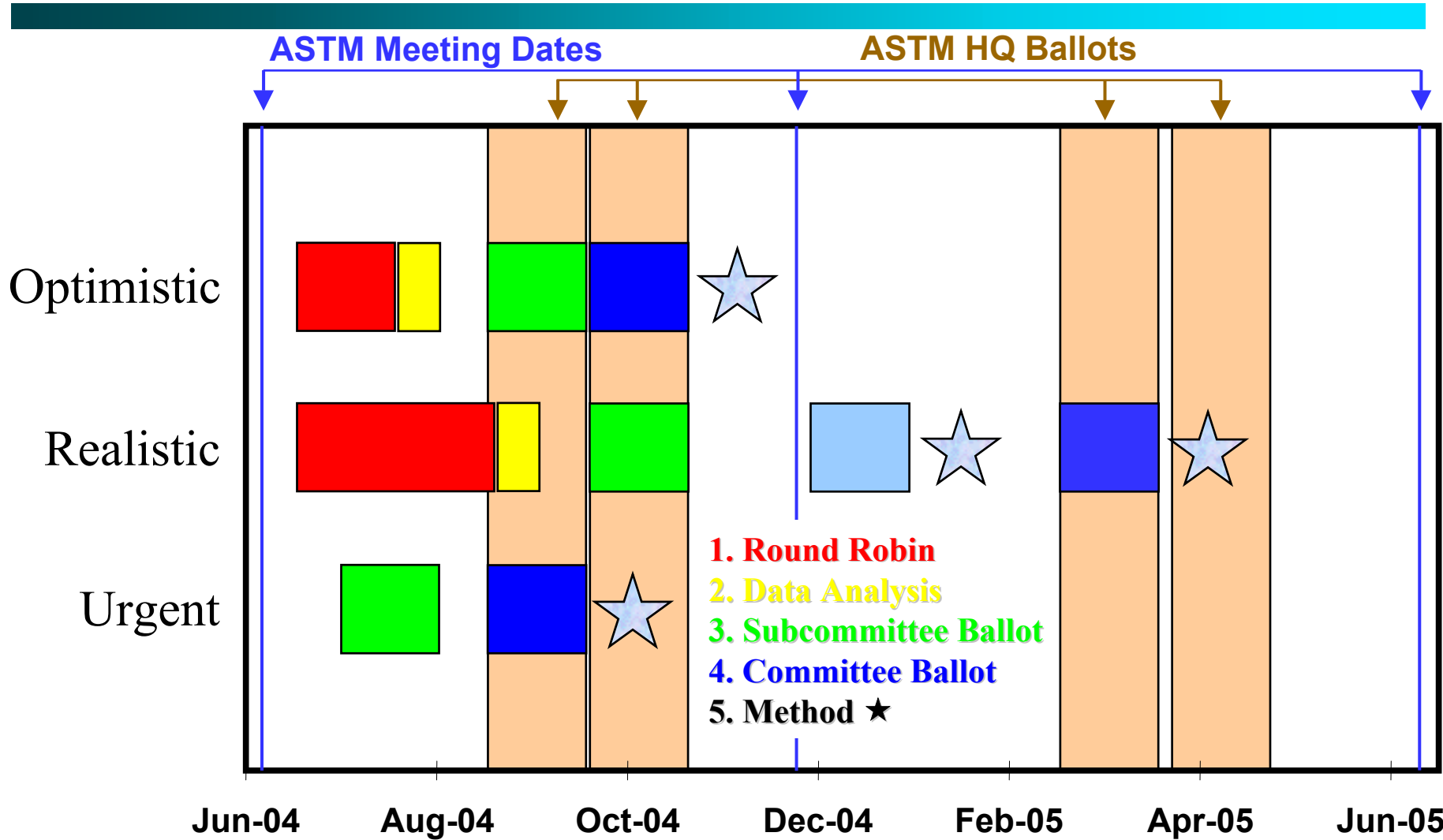
- ❖ **Expect to have an approved Test Method October 2004**
  - **Expedited ballot**
  - **Preliminary precision statement**
    - ⇒  $r = 1.8\%$  (*absolute*)
    - ⇒  $R = 2.9\%$  (*absolute*)
- ❖ **Expect to have a revised Test Method by April 2005**
  - **Approved Precision Statement**
    - ⇒ *Based on Full Round Robin*

# BRIEF HISTORY

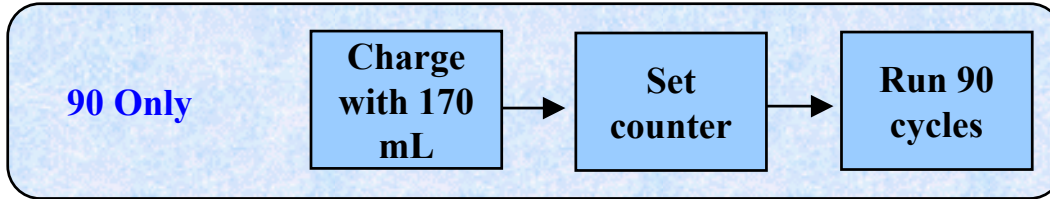
- ❖ **17 June 2003 – Task Force formed by Subcommittee 7, Section B**
  - Develop a test method for 90 cycle (only) shear stability
- ❖ **9 July 2003 – Task Force meeting (conference call)**
- ❖ **12 August 2003 – Draft 1 circulated for comment**
  - Round Robin plans proceeding
- ❖ **8 October 2003 – HDEOCP meeting**
  - Formal request from Subcommittee D02.B to develop test method
    - ⇒ *Shear stability at 90 cycles*
    - ⇒ *Include 30 cycle intermediate result*
    - ⇒ *Generate precision at both 30 and 90 cycles*
- ❖ **25 November 2003 – Draft 2 circulated for comment**
- ❖ **7 December 2003 – Task Force meeting**
  - Recommend “90 cycle only” to Subcommittee B as fastest to develop
  - Subcommittee B rejects recommendation and requests 30 & 90 cycle in same method
- ❖ **7 April 2004 – Draft 3**
- ❖ **10 May 2004 – B.2 re-confirms request for 30 & 90 results**
- ❖ **21 May 2004 – Draft 4 circulated (Perkin-Elmer method)**
- ❖ **10 June 2004 – “Mini” Round Robin data from T-11 Surveillance**
- ❖ **20 June 2004 – Task Force meeting**
  - Draft 5 finalized



# Proposed Timeline(s)

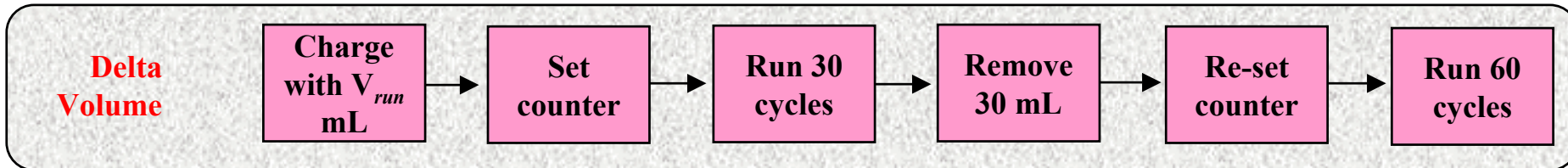


# Method Options



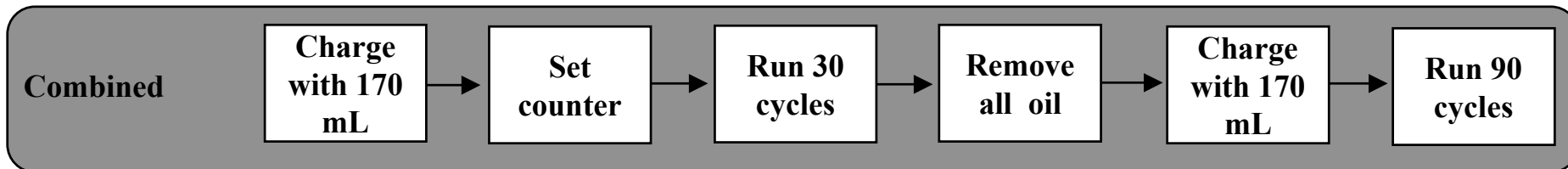
Recommended by the Task Force (7-0)

Fastest to develop



Selected by Subcommittee D02.0B

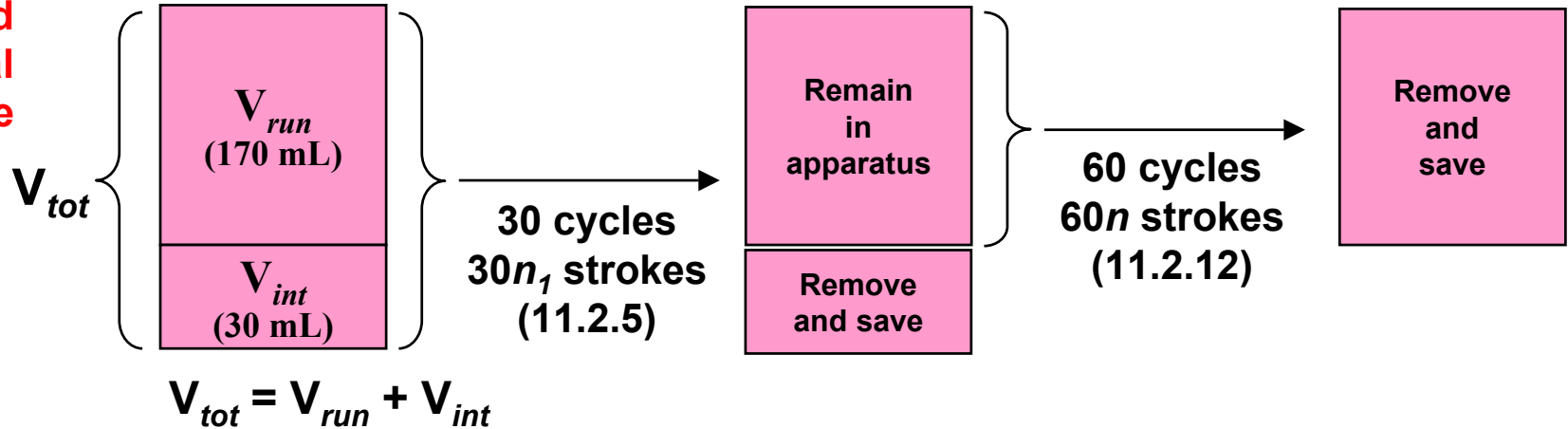
Slowest to develop



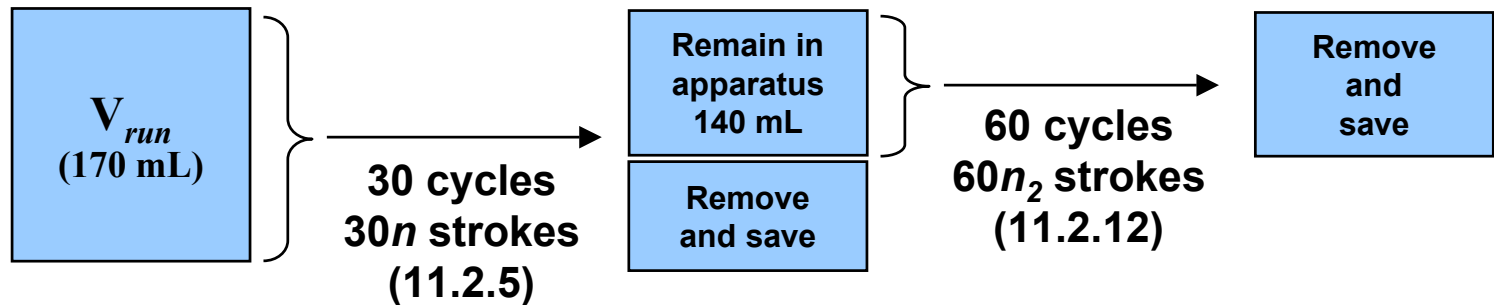
Dropped early – Doesn't make much sense

# Volume Change Methods

Increased Initial Volume



Constant Initial Volume

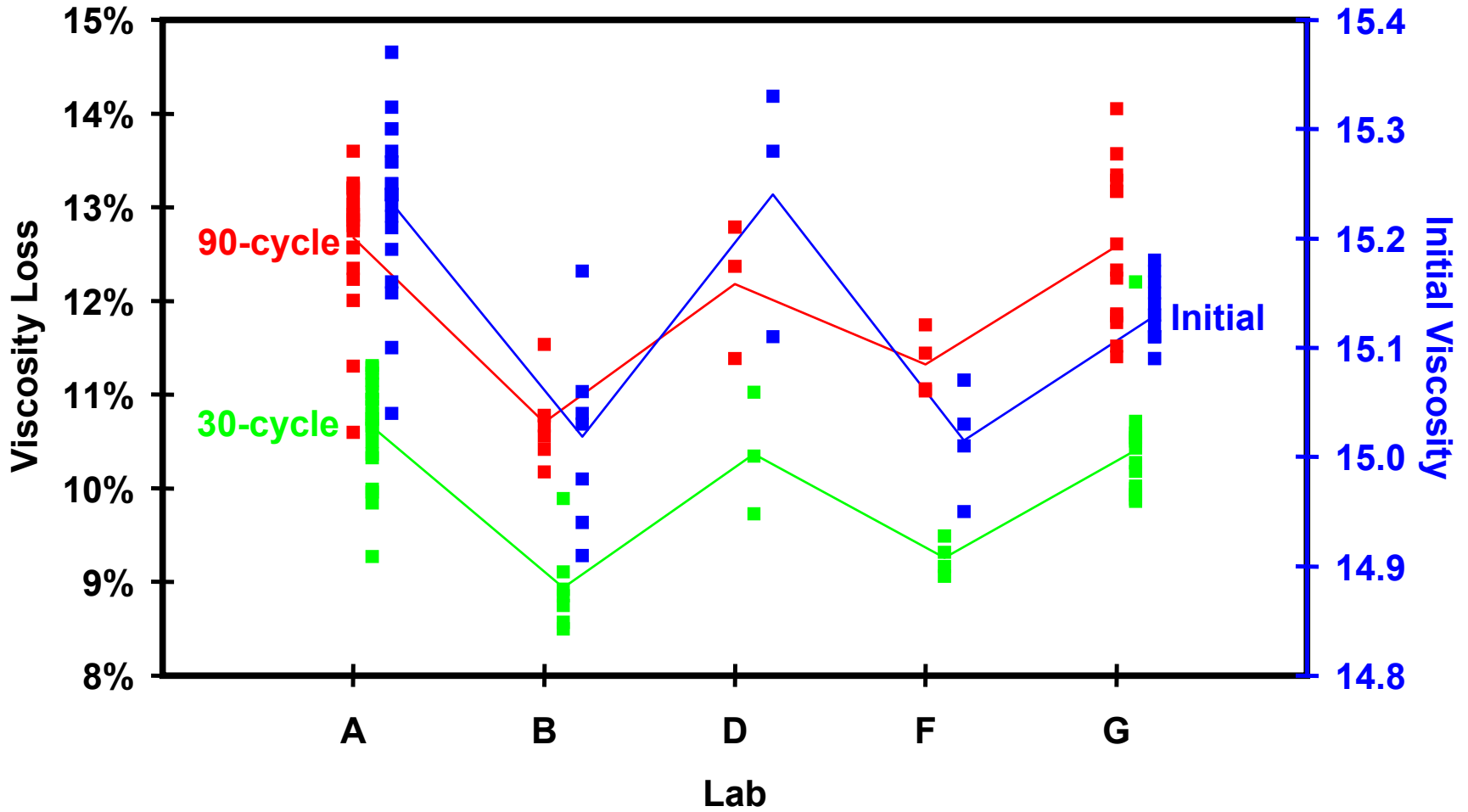


$n$  determined in section 10.3

$$n_1 = \left[ \frac{(170 + 30)}{170} \right] * n$$

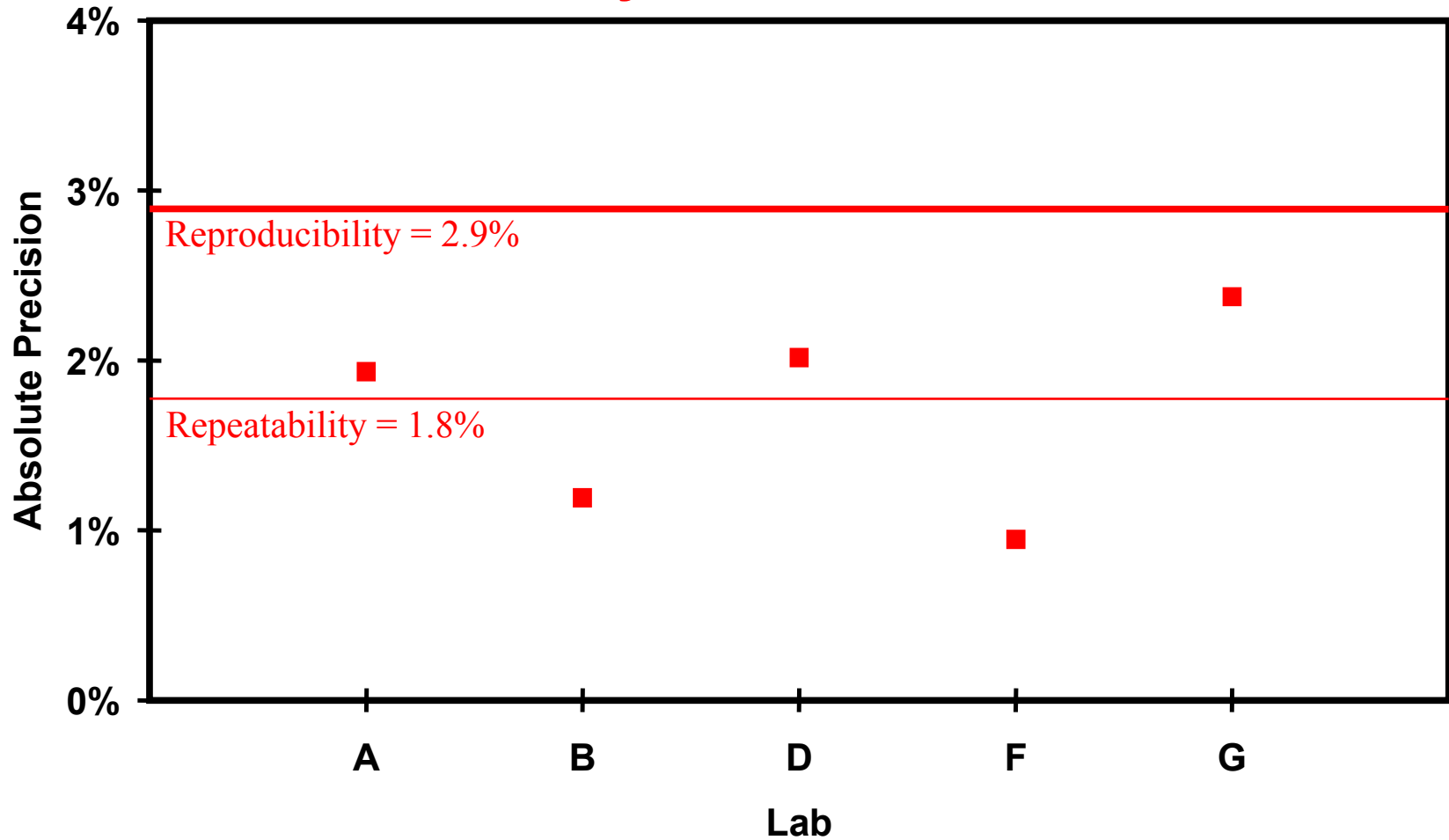
$$n_2 = \left[ \frac{(170 - 30)}{170} \right] * n$$

# ASTM 90-Cycle Shear Data (TMC 820-2)



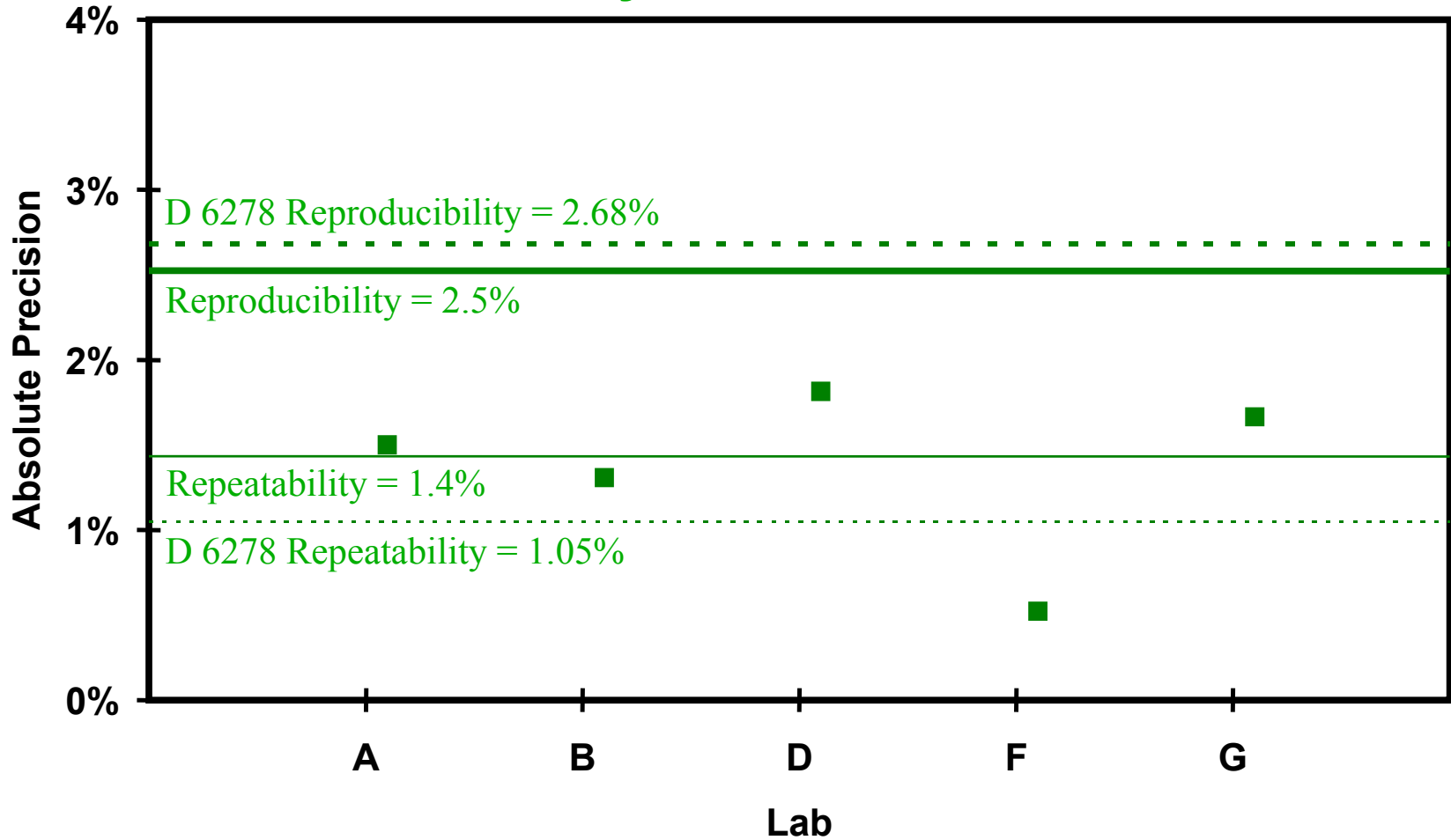
# Preliminary Precision (TMC 820-2)

## 90-Cycle Shear Loss



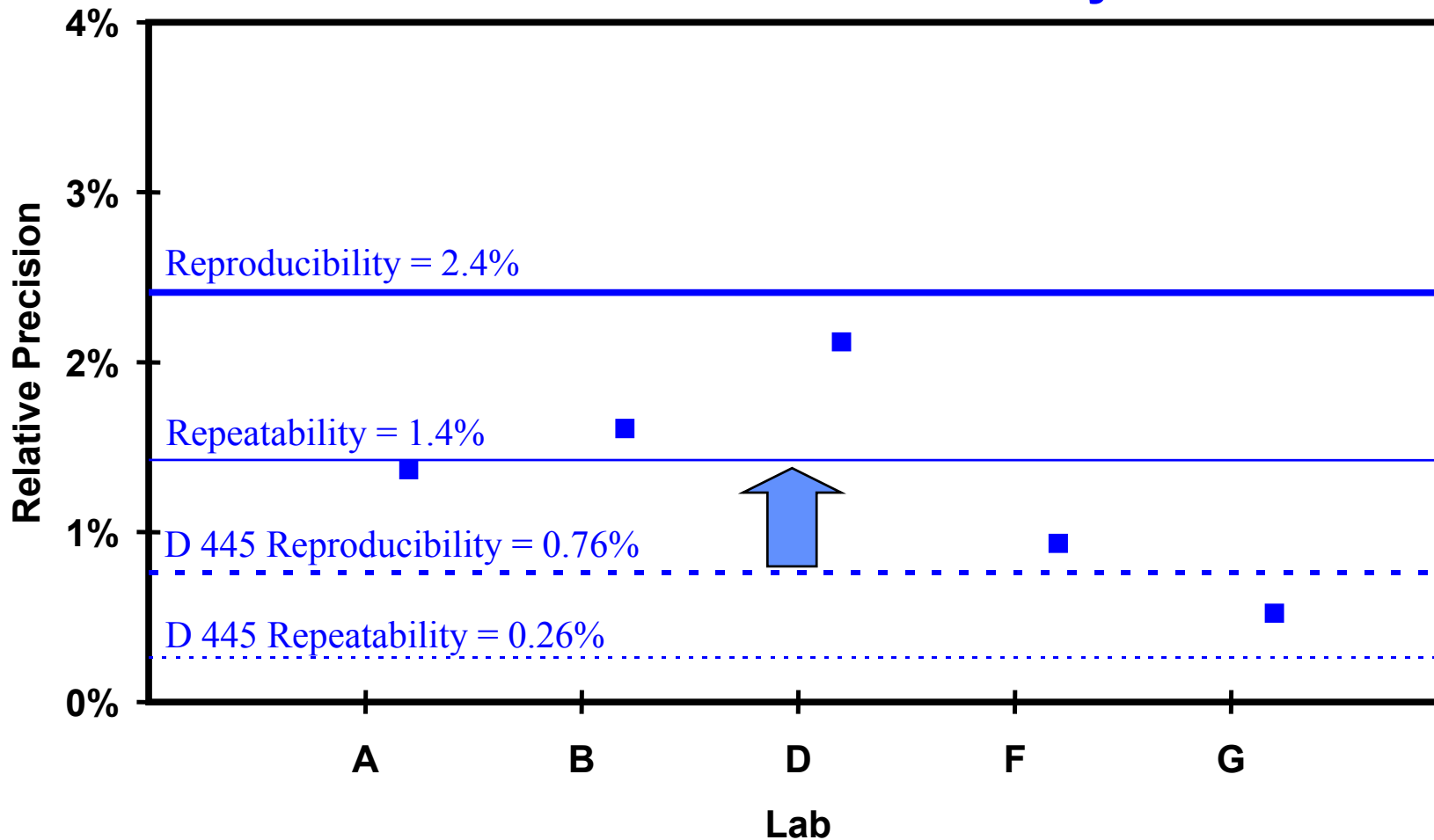
# Preliminary Precision (TMC 820-2)

## 30-Cycle Shear Loss



# Preliminary Precision (TMC 820-2)

## Initial Kinematic Viscosity



# CONCLUSIONS

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- ❖ **Expect to have an approved Test Method October 2004**
  - **Expedited ballot**
  - **Preliminary precision statement**
    - ⇒  $r = 1.8\%$  (*absolute*)
    - ⇒  $R = 2.9\%$  (*absolute*)
- ❖ **Expect to have a revised Test Method by April 2005**
  - **Approved Precision Statement**
    - ⇒ *Based on Full Round Robin*



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**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 Uejas	PDVSA		Jun 2004	

Respectfully submitted,

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 Section B, Subcommittee 7  
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## Work Item WK2880, Draft 5

Modification of D 6278 for 90-cycle shear stability with 30-cycle intermediate result

Draft 1 12 August 2003: Modification of D 6278 for 90-cycles only

Draft 2 25 November 2003: Modification of D 6278 for 30- and 90-cycles

Draft 3 7 April 2004: Clear identification of 30- and 90-cycle phases of shearing

Draft 4 21 May 2004: More consistent with Mack T-11 procedure

Draft 5 20 June 2004: Editorial changes and clarification of wording

### Standard Test Method for Shear Stability of Polymer Containing Fluids Using a European Diesel Injector Apparatus at 30 and 90 Cycles<sup>1</sup>

This standard is issued under the fixed designation D XXXX; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

#### 1. Scope

1.1 This test method covers the evaluation of the shear stability of polymer-containing fluids. The test method measures the viscosity loss, in  $\text{mm}^2/\text{s}$  and percent, at  $100^\circ\text{C}$  of polymer-containing fluids when evaluated by a diesel injector apparatus procedure that uses European diesel injector test equipment. The viscosity loss reflects polymer degradation due to shear at the nozzle. Viscosity loss is evaluated after both 30 and 90 cycles of shearing.

NOTE 1—This test method evaluates the shear stability of oils after both 30 and 90 cycles of shearing. In general, there is no correlation between results after 30 cycles and results after 90 cycles of shearing.

NOTE 2—Test Method D 6278 uses essentially the same procedure with 30 cycles only instead of both 30 and 90 cycles. The correlation between results from this test method at 30 cycles and results from test method D 6278 has not been established.

NOTE 3—Test Method D 2603 has been used for similar evaluation of shear stability; limitations are as indicated in the significance statement. No detailed attempt has been undertaken to correlate the results of this test method with those of the sonic shear test method.

NOTE 4—This test method uses test apparatus as defined in CEC L-14-A-93. This test method differs from CEC-L-14-A-93 in the period of time required for calibration.

NOTE 5—Test Method D 5275 also shears oils in a diesel injector apparatus but may give different results.

NOTE 6—This test method has different calibration and operational requirements than Test Method D 3945.

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<sup>1</sup>This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved (approval date). Published (publication date). Originally published as D XXXX–YY. Last previous edition D XXXX–ZZ.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in Section 8.

## 2. Referenced Documents

### 2.1 ASTM Standards:

- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)<sup>2</sup>
- D 2603 Test Method for Sonic Shear Stability of Polymer-Containing Oils<sup>2</sup>
- D 3945 Test Method for Shear Stability of Polymer-Containing Fluids Using a Diesel Injector Nozzle<sup>3</sup>
- D 5275 Test Method for Fuel Injector Shear Stability Test (FISST) for Polymer Containing Fluids<sup>3</sup>
- D 6278 Test Method for Shear Stability of Polymer Containing Fluids Using a European Diesel Injector Apparatus
- D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance<sup>4</sup>

### 2.2 Coordination European Council (CEC) Standard:

- CEC L-14-A-93 Evaluation of the Mechanical Shear Stability of Lubricating Oils Containing Polymers<sup>5</sup>

## 3. Terminology

### 3.1 Definitions:

- 3.1.1 *kinematic viscosity, n*—a measure of the resistance to flow of a fluid under gravity.

### 3.2 Definitions of Terms Specific to This Standard:

- 3.2.1 *calibration pressure, n*—the recorded gage pressure when calibration fluid RL 34 undergoes a viscosity loss of 2.75 to 2.85 mm<sup>2</sup>/s when the recorded gage pressure is within the range of 13.0 to 18.0 MPa (1885 to 2611 psi).
- 3.2.2 *viscosity loss, n*—the loss in viscosity determined from the difference in kinematic viscosity at 100°C of pre-sheared and post-sheared fluid.
- 3.2.3 *percent viscosity loss, n*—viscosity loss, as defined in 3.2.2, divided by the pre-sheared viscosity, and reported as a percent.

## 4. Summary of Test Method

4.1 A polymer-containing fluid is passed through a diesel injector nozzle at a shear rate that may reduce its kinematic viscosity. The percent viscosity loss is a measure of the mechanical shear stability of the fluid.

NOTE 7—This test method may also be used for oils not containing polymer. It might not be known whether an oil submitted for test contains a polymer.

## 5. Significance and Use

5.1 This test method evaluates the percent viscosity loss of fluids resulting from physical degradation in the high shear nozzle device. Thermal or oxidative effects are minimized.

5.2 This test method may be used for quality control purposes by manufacturers of polymeric lubricant additives and their customers.

5.3 This test method is not intended to predict viscosity loss in field service in different field equipment under widely varying operating conditions, which may cause lubricant viscosity to change due to thermal and oxidative changes as well as by the mechanical shearing of polymer. However, when the field service conditions, primarily or exclusively, result in the

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<sup>2</sup>Annual Book of ASTM Standards, Vol 05.01.

<sup>3</sup>Annual Book of ASTM Standards, Vol 05.02.

<sup>4</sup>Annual Book of ASTM Standards, Vol 05.03.

<sup>5</sup>Available from CEC Secretariat, Madou Plaza, 25<sup>th</sup> floor, Place Madou 1, B-1210 Brussels, Belgium.

degradation of polymer by mechanical shearing, there may be a correlation between the results from this test method and results from the field.

## 6. Apparatus

6.1 The apparatus consists of a fluid reservoir, a double-plunger pump with an electric motor drive, an atomization chamber with a diesel injector spray nozzle, and a fluid cooling vessel, installed in an area with an ambient temperature of 20 to 25°C (68 to 77°F). Figure A1.1 shows the schematic representation of equipment.

6.1.1 *Fluid Reservoir*, In Fig. A1.1, (7)<sup>6</sup> is open on the top, has approximately a 250 mL capacity, has a 45-mm (1.772-in.) inner diameter, and is calibrated in units of volume. It is fitted with an internal fluid distributor as detailed in Fig. A1.2. A 40-mm (1.575-in.) diameter watch glass with serrated edges is an acceptable distributor plate. The distributor reduces the tendency of fluid channeling. Temperature is measured by a thermometer suspended in the center of the fluid reservoir. The bottom of the thermometer bulb shall be 10 to 15 mm above the entrance to the drain tube opening. Other temperature-measuring equipment positioned at the same location may also be used. The outlet is equipped with a three-way stopcock (8). The three-way stopcock is of a cone type with a nonexchangeable solid plug with an 8-mm (0.315-in.) nominal bore size. Transparent plastic tubing, (10) in Fig. A1.1, is used to connect the three-way stopcock to the pump inlet.

6.1.2 *Double-Plunger Injection Pump*, In Fig. A1.1 (11) is defined as Bosch PE 2 A 90D 300/3 S2266. This pump is equipped with a stroke counter, (15), venting screw, (14), and a flow rate adjusting screw, (12).

6.1.3 *Injection Pump*, driven by a three-phase electric motor, (13) in Fig. A1.1., rated at a speed of  $925 \pm 25$  rpm.

6.1.3.1 This motor runs at 925 rpm on the 50 Hz current prevalent in Europe; it will run at approximately 1100 rpm on 60 Hz current. The 1100 rpm speed is not acceptable in this procedure. A suitable means shall be taken to ensure the prescribed  $925 \pm 25$  rpm speed to the injection pump. One acceptable method is to use a 6 to 5 speed reducer.

6.1.4 *Outlet of Injection Pump*, connected to the atomization chamber using high pressure steel tubing. The atomization chamber, (2) in Fig. A1.1, is defined in more detail in Fig. A1.3. To minimize foam generation, the spray chamber is designed so that the fluid under test exits from the nozzle into a chamber filled with the test fluid. A drain tube (17) fitted with a two-way stopcock is included to minimize contamination from the previous test during the system cleaning steps. The diesel injector nozzle is a Bosch DN 8 S 2-type pintle nozzle injector, number 0434 200 012, installed in a Bosch KD 43 SA 53/15 nozzle holder. The nozzle holder includes a filter cartridge.

NOTE 8—Take great care to avoid damage to the precision parts of the fuel injection equipment (the plunger and barrel in the pump and the nozzle valve assembly). Service work on the equipment should be performed by a diesel fuel injector pump specialist or with reference to the manufacturer's service manual.<sup>7</sup>

NOTE 9—An unusually rapid rise in gage pressure during testing may signify filter blockage. When this occurs, the filter cartridge shall be replaced.

6.1.5 A pressure sensing device (18), such as a glycerol-filled pressure gage or *electronic, digital display pressure indicator*, shall be installed and separated from the line by a pressure snubber or needle valve to suitably dampen pressure surges. The pressure device shall be occasionally pressure tested to ensure accuracy.

6.1.6 *Fluid Cooling Vessel*, ((5) in Fig. A1.1), used to maintain the specified temperature of the test fluid, as indicated at the outlet of the fluid reservoir. This vessel is a glass container with exterior cooling jacket constructed so that the heat transfer surface of the jacket is spherical. The exterior jacket diameter,  $d_1$ , is approximately 50 mm (1.969 in.). The interior heat transfer surface,  $d_2$ , is approximately 25 mm (0.984 in.) in diameter. The overall length,  $L$ , is approximately 180 mm (7.087 in.). A distributor plate, similar in design to the distributor plate in the fluid reservoir, is positioned in the upper portion of the fluid cooling vessel to ensure contact between the fluid and the cooling surface. The discharge from the fluid cooling vessel is through a three-way stopcock of the same design used on the discharge of the fluid reservoir. The exterior cooling jacket shall be supplied with an adjustable volume of cold water.

## 7. Materials

7.1 *Diesel Fuel (No. 2)*, initially required to adjust the diesel injector nozzle valve opening pressure.

<sup>6</sup>The number in parentheses refers to the legend in Fig. A1.1.

<sup>7</sup>Repair Instructions for Diesel Injection Pumps Size A, B, K and Z, Bulletin WJP 101/1 B EP, Robert Bosch GmbH, 2800 South 25th Ave., Broadview, IL 60153.

7.2 *Calibration Fluid RL 34*, used to ensure that when the apparatus is adjusted within a prescribed pressure range, the correct viscosity loss is obtained.

## 8. Hazards

8.1 **Warning**—Use a safety shield between the high-pressure components and the operator during use of equipment.

8.2 **Precaution**—During operation, the line between the pump and nozzle, ((16) in Fig. A1.1), is under a pressure of at least 13.0 MPa (130 bar, or 1,885 psi). Pressures above the upper limit of 18.0 MPa (180 bar or 2611 psi) are possible if filter plugging occurs. Shut off the pump prior to tightening any fitting that is not properly sealed.

## 9. Sampling

9.1 Approximately 650 mL of fluid is needed per test.

9.2 The test fluid shall be at room temperature, uniform in appearance, and free of any visible insoluble material prior to placing it in the test equipment.

9.3 Water and insolubles shall be removed before testing, or filter blocking and nozzle wear may occur. Filter blocking can be detected by a sudden change in gage pressure. The transport of insolubles to the shear zone will shorten nozzle life.

## 10. Calibration and Standardization

10.1 *Nozzle Adjustments*—If the nozzle to be used is new or has not been pre-calibrated, adjust the diesel injector nozzle holder with the nozzle in place. Adjust the nozzle using diesel fuel and a nozzle tester so that the valve opening pressure is 13.0 MPa (1885 psi) under static conditions. If the nozzle has been pre-calibrated with RL34 calibration oil, adjust the valve opening pressure to the calibration pressure prescribed, which must be between 13.0 MPa (1885 psi) and 18.0 MPa (2611 psi).

10.1.1 Install the nozzle and the nozzle holder in the test apparatus. The pintle/spray nozzle shall be tightly fitted in the chamber to avoid leakage of oil around the external surface of the spray nozzle.

10.2 *Measurement of Residual Undrained Volume,  $V_{res}$* :

10.2.1 The residual undrained oil volume of the system is the volume of the system between the three-way stopcock below the fluid reservoir, (8) in Fig. A1.1, and the injector nozzle orifice, (1).  $V_{res}$  does not include the atomization chamber volume. When the residual undrained volume is known, go to 10.3.

10.2.2 To determine residual undrained volume, first remove as much fluid as possible by briefly running the pump.

10.2.3 Remove the high-pressure lines, (16) in Fig. A1.1, and drain. Remove the plug at the end of the pump gallery to drain the remaining oil in the pump. Drain atomization chamber (2).

10.2.4 Reassemble the system and close all drains. The upper three-way stopcock (6) shall be open to the lower reservoir (7) and the lower three-way cock (8) shall be open to the pump suction (10).

10.2.5 Add 170 mL of RL34 calibration oil to the lower reservoir (7) and observe the level. Start the pump and run for several minutes until the oil is transparent and free of suspended air.

10.2.6 Stop the pump. Drain the fluid in the atomization chamber into a beaker and then pour the fluid back into the lower reservoir; draining to waste will result in an error in the measurement of  $V_{res}$ . Allow the system to drain for 20 min and free air trapped in the transparent connecting tube between the lower reservoir and pump.

10.2.7 Observe the difference in oil level in the lower reservoir compared to that noted in 10.2.5. Record this difference as the residual volume,  $V_{res}$ .

NOTE 10—Undrained residual volumes of 15 to 30 mL have been reported by various users of this test.  $V_{res}$  measurements in excess of this may occur when fluid in the atomization chamber is not poured back into the lower reservoir as in 10.2.6, or if the length of line (10) is excessive.

10.2.8 Calculate the run volume,  $V_{run}$ , which is the difference between 170 mL and  $V_{res}$ ,  $V_{run} = 170 - V_{res}$ .

10.3 *Cleaning the Apparatus, Setting the Stroke Counter, and Adjusting the Pump Stroke*:

10.3.1 Drain residual oil by way of drain line (17) from the atomization chamber into a waste container. Drain fluid in the cooling jacket by means of stopcock (6) (Fig. A1.1) and the fluid reservoir by means of stopcock (8), into suitable waste containers.

10.3.2 After fluid has drained, leave the stopcock on the drain line to the atomization chamber open and the three-way stopcock (6) positioned so that fluid in the cooling jacket drains to a waste container. Position stopcock (8) so that the drain is closed but the fluid reservoir is open to pump suction through line (10). Add a minimum of 50 mL of RL34 to the fluid reservoir.



NOTE 11—Steps 10.3.2 to 10.3.7 are representative of the first and second purges with 50 mL fluid that are needed to remove used oil from the apparatus prior to calibration and testing. For these steps, the stopcock below the atomization chamber and cooling jackets are set so that oil will flow into waste containers.

10.3.3 Free the apparatus of air in the line by use of the venting screw, (14), and by manual compression of the transparent flexible tube that connects the pump to the fluid reservoir.

10.3.4 Set the stroke counter so that the pump will run a sufficient length of time to evacuate the fluid out of the fluid reservoir.

10.3.5 Start the pump. Observe the fluid level in the reservoir and stop the pump when all the fluid is out of the base of the reservoir but is still fully-retained in line (10).

10.3.6 Add a minimum of 50 mL of RL34 fluid to the fluid reservoir a second time and operate the pump until the fluid reservoir is empty but line (10) is still filled with fluid.

10.3.7 After all oil has drained, close the stopcock on the atomization chamber drain line (17), position stopcock (6) so that fluid will flow from the cooling jacket into the fluid reservoir.

10.3.8 Remove the thermometer or temperature probe from the fluid reservoir.

NOTE 12—The thermometer and assembly can interfere with the obtainment of accurate volume measurements in the fluid reservoir, hence its removal is called for when the accurate determination of fluid volume is needed. A thermocouple or thermistor probe is a suitable alternative to a thermometer.

10.3.9 Add a minimum amount of fluid equal to the sum of 30 mL plus  $V_{run}$ , determined in 10.2.8, to the fluid reservoir.

10.3.10 Close the stopcock below the atomization chamber drain line (17) and position stopcock (6) so that the fluid will drain from the cooling jacket into the fluid reservoir.

NOTE 13—The atomization chamber drain line is always closed for the third cleaning run and all test runs.

10.3.11 Free the apparatus of air in the line by manual compression of the flexible tube (10) that connects the pump to the fluid reservoir. The venting screw, (14), is also used for this purpose.

10.3.12 Record the number on the stroke counter.

10.3.13 Use a stopwatch or other timing device and run the pump for  $1 \text{ min} \pm 1 \text{ s}$ .

10.3.14 Determine  $n$ , the difference in the stroke count from 10.3.12.  $n$  is the number of strokes per minute.

10.3.15 Set the stroke counter shutoff to the product of three times  $n$ . The pump shall run for  $3 \pm x \text{ min}$ . Obtain a timing device to observe the time the stroke counter is on to ensure  $n$  is correct. Start the pump and allow oil to circulate until the impulse counter shuts down the instrument.

10.3.16 When all fluid has drained, adjust the volume of oil in the fluid reservoir so that the volume is equal to  $V_{run}$ .

10.3.17 Set the impulse counter to  $0.5 (n)$ .

10.3.18 Close stopcock (6) so that fluid will be stored in the cooling jacket after the pump is started.

10.3.19 Start the pump. When the pump stops and draining is complete, subtract the volume now in the fluid reservoir from  $V_{run}$ .

10.3.20 If the difference is within  $\pm 2.5 \text{ mL}$  of  $\frac{1}{2}$  of the total volume ( $V_{tot} = V_{run} + V_{res}$ ), proceed to 10.4.

10.3.21 When the volume in the fluid reservoir is not within  $\pm 2.5 \text{ mL}$  of  $V_{tot}$ , drain the fluid from the cooling jacket back into the fluid reservoir, adjust the pump stroke by means of the pump adjustment screw (12), and repeat steps beginning with 10.3.16.

10.4 *Warm-up*—A half-hour warm up period is required before proceeding to calibrate with RL34. Set the stroke counter shut-off to 30 times  $n$  strokes, and start the pump.

NOTE 14—This warm up period is only required for the first within-day calibration.

10.5 *Removal of Fluid*—Open the stopcock below the atomization chamber and drain to waste. Drain the fluid from the cooling jacket into a waste container. Position stopcock (8) so that all fluid in the fluid reservoir is removed to a waste container. When drainage is complete, position stopcock (8) so that the drain is closed and the pump inlet line (10) is open.

10.6 Calibration with RL34:

10.6.1 Ensure that the ambient (room) temperature is between 20 and 25°C.

10.6.2 Add a minimum of 50 mL of RL34 to the fluid reservoir. Position the three-way stopcock, (6) in Fig. A1.1, below the cooling vessel to discharge fluid into a suitable waste container and leave the stopcock open below the atomization chamber. Operate the pump until the fluid reservoir is empty but line (10) is still filled with fluid.

10.6.3 Free the apparatus of air in the line by manual compression of the flexible tube that connects the pump to the fluid reservoir. When necessary, venting screw (14) is also used for this purpose.

10.6.4 Add a minimum of 50 mL of test fluid to the fluid reservoir a second time and operate the pump until the fluid reservoir is empty again but line (10) is full.

10.6.5 Close the stopcock below the atomization chamber, position the stopcock below the fluid reservoir so that the line to the pump is open, and retain the position of the stopcock below the cooling jacket so that the first 50 mL of RL34 can be drained into a waste container.

10.6.6 Place a volume of RL34 in the fluid reservoir equal to  $V_{run}$  plus 30 mL.

10.6.7 Start the pump, and stop the pump when there is a 50 mL drop of fluid in the fluid reservoir. After draining is complete, re-position the stopcock below the cooling jacket so subsequent fluid flows directly into the fluid reservoir.

10.6.8 Set the stroke counter for automatic shutoff at the required number of impulses (30 multiplied by  $n$  impulses per minute). The flow rate will be 170 mL/min as set in 10.3.

10.6.9 Adjust, if necessary, the volume of fluid in the fluid reservoir to  $V_{run}$ .

10.6.10 Place the temperature measuring device in the fluid reservoir, and start the pump.

10.6.11 After about 10 mins of operation, adjust the water flow to control the fluid temperature at 30 to 35° C, as measured at the discharge point of the fluid reservoir. Approximately 10 mins of operation will be required before the temperature can be stabilized.

10.6.12 At approximately ten cycles of operation, record the gage pressure reading to the nearest 0.1 MPa (15 psi), when a glycerol-filled pressure gage is being used, or to 0.01 MPa (1.5 psi), when an electronic pressure device is employed.

10.6.12.1 The pressure measurement device must occasionally be pressure tested to ensure accuracy.

10.6.13 After 30 cycles has elapsed and the pump has stopped, open the stopcock below the atomization chamber and drain fluid into a waste container. Open the three-way stopcock below the fluid reservoir and discharge the first 10 to 15 mL as waste in order to flush out the drain line. Discharge the remaining fluid into a clean sample container. After the fluid has drained, close the three-way stopcock.

10.6.14 Remove the thermometer or temperature probe.

10.6.15 Using Test Method D 445, determine the kinematic viscosity at 100° C of unsheared (untested) RL 34, as well as the sheared fluid from 10.6.13. Use the same viscometer tube for the measurement of each oil.

10.6.16 Calculate viscosity loss ( $V_L$ ) as follows:

$$V_L = V_u - V_s \tag{1}$$

where

$V_u$  = kinematic viscosity of unsheared oil at 100°C, mm<sup>2</sup>/s, and  
 $V_s$  = kinematic viscosity of sheared oil at 100°C, mm<sup>2</sup>/s

10.6.17  $V_L$  for RL34 shall be within the range of 2.75 to 2.85 mm<sup>2</sup>/s at 100°C at a gage pressure reading between 13.0 and 18.0 MPa, as recorded after 10 min of test time. When this is achieved, the gage pressure recorded in 10.6.12 shall subsequently be referred to as the calibration pressure.

10.6.18 If  $V_L$  is less than 2.75 mm<sup>2</sup>/s, increase the gage pressure. If  $V_L$  is greater than 2.85 mm<sup>2</sup>/s, reduce the gage pressure, provided that the gage pressure recorded in 10.6.12 is greater than 13.0 MPa and less than 18.0 MPa. To alter the pressure, remove the dust cover of the spray nozzle holder (see Fig. A1.4), loosen the locking nut, and turn the adjustment screw that regulates valve opening pressure. Then, tighten the locking nut and replace the dust cover. The nozzle and nozzle holder need not be removed from the apparatus. Continue to retest RL 34 and make adjustments until calibration is achieved.

NOTE 15—It is extremely important that the locking nut be completely tightened. When it is not, some leakage of fluid around the outside of the nozzle assembly may occur. This may result in a reduction of mechanical shearing for some oils, which can adversely influence precision. This condition can be monitored by use of a recorder and an electronic pressure measurement device. Leakage results in a sudden drop in pressure when fluid by-passes the nozzle orifice.

10.6.19 When  $V_L$  is greater than 2.85 mm<sup>2</sup>/s at a gage pressure of only 13.0 MPa, pre-condition the nozzle by substitution of a fully-formulated engine lubricant as the test fluid. The stroke counter shut-off shall be adjusted so that the test time is at least 8 h, instead of 30 min. Then retest RL34, beginning with 10.5. Continue to pre-condition and evaluate new nozzles until the calibration requirement of 10.6.17 is achieved.

NOTE 16—Suitable *break-in* oils include, but are not limited to, fully-formulated SAE 15W-40 heavy-duty engine lubricants.

10.6.20 When viscosity decrease is below 2.75 mm<sup>2</sup>/s at a gage pressure of 18.0 MPa, another nozzle shall be installed and the calibration procedure shall be repeated.

NOTE 17—Before calibration with a new nozzle, it is advisable to subject the nozzle to at least a 4 h run-in with break-in oil.

#### 10.7 Calibration Period:

10.7.1 *Calibration with RL34 Fluid*—Frequent testing of the apparatus with the calibration oil is recommended. The apparatus shall be recalibrated after 450 cycles.

NOTE 18—It has been found that calibration no less frequently than every 450 shear cycles improves the precision of test methods using this apparatus.<sup>8</sup>

10.7.2 *Calibration with RL34 and Monitoring System Stability and Precision with a Quality Control Oil per Practice D 6299*—A quality control oil can be used to monitor calibration once the nozzle has been calibrated with RL34 fluid. This Quality Control fluid shall have a new oil kinematic viscosity at 100°C of between 14.0–17.0 mm<sup>2</sup>/s and after test kinematic viscosity decrease at 100°C of between 2.0 and 3.0 mm<sup>2</sup>/s. The base oil for this fluid shall have a kinematic viscosity of between 4.0–8.0 mm<sup>2</sup>/s at 100°C. The calibration procedure is as follows:

10.7.2.1 Calibrate with RL34.

10.7.2.2 Monitor stability and precision of the system through QC sample testing per Practice D 6299, paragraph 7.1. This will initially require 15 control samples to develop a control chart.

10.7.2.3 The quality control oil shall be run on the same day that a test fluid is evaluated.

10.7.2.4 Any deviation or trend indicated in the control chart shall call for a recheck with RL34 fluid. A recheck with RL34 shall be done after 7 days even if no recheck has been required.

—

Note 19 – Calibration of the apparatus for this test method is identical to that for Test Method D 6278, and is valid for either method.

## 11. Procedure

11.1 *Flow Rate Adjustment for Test Oil*—Open the stopcock on the atomization chamber and drain any previous fluid out of the chamber. Position the three-way stopcock ((6) in Fig. A1.1) below the cooling jacket to discharge fluid into a suitable waste container. Then, position stopcock (8) so that the drain line is closed but line (10) is open from the fluid reservoir to the pump.

11.1.1 Add a minimum of 50 mL of test fluid to the fluid reservoir.

11.1.2 Free the apparatus of air in the line by manual compression of the flexible tube that connects the pump to the fluid reservoir. When necessary, the venting screw, (14), is also used for this purpose.

11.1.3 Operate the pump until the fluid reservoir is empty, but line (10) is full.

11.1.4 Add a minimum of 50 mL of test fluid to the fluid reservoir a second time and operate the pump until the fluid reservoir is empty again but line (10) is still full.

11.1.5 After draining is complete, close the stopcock on the atomization chamber and position stopcock (6) so that fluid will flow from the cooling jacket into the fluid reservoir.

<sup>8</sup> Rhodes, R. E., “Diesel Injector Shear Stability of Engine Oil – Factors Affecting Reproducibility and Relevance to Engine Performance,” SAE 922193, Society of Automotive Engineers, 1992,

11.1.6 Add an amount of test fluid to the fluid reservoir equal to the sum of 30 mL plus  $V_{run}$ .

11.1.7 Free the apparatus of air in the line by use of the venting screw, (14), and by manual compression of the flexible tube that connects the pump to the fluid reservoir.

11.1.8 Set the stroke counter to the product of three times  $n$ , and start the pump and allow oil to circulate until the impulse counter shuts down the instrument.

11.1.9 Adjust the oil level in the fluid reservoir to  $V_{run}$  by draining any excess oil to a waste container, or adding oil when needed.

11.1.10 Set the impulse counter to the product of 0.5 times  $n$ .

11.1.11 Close stopcock (6) so that fluid will be stored in the cooling jacket after the pump is started.

11.1.12 Start the pump. When the pump stops, subtract the volume now in the fluid reservoir, (7), from  $V_{run}$ .

11.1.13 If the difference is within  $\pm 2.5$  mL of  $\frac{1}{2}$  of the total volume, ( $V_{tot} = V_{run} + V_{res}$ ), proceed to 11.1.15.

11.1.14 When the volume in the fluid reservoir is not within  $\pm 2.5$  mL of  $\frac{1}{2} V_{tot}$ , adjust the pump stroke slightly by means of the pump adjustment screw, (12), drain the fluid from the cooling jacket into the fluid reservoir, and repeat steps beginning with 11.1.6.

11.1.15 Calculate  $n_1$ , the number of pulses strokes required to circulate 200 mL of test oil ~~one (one cycle)~~ for one minute by the following equation:

$$n_1 = \left[ \frac{(170 + 30)}{170} \right] * n = 1.176n \quad (2)$$

11.2 *Removal of Fluid*— Leave stopcock below atomization chamber closed. Drain the fluid from the cooling jacket into a waste container then re-position the stopcock so that the fluid will flow into the fluid reservoir. Then open the three-way stopcock below the fluid reservoir to discharge fluid into a waste container.

11.2.1 *Test Oil Evaluation*—Re-position stopcock (8) so that line (10) is open. Leave the stopcock below the atomization chamber closed. Re-position stopcock (6) below the cooling jacket so that the first 50 mL of test oil is sent to a waste container.

11.2.2 Place a volume of test oil in the fluid reservoir equal to  $V_{run}$  plus 60 mL. The total volume should be approximately 200 mL.

11.2.3 Free the apparatus of air in the line by manual compression of the flexible tube that connects the pump to the fluid reservoir. When necessary, the venting screw, (14), is also used for this purpose.

11.2.4 Start the pump, and stop the pump when there is a 50 mL drop of fluid in the fluid reservoir. When draining is complete, re-position the stopcock below the cooling jacket so subsequent fluid flows directly into the fluid reservoir.

11.2.5 Set the stroke counter for automatic shutoff at the required number of impulses (30 multiplied by  $n_1$ ). This will correct for the additional volume to attain a 30 cycle intermediate result.

11.2.6 When necessary, adjust the volume in the fluid reservoir to ( $V_{run} + 30$ ) mL.

11.2.7 Insert the thermometer assembly or temperature probe in the fluid reservoir.

11.2.8 Start the pump for the 30 cycle phase of shearing.

11.2.9 Within the first 10 mins, adjust the water flow to control the fluid temperature at 30 to 35° C, as measured at the discharge point of the fluid reservoir.

NOTE 20—It is not necessary to record the gage pressure reading here, which may differ from the previously recorded calibration pressure.

11.2.10 After 30 cycles have elapsed and the pump has stopped, open the three-way stopcock below the fluid reservoir and discharge the first 10 to 15 mL as waste in order to flush out the drain line. Remove the thermometer assembly or probe from the fluid in the reservoir to obtain an accurate volume measurement (hold above fluid for 1 min to allow drainage into the reservoir). Discharge 15 to 20 mL of the remaining fluid into a clean sample container until the reservoir has  $V_{run}$  mL remaining. This is the 30-cycle sheared sample. Save the 30-cycle sheared sample for further testing.

11.2.11 Replace the thermometer assembly or probe in the fluid reservoir.

11.2.12 Set the stroke counter for automatic shutoff at the required number of pulses (60 times  $n$ ) to achieve a total of 90 cycles on the remaining material.

11.2.13 Restart the pump for the remaining 60 cycles of the 90 cycle test.

11.2.14 Within the first 10 min, adjust the water flow to control the fluid temperature at 30 to 35°C, as measured at the discharge point of the fluid reservoir.

11.2.15 After the additional 60 cycles have elapsed and the pump has stopped, open the stopcock below the atomization chamber and drain fluid into a waste container. Open the three-way stopcock below the fluid reservoir and discharge the first 10 to 15 mL as waste in order to flush out the drain line. Discharge the remaining fluid into a clean sample container. This is the 90-cycle sheared sample. Remove the thermometer assembly or probe.

11.2.16 Using Test Method D 445, determine the kinematic viscosity at 100°C of unsheared (untested) test oil, as well as the 30-cycle sheared sample (from 11.2.10) and the 90-cycle sheared sample (from 11.2.15). Use the same viscometer tube for the measurement of each sample.

## 12. Calculation

12.1 Calculate the percent viscosity loss (PVL) of the sheared oil samples as follows:

$$PVL_{30} = 100 \left[ \frac{(V_u - V_{30})}{V_u} \right] \quad (3)$$

$$PVL_{90} = 100 \left[ \frac{(V_u - V_{90})}{V_u} \right] \quad (4)$$

where

$V_u$  = kinematic viscosity of unsheared oil at 100°C, mm<sup>2</sup>/s,

$V_{30}$  = kinematic viscosity of the 30-cycle sheared oil sample at 100°C, mm<sup>2</sup>/s, and

$V_{90}$  = kinematic viscosity of the 90-cycle sheared oil sample at 100°C, mm<sup>2</sup>/s, and

## 13. Report

13.1 Report the following information:

13.1.1 The calibration pressure, in MPa.

13.1.2 Kinematic viscosity of the unsheared oil at 100°C.

13.1.3 Kinematic viscosity of the 30-cycle sheared oil at 100°C.

13.1.4 Kinematic viscosity of the 90-cycle sheared oil at 100°C.

13.1.5 Percent viscosity loss of the 30-cycle sheared oil (PVL<sub>30</sub>) as calculated in 12.1.

13.1.6 Percent viscosity loss of the 90-cycle sheared oil (PVL<sub>90</sub>) as calculated in 12.1.

## 14. Precision and Bias

14.1 The precision of this test method is currently being determined through an interlaboratory cooperative study conforming to ASTM E691 or equivalent.

14.2 A preliminary precision statement for this test method as determined by the statistical examination of limited interlaboratory test results is as follows:<sup>9</sup>

14.2.1 *Repeatability*— The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, and in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

PVL <sub>30</sub>	1.4%
PVL <sub>90</sub>	1.8%

<sup>9</sup>Supporting data are available from ASTM International Headquarters. Request RR:D02-ZZZZ.

14.2.2 *Reproducibility*— The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material would, in the long run, and in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

PVL <sub>30</sub>	2.5%
PVL <sub>90</sub>	2.9%

NOTE 21—The indicated repeatability and reproducibility values for PVL<sub>30</sub> and PVL<sub>90</sub> represent the arithmetic difference between the reported percent kinematic viscosity loss values for the two results being compared.

14.3 *Bias*—All test results are relative to those of the calibration fluid. Therefore, no estimate of bias can be justified.

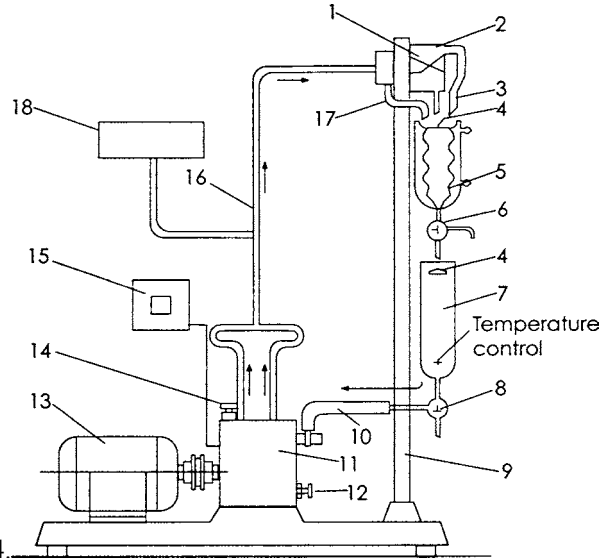
14.4 The preliminary precision and bias statement was determined through statistical examination of 49 results from five laboratories on a single SAE 15W-40 industry reference oil, TMC 820-2.

**15. Keywords**

15.1 diesel injector apparatus; mechanical shear stability; polymer containing fluid; viscosity loss

ANNEX  
(Mandatory Information)

A1. EQUIPMENT



A1.1 The equipment is presented in Figs. A1.1–A1.4.

NOTE 1—Legend

- (1) Spray Nozzle
- (2) Atomization chamber
- (3) Outlet of the atomization chamber
- (4) Distributor plate
- (5) Glass container fluid reservoir
- (6) Three-way cock downstream of glass
- (7) Glass container fluid reservoir
- (8) Three-way cock downstream of glass container
- (9) Support column
- (10) Connection with pump-suction opening
- (11) Double-plunger injection pump
- (12) Pump setting screw
- (13) Electric motor
- (14) Venting screw/pump
- (15) Stroke counter
- (16) Pressure tubing from pump to injector
- (17) Return line for overflowing liquid
- (18) Pressure sensing device

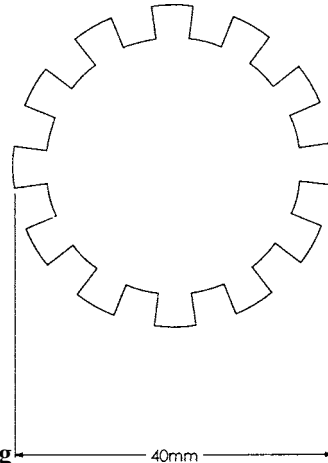


FIG. A1.1 Apparatus for Shear Stability Testing

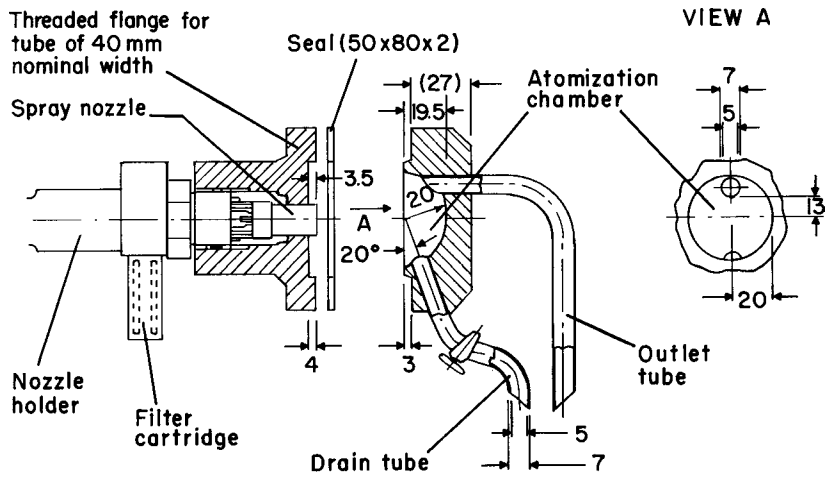


FIG. A1.2 Distributor Plate Dimensions in mm

FIG. A1.3 Atomization Chamber with Spray Nozzle and Nozzle

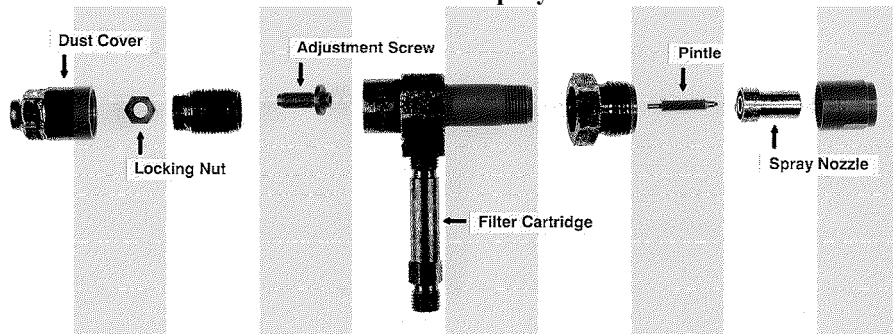


FIG. A1.4 Spray Nozzle and Nozzle Holder



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# PC-10 Chemical Limits Task Force

Report to HDEOCP

June 22, 2004

R. F. Finn, Jr.

# Status Update

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- ☑ Consensus was reached during May, 2004 on a set of Chemical Limits for the API Proposed Category PC-10:
    - ♠ Sulfated Ash (D874) – 1.0% max
    - ♠ Phosphorus (D4951) – 0.12% max
    - ♠ Sulfur (D4951 or D2622) – 0.4% max
  - ☑ The Task Force was unanimous in this conclusion
  - ☑ EMA member companies agreed they will design their engines and after-treatment systems around lubricants meeting the agreed chemical limits and will support them (including the 0.4% sulfur maximum) for their 2007-2009 MY engines unless they encounter ‘catastrophic problems’. ‘Catastrophic problems’ are defined as the inability to meet 2007 emissions standards despite good faith efforts to resolve any issues with after-treatment technology changes before requesting any lubricant changes
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# Status Update

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- ☑ Exit Criteria Ballot by HDEOCP closed 6/15/04
    - ♠ 24 positives and 7 negatives
  - ☑ Negatives encompass
    - ♠ SASH method (ASTM D874) not adequate (need to improve method, select a different method or make it a critical limit)
    - ♠ Sulfur limit too high
    - ♠ Phosphorus limit too high
    - ♠ Wait to set limits until later when more data is available ?  
Sulfur and Phosphorus limits too high
  - ☑ A number of comments included in positive ballots reinforced the SASH issue
- 
-

# Resolution of Negative Ballots

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- ☑ One negative on ‘Phosphorus limit set too high’ was withdrawn after discussion
- ☑ Four negatives on ‘SASH test inadequate’ tentatively resolved – without apparent new negatives - by establishing Multiple Test Acceptance Criteria for SASH. Task Force vote of 14 for, 0 against, 1 abstain included positive votes by all four Exit Criteria ballot negatives
  - ♠ SASH limit of 1.0% max. by D874
  - ♠ Non critical limit
  - ♠ MTAC established (exact limits to be determined with statistical help, but in concept:)
    - ☞ One Test – 0.96
    - ☞ Two Test – 1.00
    - ☞ Three Test – 1.05
  - ♠ Recommendation for an ASTM task group to work on D874 to improve precision

# Resolution of Negative Ballots

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☑ Two negatives lack 100% consensus

♠ Sulfur limit too high (Detroit Diesel)

☞ Need to balance against a number of comments on positive ballots that stated concern that the agreed limit was not supported by compelling data, or forewarned opposition if the limit was reduced

♠ Wait to set limits until later when more data is available ? Sulfur and Phosphorus limits too high (Caterpillar)

☞ As above

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# Next Steps

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- ☑ Resolve the two remaining negatives and confirm ongoing support for the SASH MTAC proposal once we have the details
  - ♠ Target Timing: Ideally today, but within next 30 days latest

or

- ☑ Acknowledge that we have achieved a consensus recommendation in the Chemical Limits Task Force (May) and defer the resolution of the Exit Criteria Ballot negatives to HDEOCP

Basis: one oil drain interval at 15,000 miles

Engine Operating on 13ppm S fuel at 5.8 miles/gal fuel economy

Total fuel consumed 2586.2 gallons  
 15,000 miles/5.8

Sulfur from fuel 0.236 lbs  
 2586 gallons x 7.02 lbs/gal x 13ppm

Sulfur from Oil

Total Oil consumed 7.5 quarts  
 15,000 miles/2000 miles/qt

lbs oil Consumed 13.5 lbs  
 7.5 quarts x 7.2 lbs/gal/4 quarts/gal

**Sulfur in lube at 0.4 mass% S**

13.5 lbs \* 0.4% 0.054 lbs

Total Sulfur from lube and fuel 0.290 lbs

% of sulfur from lube @ 0.4% S max in lube 18.6%

**Sulfur in lube at 0.3 mass %S**

13.5 lbs x 0.3% 0.041 lbs

Total Sulfur from lube and fuel 0.277 lbs

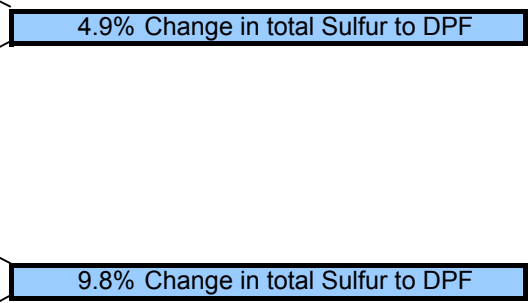
% of sulfur from lube @ 0.3% max in lube 14.6%

**Sulfur in lube at 0.5 mass %S**

13.5 lbs x 0.5% 0.0675 lbs

Total Sulfur from lube and fuel 0.304 lbs

% of sulfur from lube @ 0.3% max in lube 22.2%





# Multiple Test Acceptance Criteria for PC-10 Chemical Limits

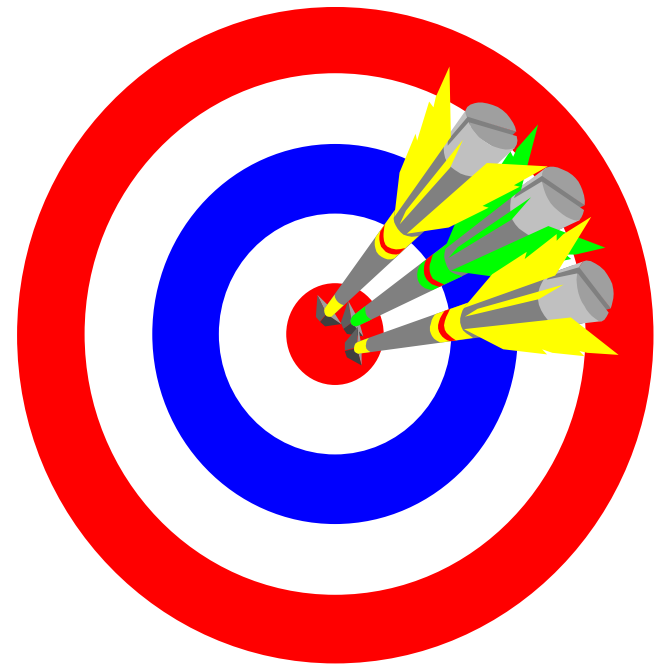
Presented to the ASTM  
Heavy Duty Engine Oil  
Classification Panel  
6/22/2004 Salt Lake City

Prepared by Jim Rutherford

# Multiple Test Acceptance Criteria - MTAC

**MTAC** is any data based approach for evaluation of the quality and performance of a formulation where more than one test may be run.

The method selected should recognize the precision of the test and the statistical reality that confidence in results increases as the number of tests increases.



**Designation: D 3244 – 97 (Reapproved 2002)**  
**Standard Practice for**  
**Utilization of Test Data to Determine Conformance with**  
**Specifications<sup>1</sup>**

- “7.2.1 Some specifications, because of the product characteristic or the end use of the product, or both, require that the receiver have a high degree of assurance that the product actually meets or exceeds the quality level indicated by the specification value. For the purpose of this practice, such specifications are called *critical* specifications.
- 7.2.2 Specifications that require assurance only that the product quality is not substantially poorer than is indicated by the specification level are called *noncritical* specifications for the purposes of this practice.”

# D3244 Type Limits

<u>One test limits</u>	<u>Non-critical acceptance limit</u>	<u>Critical acceptance limit</u>
Sulfated Ash (D874) – 1.0% Max.	1.08	0.92
Phosphorus (D4951) – 0.12% Max.	0.127	0.113
Sulfur (D4951) – 0.4% Max.	0.44	0.36
Sulfur (D2622) – 0.4% Max.	0.42	0.38
<u>Two test limits</u>	<u>Non-critical acceptance limit</u>	<u>Critical acceptance limit</u>
Sulfated Ash (D874) – 1.0% Max.	1.06	0.94
Phosphorus (D4951) – 0.12% Max.	0.125	0.115
Sulfur (D4951) – 0.4% Max.	0.43	0.37
Sulfur (D2622) – 0.4% Max.	0.42	0.38
<u>Three test limits</u>	<u>Non-critical acceptance limit</u>	<u>Critical acceptance limit</u>
Sulfated Ash (D874) – 1.0% Max.	1.05	0.95
Phosphorus (D4951) – 0.12% Max.	0.124	0.116
Sulfur (D4951) – 0.4% Max.	0.42	0.38
Sulfur (D2622) – 0.4% Max.	0.41	0.39

# Traditional HD Tiered Limits

Tiered pass/fail limits reflect increased confidence in multiple test results

Parameter	1 Test Limit	2 Test Limit	3 Test Limit
Wear	a	a + b	a + b + c
Rating	a	a - b	a - b - c

A one tailed confidence interval is used to give less than a 5% chance that an oil to be excluded will pass the test

# Traditional HD Tiered Limits

If the 2 test pass limit for sulfated ash is set at 1.0 and the standard deviation is 0.05 (0.142/2.8):

$$\begin{aligned} 1.0 &= \text{Excluded mean} - 0.05 * 1.645 / \sqrt{2} \\ & \quad [ \text{Excluded mean} \\ &= 1.0 + 0.05 * 1.645 / \sqrt{2} = 1.06 \end{aligned}$$

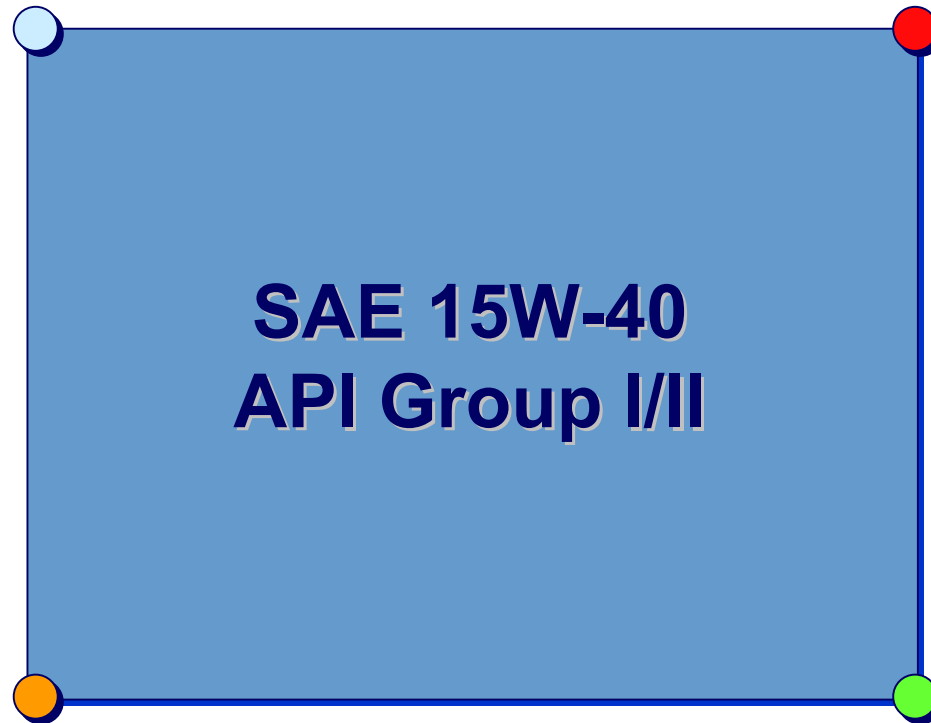
$$1 \text{ test limit} = 1.06 - 0.05 * 1.645 / \sqrt{1} = 0.98$$

$$3 \text{ test limit} = 1.06 - 0.05 * 1.645 / \sqrt{3} = 1.01$$

# Chemical Box for PC-10 for 2007

**1.00% Max Ash**

**0.4% Max Sulfur**



**13% Volatility**

**0.12% Max Phosphorus**



# Mack PC10 Engine Test Update

ASTM HDEOCP

June 22 nd 2004





# Ring & Liner Wear (Corrosive), Bearing Corrosion / Oxidation / Oil Consumption

- Mack T-12
- Based on Mack T10 & Mack T11
- With ULSD Fuel ??
- Length - ~ 300 Hours
- Two Phase Test
- 260 F Oil Temp
- Increased EGR Flow (Heavy EGR)  
(35% Phase 1 – 20% Phase 2)
- Precision Matrix Required
- Hardware Available 3rd QTR
- Test Procedure & Discrimination 4<sup>th</sup> QTR 04

# ISB Cam and Tappet Test Industry Report Packet



Warren Totten  
July 2004



# Test History

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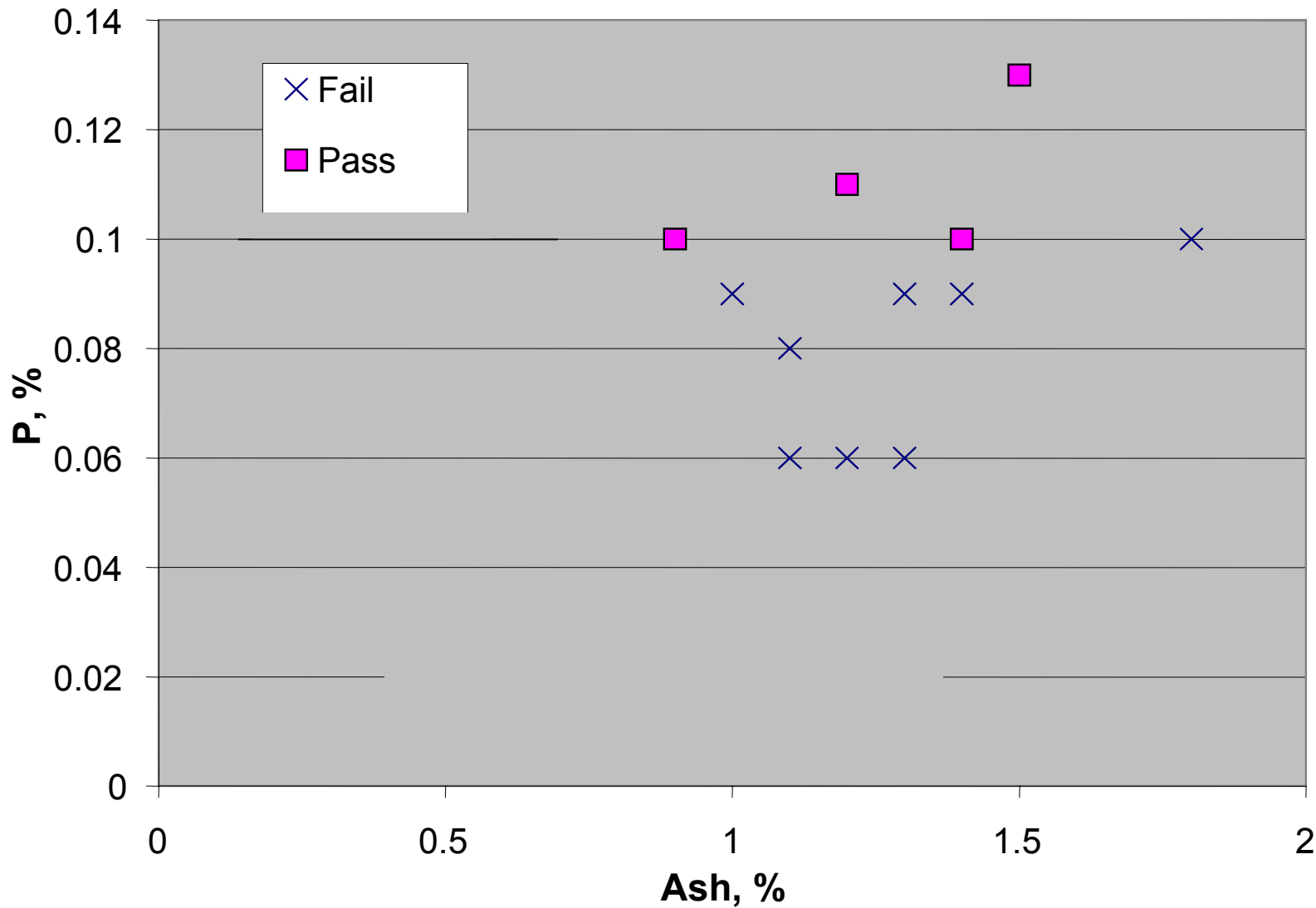
The ISB Valvetrain Wear Test was developed and based upon a Cummins, internal accelerated camshaft and tappet test used to evaluate the performance of engine hardware with various grades of engine oil. During the course of the development of the accelerated test it was found that cam lobe pitting and tappet wear directly correlated with the quality of an engine oil. The end of test results were evaluated on a visual inspection basis for pitting and wear severity. The cam lobes and tappets were rated on a 5 point scale from good condition down to strong pitting observed.

One round of testing, including 8 engine oils representing North America and SE Asia regions, found 6 of the oils tested failed to meet the wear criteria. A note of interest was that of the cam lobe failures 20% were represented by intake cam lobes the remaining were represented by the exhaust lobes.

# Test History – B Camshaft Pitting

## Phosphorus and Ash Effects

---



# Test History

## Lessons Learned

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- Increase in stress and the presence of an edge stress on the tappet face are accompanied by a reduction in the velocity of lubricant flowing into the contact. It is solely a function of the cam lobe profile. The lubricant entrainment velocity for the cam lobe and sliding tappet systems is nearly zero when the pitch lift velocity is a maximum.
- The distance between the cam lobe center and the line of contact is equal to the instantaneous pitch lift velocity.
- Pre-sooting the oil prior to the Cummins ISB Valvetrain Wear Test provides for the most severe wear scenario.

# Test Development

---

The intent of the ISB Valvetrain Wear Test is to take the internal accelerated cam and tappet test forward by allowing the end of test results to be evaluated on a mostly objective basis. Using previous experience with test development on heavy-duty engines for ASTM, the test procedure was proposed, drafted and supported internally. This procedure is now being finalized with the help of the industry through the ASTM ISB Test Development Task Force. Currently there are 6 labs participating on the task force. Of the six, one lab ran evaluation tests and is now upgrading hardware, two labs will be prepared to run evaluation tests in July and the remaining three should be running in August.

# Looking Forward

## Meeting the Timing

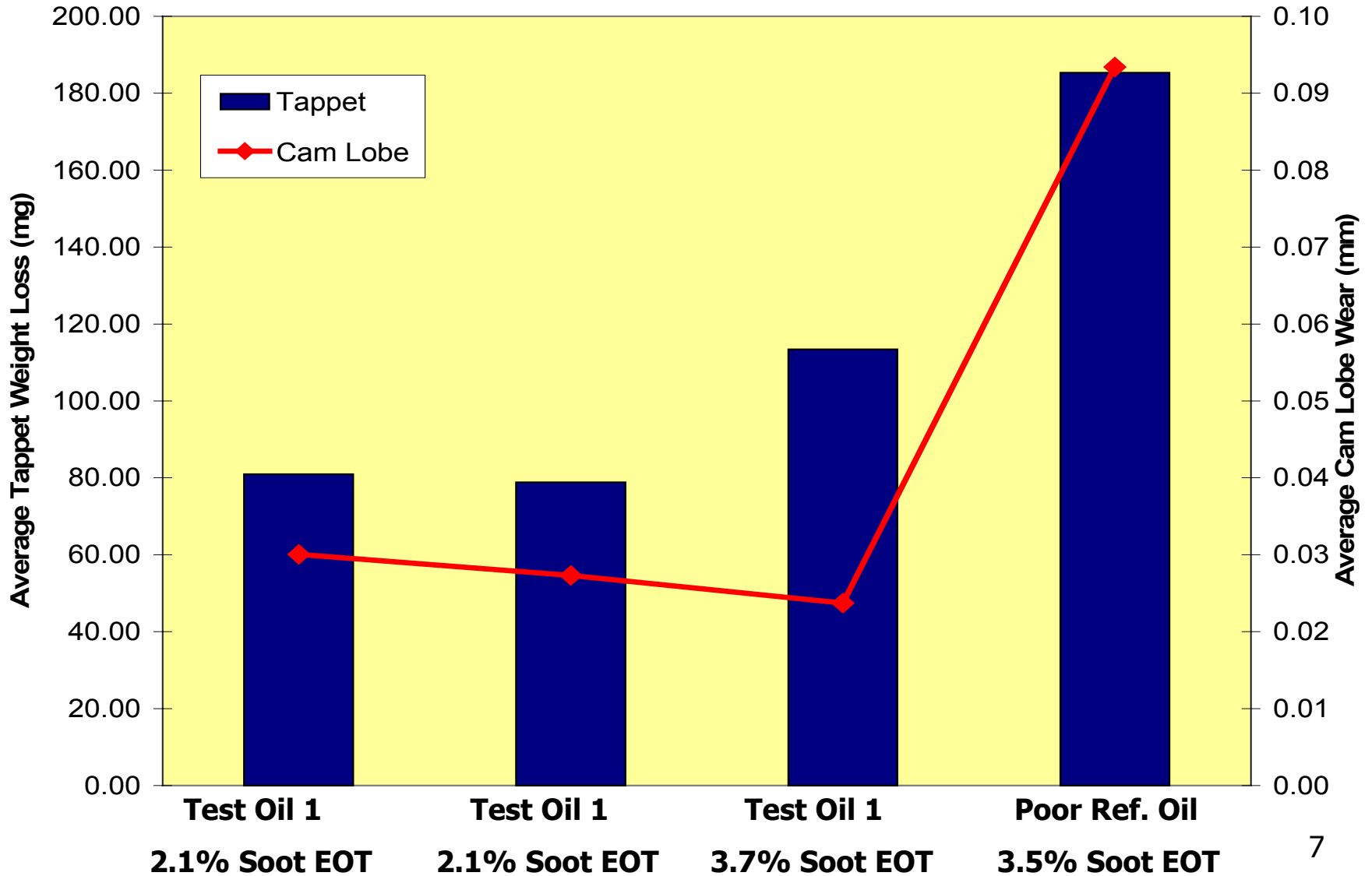
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Cummins proposes that the precision matrix testing begin on the ISB test as soon as the test is ready. However, before the matrix can begin, proof of concept data indicating the ability of the test to discriminate and repeat must be presented to the HDEOCP. This data is included in the presentation.

Once the remaining engine labs have ISB test stands on-line and the Operation and Hardware subgroup of the ISB Test Development Task Force provides positive feedback on the all test stands participating in the matrix, Cummins will move that the ISB matrix begin.

# B Engine Camshaft and Tappet Testing

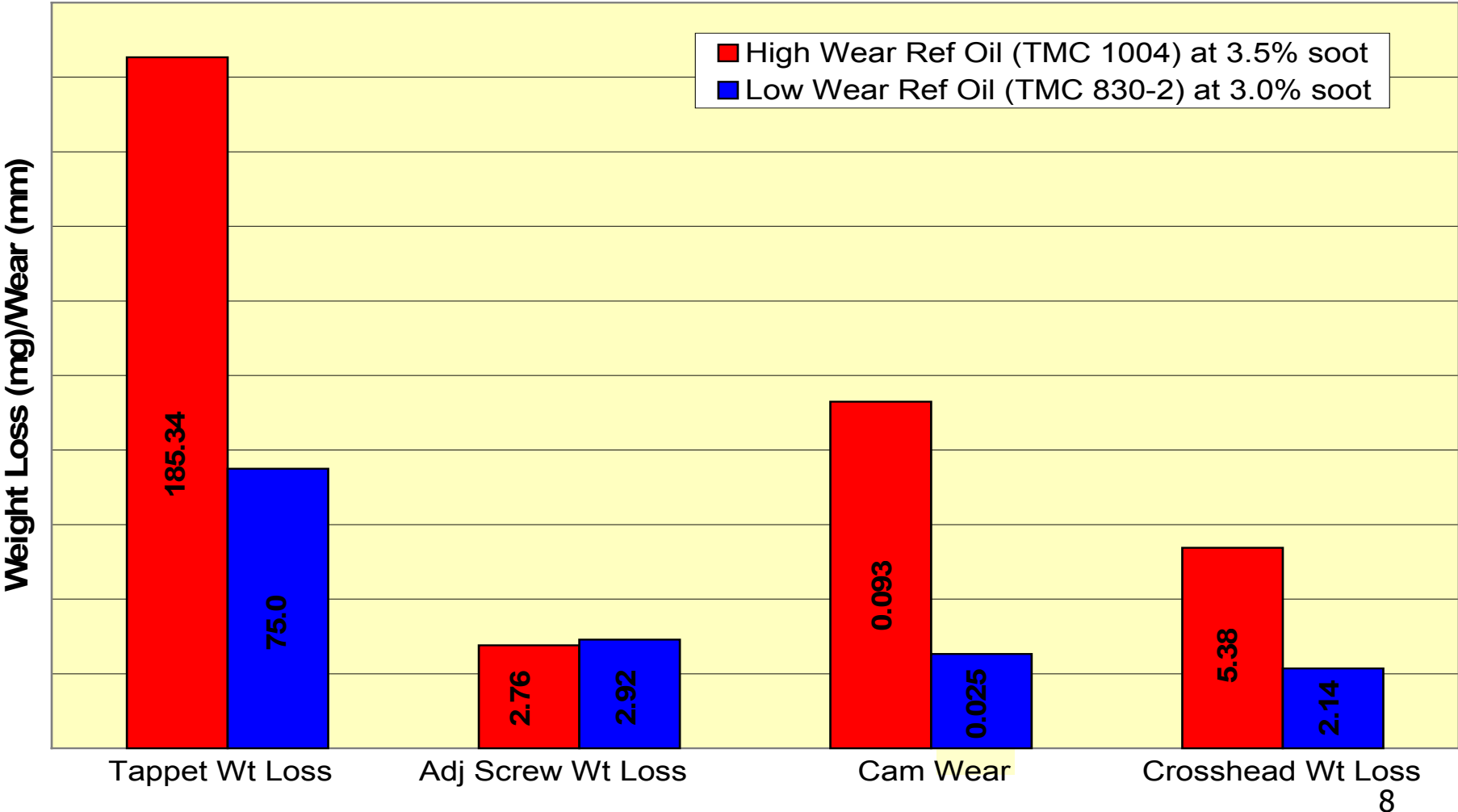
## Repeatability and Discrimination





# ISB '02 Camshaft and Tappet Data Discrimination

ISB Cam Cycle Test Data



# ISB Test Overview

---

The ISB Valvetrain Wear Test is based upon a 2004 EPA Compliant engine rated at 300 HP and 600 lbf-ft torque. Prior to starting the test, the engine is run through a series of warm-up cycles to flush the engine oil with reference or candidate oil. After the final engine oil flush, the first portion of test cycle begins. This portion (Stage I) consists of a 100 hour soot generation steady-state cycle at 1600 RPM and 325 lbf-ft torque. The timing is artificially retarded using electronic engine control hardware to hit a soot window of 3.25 +/- 0.25%. The oil level is verified as full. The test then continues repeating a 28 second accelerated wear cycle for 250 hours. The wear components and other test parameters are evaluated upon successful test completion.

# Scope

---

To develop a lubricant performance test on a Cummins ISB test platform that can discriminate and provide a quality assessment of motor oils in a sliding tappet engine under cyclic conditions. The ISB test development will consider the following parameters for lubricant quality evaluation:

## Primary Parameters

Tappet Weight Loss

Cam Lobe Wear

Cam Journal Wear

## Secondary Parameters

Push tube scuffing

Sludge

Oil filter delta P

Adjusting screw wt. loss

Crosshead weight loss

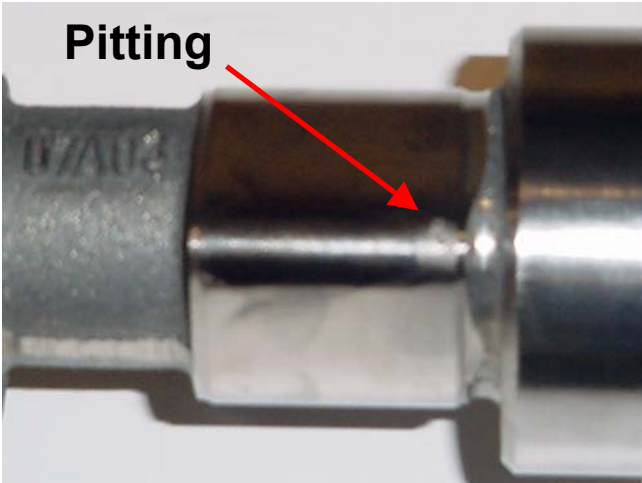
# Objectives

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- Draft of test procedure 12/03
  - **Preliminary draft completed 01/04**
  - **Work continues within the ISB Test Development Task Force to refine and standardize the procedure**
  
- Test engines to six labs 1/04
  - ExxonMobil, Lubrizol, PerkinElmer, SwRI, Valvoline
  - Ethyl engine 6/04
  
- 3. Initiate matrix design 1/04
  - **Preliminary proposal based upon 4 labs attached**
  
- 4. Begin matrix testing third quarter, 2004

# Cam and Tappets After Test

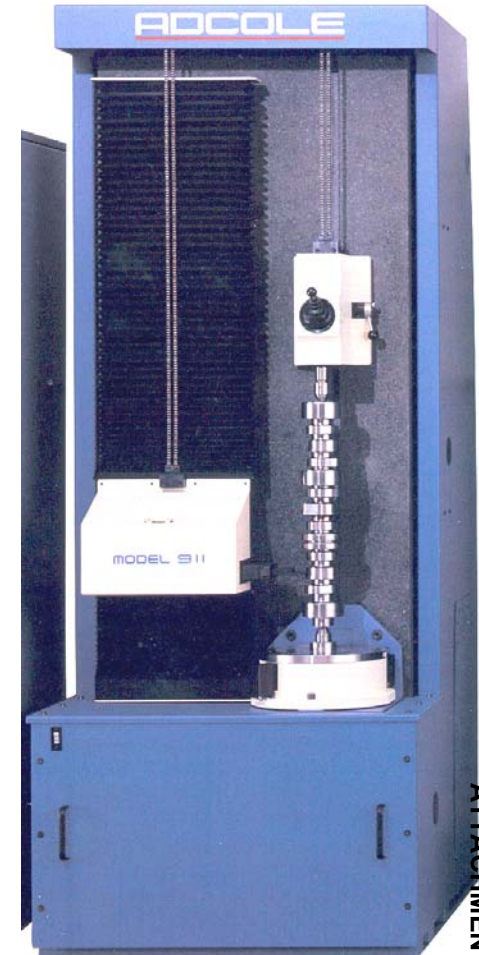
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# ISB Test Parameters

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- Parameters to be rated
  - Primary Parameters
    - Tappet Wear
      - mg wt loss
    - Cam lobe wear
      - mm wear
        - » ADCOLE measurement
        - » Cams will be pre and post measured by CPD
        - » The O&H Sub-group is evaluating alternative wear measurement methods
    - Cam journal wear
      - mm wear
        - » ADCOLE measurement



# ISB Test Parameters

---

- Parameters to be rated
  - Secondary Parameters
    - Overhead wear
      - Crosshead Weight Loss, mg loss
      - Adjusting Screw Weight Loss, mg loss
      - Push Tube Scuffing
    - Other parameters
      - Oil Filter Delta Pressure, kPa
      - Sludge, rocker cover and oil pan

# Precision ISB Matrix Design

## Reducing the costs

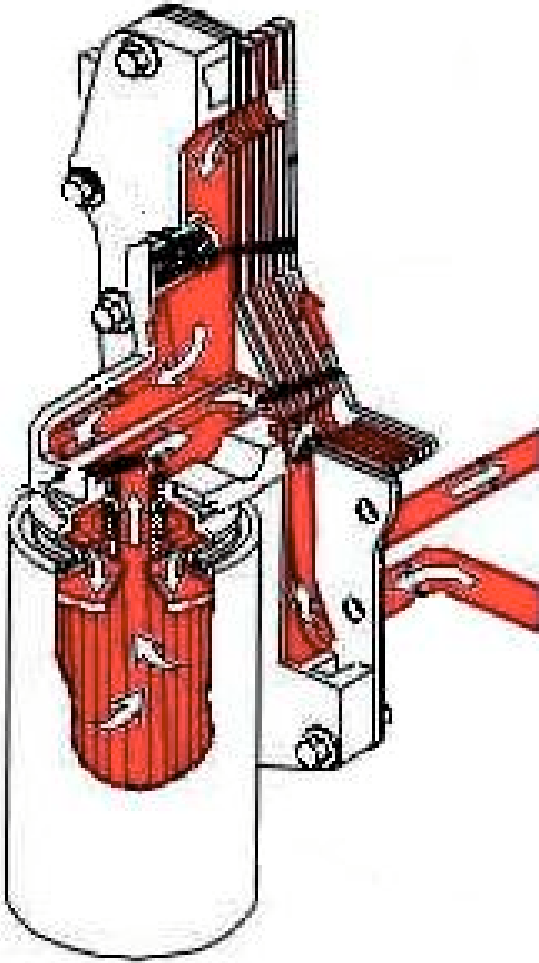
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### Ideas

- Each test stand will generate similar wear performance as the Cummins test stand based upon historical data (mean and standard deviation)
- 3 DI/VI combinations, 1 base oil, and 1 Reference Oil
- Each successful test generates 12 tappet, cam and crosshead wear points
- No VGRA or BOI included in matrix design



# Hardware Modifications



Remote Oil filter

Remote Oil Cooler



# Cummins Surveillance Panel Report to HDEOCP



Warren Totten  
David Stehouwer  
June 22, 2003  
Salt Lake City, UT



# Scope

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The Cummins Surveillance Panel is responsible for the Cummins M-11 HST and M-11 EGR test procedures. The Panel works with the ASTM Test Monitoring Center to monitor test operations, test statistics, test severity and test precision for these tests. Overall improvements in the test operation and test monitoring are accomplished with the cooperation of the test developer, the Test Monitoring Center and ASTM Subcommittee B0.02.

# Objectives

---

- Monitor and make improvements to existing tests
  - Develop an ASTM engine test method as a replacement test for the M-11 EGR and M-11 HST using the Cummins ISM engine platform. Target is to have a replacement test in place by September 2004. This engine test will be carried forward be included in the 2007 PC-10 specification for Heavy Duty Diesel Engine Oils.
- Develop an ASTM engine test method for the evaluation of a lubricant's capability to protect against overhead valve train wear using a Cummins ISB engine platform. This engine test is intended to be included in the 2007 PC-10 specification for Heavy Duty Diesel Engine Oils.

# ISB Update

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- **Test has shown ability to discriminate between oils.**
- **O&M panel meets next week in San Antonio to finalize procedural details and insure stand readiness**
- **HDEOCP is urged to select reference oils and define matrix by mid August so matrix testing can begin.**

# **ISM:**

## **Overview of status of Test Development**

- Initial phase of matrix testing complete.
- Additional data generated on two tests run to 300 hours.
- Statistical data review of the initial phase of matrix is complete.
- Test shows discrimination on CHWL, but other parameters unclear.

# Next Steps for ISM Development

---

- Cummins has expressed a desire to move toward a higher level of soot in the ISM test (6.0 min)
  - This will require a modification to the original matrix design. Additional tests that will help develop a soot correlation from 5.5% to 6.5% are now being built into a revised matrix plan.
- Next phase of testing to begin once new matrix is finalized and approved.

# Cummins ISM Test Plan

---

- Test in Stages
- Use Decision Points
- Use a Range of Oils – Quality Determined by Test Results/Models
  - Poor (1004)
  - Borderline (1005)
  - Good (830)
  - Great (ISMA)



# Cummins ISM Test Plan

---

STAGE1 COMPLETE	SwRI	PE	LZ
1004	v 5.5% Soot	v 5.5% Soot	v 5.5% Soot
ISMA	v 5.5% Soot	v 5.5% Soot	

# Cummins ISM Test Plan

STAGE2 <b>PLAN</b>	SwRI	PE	LZ
1004			v 6.5% Soot
ISMA	v 6.5% Soot		
830		v 6.5% Soot	v 6.5% Soot
1005	v 6.5% Soot	v 6.5% Soot	

# Cummins ISM Test Plan

---

- Is the Correlation Similar to the M11 EGR?
  - No: Stop the Matrix
  - Somewhat: Complete Matrix by Running the Reference Oil Twice in Each Lab (6 Tests)
  - Yes: Complete Matrix by Running the Reference Oil Once in Each Lab (3 Tests)

# Cummins ISM Matrix: Issues

---

- **Surveillance panel ran out of time before matrix could be resolved.**
- **Does not appear to be sufficient spread between poor 1004 and ISMA to offer 4 levels of discrimination**
- **Cummins believes phase 2 design should include only 3 oils and target 6.0 % soot**
  - **ISMA      Great oil**
  - **830        Borderline Good**
  - **1004      Poor**
- **Panel seeks HDEOCP discussion; approval of matrix design; and go-ahead to proceed to finalize Phase 2 on Thursday**

## ASTM HDEOAP Update – June 22, 2004

- Current C13 Oil Consumption Variability still being investigated.
  - Not entirely related to Piston Profile.
  - Mixed batches in test difficult to attribute to any particular feature
- Working with production suppliers to ensure consistency of parts.
- Target parts availability date: Aug 4 2004



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Slide 1 of 3

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## ASTM HDEOAP Update – June 22, 2004

- **Current C13 PRL to be used with tight tolerances.**
  - Pre-matrix tests to determine Oil Cons stability (July 04).
  - Procure 2000 pistons, 100 % inspection (50 sets)
- **Matrix production parts Aug 04.**
- **Low Ref Oil defined**
- **High Ref Oil Sought, Second run to be conducted with existing Commercial Oil (ULSDF)**
- **Mini-matrix funded by CAT**



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Slide 2 of 3

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# C13 Task Force

## Abbreviated Report to the HDEOCP

### June 22, 2004

- The test has been developed using PC-9 fuel, open CV and production C13 hardware. Reference #1 consistently gave oil consumption increases of greater than 50% and stuck or sluggish rings. At least one commercial CI-4 oil gave satisfactory performance.
- Caterpillar will supply 100% inspected pistons from a large batch for future testing.
- The task force has agreed on a mini-matrix with 4 tests on two oils at the independent labs to begin (Aug – Sept time frame) followed by at least 2 additional tests at other labs. This matrix will run using ULSD, CCV, low and high reference oils.
- There is a desperate need for a reference oil that can be expected to give a consistent level of excellent performance in this test!
- The task force intends to hold two teleconferences during the month of July to:
  - Establish the calibration requirements for the C13
  - Define when a C13 test stand is "ready for matrix testing"
- It is anticipated that the task force will supply discrimination and limited precision data to the HDEOCP in the November 2004 time frame.

# NCDT UPDATE

HDEOCP MEETING

Salt Lake City, Utah

June 22, 2004



# Caterpillar PC-10 Test Proposals

## Forward and Backward Compatibility

- High Temp deposits tests in past
- New lower Temp combustion with lower Piston Temps

This will drive two piston deposit tests for PC-10:

- 1) 1P for High Temperature Backward Compatibility
- 2) C13 for lower temperature (low NO<sub>x</sub>) engines



# Caterpillar PC-10 Test Proposals

Fuel Sulfur for PC-10 Tests:

- 1) 1P 500 ppm for Backward Compatibility
- 2) C13 <15 ppm for Forward Compatibility
  
- 3) CCV test



# Iron Piston Deposits, Oil Consumption

- New Test
- New Engine - Caterpillar C-13
- Length - ~500 hours
- ULS Diesel Fuel
- Combined Steady State (CCV?)
- Matrix Required
- Hardware Available - 13(7 Installed)
- Discrimination - 12/04?

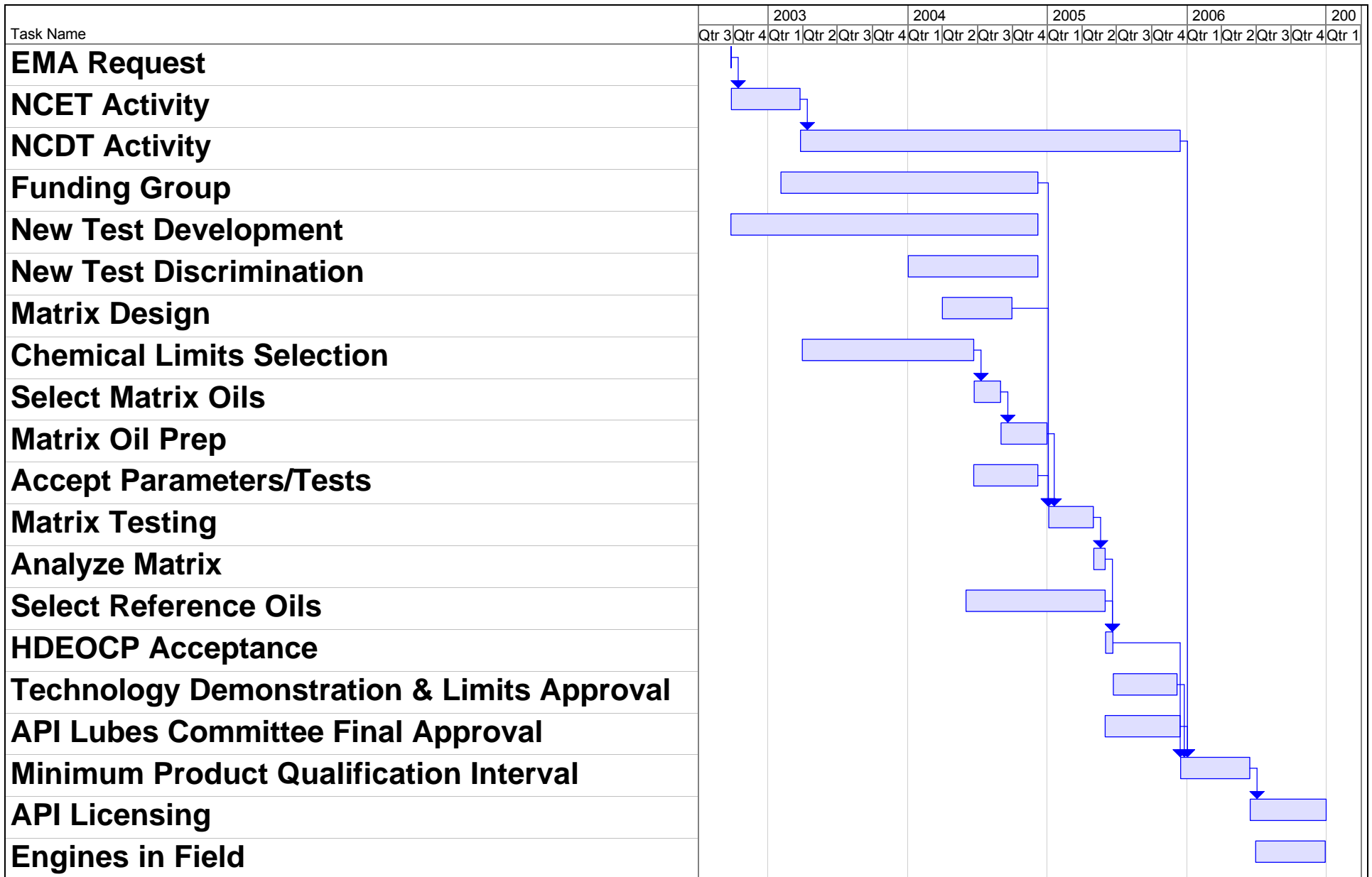
# Aluminum Piston Deposits, Oil Consumption

- CI-4 Requirement
- Caterpillar 1N or 1K
- Required for Backward Compatibility
- Matrix Not Required
- Caterpillar Sees Continued Need for Aluminum Piston Test










# NCDT CONFERENCE CALL

06/11/2004

- Reviewed Caterpillar 5/18/2004 Request
- Modifies EMA Category Development Request
- EMA Has Not Reached Consensus
- Some EMA Interest in Retaining Aluminum Piston Test
- NCDT Awaiting EMA Request for Modification



Project: PC-10 ACC-2  
Date: Tue 6/22/04

Task		Milestone		External Tasks	
Split		Summary		External Milestone	
Progress		Project Summary		Deadline	

# PC-10 Performance Requirements and Engine Tests



Performance Criteria	Fuel Sulfur, Wt %	Test	PC-10 2006
Aluminum Piston Deposits, Oil Consumption	0.05	Caterpillar 1N	X
Viscosity Increase Due to Soot at 6.0%	0.05	Mack T-11	X
Roller-Follower Valve Train Wear	0.05	GM 6.5-Liter PC – Diesel	X
Aeration	0.05	Navistar HEUI 7.3-Liter EOAT	X
Foam	–	Bench Test Sequence I, II, III	X
Volatility	–	Noack D 5800 or Distillation D 2887	X
Used Oil Viscometrics at Low Temperature	–	J300 Bench Tests MRV TP-1 Soot	X
Elastomer Compatibility		D-471, Ref. Oils	X
High Temperature/High Shear		Bosch Injector	X
Valve Train Wear, Filter $\Delta$ P and Sludge	.05	Cummins ISM	X
Valve Train Wear	15 ppm	Cummins ISB	X
Oil Consumption and Piston Deposit	15 ppm	Caterpillar C-13	X
Ring, Liner Bearing Wear & Oil Consumption	15 ppm	MackT-12	X
Oil Oxidation	0.10	See III G	X
Shear Stability – 90 Cycles	–	Bosch Injector ASTM D 3945	X
<i>Total Number of Engine and Bench Tests</i>			<b>15</b>

**PC-10 Matrix Funding & Design TF**  
**ASTM HDEOCP Meeting**  
**June 22, 2004**  
**Salt Lake City, UT**



# PC-10 Engine Test Matrix

## *Funding*

- Both API & ACC have agreed to match up to \$1MM EMA cash & in-kind contributions for PC-10 Precision & BOI matrix testing
- Three million dollars from trade associations will not fully fund the PC-10 matrix (C13, ISB, T-12)
  - ❖ Precision only = \$4.6MM (18 tests)
  - ❖ Precision + BOI = \$7.1MM (28 tests)
- Next steps
  - ❖ Establish the level of EMA funding
  - ❖ Define matrix parameters to provide the PC-10 MDTF with input to generate designs for industry review and approval

# PC-10 Engine Test Matrix

## *Design Task Force*

- **Membership established**

Nancy Diggs (Infineum)

Joan Evans (Infineum/ACC)

Tom Franklin (PerkinElmer)

Charlie Passut (Ethyl)

Bill Runkle (Valvoline)

Jim Rutherford (Oronite)

Elisa Santos (Infineum)

Phil Scinto (Lubrizol)

Greg Shank (Volvo-Mack)

Matt Urbanak (Shell)

Jim Wells (SWRI)

Wim Van Dam (Oronite)

John Zalar (TMC)

Scott Zechiel (Detroit Diesel)

*Any other volunteers?*

- **Initial teleconference on June 16**

- ❖ Reviewed Scope & Objectives and time line

- ❖ Discussed inputs required to deliver suitable designs

- ❖ Agreed on next steps

# PC-10 Engine Test Matrix

## *Input for MDTF*

	HDEOCP	Test TF / SP	Test Labs	PC-10 NCDT	BOI-VGRA TF	DEOAP / LC
<b>Identify / Finalize New Tests</b>	X	X		X		
<b>Precision only or Precision/BOI</b>	X			X	X	
<b>Matrix Oils</b>						
Number of Additive Technologies	X	X		X	X	X
Number of Base Oils				X	X	X
<b>Test Stands</b>						
Number of Labs	X	X	X	X		
Stands per Lab		X	X			
<b>Stand Calibration Criteria</b>	X	X				

- **MDTF will seek guidance from the appropriate industry groups to define matrix parameters**
- **Target to have preliminary input within 2 to 3 weeks**

# Motions By HDEOCP to date

- **75% membership vote by HDEOCP to move forward with PC-10.**
- **Chemical limits “frozen” on June 22<sup>nd</sup> 2004**
- **Engine tests to be “frozen” by Dec 7<sup>th</sup> 2004**
- **13% Noack Volatility**
- **90 cycle shear stability**
- **Vamac added to API CI-4 seal tests**

# CAT 1N

## Industry Correction Factor



James McCord

*June 22, 2004*

# CAT 1N

## Hardware Change

- As of 5/31/04, the 1Y3998 liner has replaced the 1Y3555 liner for use in the CAT-1N
- Based on 5 data points, 1Y3998 liner required a TLHC industry correction factor of  $-1.135$  (transformed value)
- TGF appeared to be mild, but, no action will be taken until the p value is less than 0.05
- 5 additional tests will be run in December 04 to verify the severity of the CAT-1N

June 22, 2004

# CAT 1N

## Effective Pass Limit

<b>Parameter</b>	<b>ASTM 1st Test Limit</b>	<b>Effective Pass Limit</b>
<b>TLHC</b>	<b>3</b>	<b>13</b>
<b>BSOC</b>	<b>0.5</b>	<b>0.5</b>
<b>TGF</b>	<b>20</b>	<b>20</b>
<b>WDN</b>	<b>286.2</b>	<b>286.2</b>

June 22, 2004

## Summary of New 1N Liner Performance

Parameter		units	N	MIN	MAX	MEAN	STD	Significant?
TGFyi	1Y3555	yi	237	-1.601	2.829	0.046	1.006	
TGFyi	New Liner	yi	5	-1.155	-0.917	-1.071	0.090	
TGF	Range	%		11	15	12.4		
TGF	Shift	%				-15.6		p=0.0139
WDNyi	1Y3555	yi	237	-2.221	3.070	-0.238	1.012	
WDNyi	New Liner	yi	5	-2.545	-0.132	-1.048	0.989	
WDN	Range	demerits		138.6	200.6	177.1		
WDN	Shift	demerits				-28.4		p=0.0776
With SA	Range	demerits		157.8	200.6	188.4		
With SA	Shift	demerits				-16.5		p=0.4174
TLHCyi	1Y3555	yi	237	-1.260	3.368	-0.138	0.955	
TLHCyi	New Liner	yi	5	0.253	1.619	0.799	0.586	
TLHC	Range	%		1	5	2.1		
TLHC	Shift	transformed				0.719		
TLHC %	Shift	%				1		p=0.0301
BSOCyi	1Y3555	yi	237	-2.689	5.978	-0.215	1.166	
BSOCyi	New Liner	yi	5	-1.680	0.320	-0.480	0.754	
BSOC	Range	g/kWh		0.08	0.23	0.17		
BSOC	Shift	g/kWh				-0.02		p=0.6142

### Discussion:

The table above is laid out with the first two rows of each group showing descriptive statistics for the two liner types (1Y3555 vs New). As is the case for all TMC analysis, yi values are used to account for the differing performance levels of the several reference oils and, in the case of TLHC, to incorporate the transformation calculation. All rows after the first two refer to New Liner data.

The next row, labeled “Range”, shows the minimum, maximum, and mean values from the New Liner runs in reported units. The value shown for TLHC is the back-transformed value of the mean of the transformed values. This will be different from the mean of the percent values (2.1% vs 2.4%). Keep in mind that the *reported* units for TLHC is *transformed TLHC*, not percent.

The row following that, labeled “Shift”, the shift from target that the mean New Liner value represents. This is shown first in reported units. Again note that for TLHC this will be *transformed TLHC* and not percent. In the case of TLHC, there is an additional “Shift” line showing the offset amount back-transformed into percent. This value (1%) is provided as a point of reference only. The “Shift” values were all calculated from the mean yi for the New Liners using the same standard deviation used to generate lab severity adjustments (TGF = 14.6, WDN = 27.1, TLHC = 0.9, BSOC = 0.45).

Two of the “Shift” values would be considered significant; TGF and TLHC. TGF is mild by 15.6%; TLHC is severe by 0.719 transformed TLHC (the criteria for significance being a p-value less than 0.05).

The p-value for WDN, though not significant, is low enough to garner some attention. An assumption made here is that the New Liner data was generated by stands operating on target. A review of severity adjustments shows that for TGF, TLHC, and BSOC this is true. For WDN, however, three of the 4 labs have been producing mild WDN results irrespective of liner type. So, I severity-adjusted the 5 New Liner results and re-computed the analysis. The results are shown on the additional “Range” and “Shift” rows of the WDN table. In this scenario, the p-value becomes comfortably insignificant (0.4174).



**Update following April 8 teleconference:**

Updating the 1004-3 targets to include all operationally valid runs to date results in:

Variable	N	Mean	Std Dev	Minimum	Maximum
TGF	16	23.9	14.6	9	58
WDN	16	190.7	24.7	159.8	246.4
TLHCti	16	0.1806	0.3977	0	1.098612
BSOC	16	0.148	0.038	0.09	0.25

Recomputing all of the previous analysis gives:

**Revised 1004-3 Targets**

Parameter	units	N	MIN	MAX	MEAN	STD	Significant?
TGFyi	1Y3555	237	-1.601	2.829	0.072	1.008	
TGFyi	NEW	5	-0.884	-0.610	-0.788	0.104	
TGF	RANGE		11	15	12.4		
TGF	SHIFT				-11.5		p=0.0581
WDNyi	1Y3555	237	-2.221	3.070	-0.203	1.013	
WDNyi	NEW	5	-2.109	0.401	-0.552	1.029	
WDN	RANGE		138.6	200.6	177.1		
WDN	SHIFT				-15.0		p=0.4464
TLHCyi	1Y3555	237	-1.260	3.368	-0.112	0.978	
TLHCyi	NEW	5	1.289	4.051	2.394	1.184	
TLHC	RANGE		1	5	2.1		
TLHC	SHIFT				2.154		
TLHC %	SHIFT				7.6		p<.0001
BSOCyi	1Y3555	237	-2.689	5.978	-0.164	1.177	
BSOCyi	NEW	5	-1.790	2.158	0.579	1.489	
BSOC	RANGE		0.08	0.23	0.17		
BSOC	SHIFT				0.03		p=0.1660

With these 1004-3 targets, the WDN and BSOC shifts are insignificant (as was the case before). For TLHC, the shift becomes both more pronounced and more significant. The TGF shift using these targets would be considered insignificant. However, the p-value is low enough to warrant further investigation. The question raised is: What is future testing likely to bring for TGF?

To try to answer that question, I extrapolated five tests into the future by duplicating each of the five New Liner runs completed so far. This is probably a fair approximation of what might result from five more runs. The outcome of this hypothetical is shown on the next page.

**Five Additional Tests**  
( & revised 1004-3 targets)

Parameter		units	N	MIN	MAX	MEAN	STD	Significant?
TGFyi	1Y3555	yi	237	-1.601	2.829	0.072	1.008	
TGFyi	NEW	yi	10	-0.884	-0.610	-0.788	0.098	
TGF	RANGE	%		11	15	12.4		
TGF	SHIFT	%				-11.5		p= 0.0076
WDNyi	1Y3555	yi	237	-2.221	3.070	-0.203	1.013	
WDNyi	NEW	yi	10	-2.109	0.401	-0.552	0.970	
WDN	RANGE	demerits		138.6	200.6	177.1		
WDN	SHIFT	demerits				-15.0		p= 0.2859
TLHCyi	1Y3555	yi	237	-1.260	3.368	-0.112	0.978	
TLHCyi	NEW	yi	10	1.289	4.051	2.394	1.116	
TLHC	RANGE	%		1	5	2.1		
TLHC	SHIFT	transformed				2.154		
TLHC %	SHIFT	%				7.6		p<.0001
BSOCyi	1Y3555	yi	237	-2.689	5.978	-0.164	1.177	
BSOCyi	NEW	yi	10	-1.790	2.158	0.579	1.404	
BSOC	RANGE	g/kWh		0.08	0.23	0.17		
BSOC	SHIFT	g/kWh				0.03		p= 0.0536

Assuming that this is a reasonable approximation of future testing, the TGF shift will again become significant.

**Further update to revise estimate of shift for TLHC:**

Because the transformation applied to TLHC includes the natural log function, small changes to transformed test results have exponential impact on results expressed as percent. This fact was overlooked by everyone during the April 8 teleconference. Consequently, I've been asked to reexamine the TLHC shift neglecting the transformation.

Because untransformed TLHC data is not normally distributed, neglecting the transformation does compromise the analysis somewhat (there is a reason we use the transformation in the first place, after all; most statistical analyses assume that the data is normally distributed). However, the shift between the New Liner data and historic data is sufficiently large that the general results should still be valid even if the exact p-values must be taken with a grain of salt.

With the transformation removed and using the recomputed 1004-3 targets the TLHC yi shift is 2.9645. Using the untransformed equivalent of the TLHC SA standard deviation (3.7) to convert this Δ/s shift to a Δ gives 10.9686%. As before, this shift is significant.

If this shift is linear and universally applicable, then a 1Y3555 pass-limit result of 3% would be expected to produce 13.9686% on New Liners. The value to add to the transformed test result to compensate for the shift would be:

$$\ln(3\%+1) - \ln(13.9686\%+1) = -1.320$$

Two examples:

Rated TLHC result	14%	13%
Transformed result	$\ln(14\%+1) = 2.708$	$\ln(13\%+1) = 2.639$
Plus -1.320 shift	$2.708-1.320 = 1.388$	$2.639-1.320 = 1.319$
Reported TLHC result	$e^{(1.388)}-1 = 3.007\%$	$e^{(1.319)}-1 = 2.740\%$

What does adding this value to the five New Liner results look like?

Rated TLHC result of the 5 New Liner tests	Transformed	Back-transformed
1%	-0.627	-0.466%
1%	-0.627	-0.466%
2%	-0.221	-0.198%
3%	0.066	0.068%
5%	0.472	0.603%

Does adding this value to the New Liner results return TLHC performance to historic levels? Using untransformed values, the resultant p-value is 0.2338. Though not exactly correct due to the non-normal distribution of the untransformed data, this is probably good enough to deem the difference between the New Liner group and the 1Y3555's not significant.

What if the transformation is restored? The p-value then becomes 0.0675 which would make the shift still not significant.

# HDEOCP Report

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**James Mc Geehan**

**Chairman**

**Heavy-Duty Engine Oil Classification Panel**

*June 22<sup>st</sup> 2004*

*Salt Lake City, UT*



# Motions Passed by HDEOCP

- **75% membership vote by HDEOCP to move forward with PC-10**
- **13% Noack for volatility**
- **90 cycle for shear stability**
- **Vamac added to API CI-4 seal tests**
- **Chemical limits to be frozen by June 22, 04**
- **Engine test to be frozen by Dec. 7<sup>th</sup> 2004**

# Motion passed for Chemical limits

- **Adopted Chemical Limits for PC-10 at:**
  - 1.00% SASH max. (D874)**
  - 0.12% Phosphorus max. (D4951)**
  - 0.4% Sulfur max. (D4951 or D2622)**

**As “Non Critical” limits.**

**Footnote to be added to the document for SASH:**

- 1.00% for one test**
- 1.02% for two tests**
- 1.03% for three tests**

# Motions passed by HDEOCP

- **The matrix oils may be blended at lower SASH, Phosphorus and Sulfur than the PC-10 Chemical Limits.**
- **Request an endorsement of aromatic in PC-10 fuel to change from, 28-33.5% to 26-31.5%**

# Motion votes

- **Sulfur limit: 16 for—one negative—one abstain.**
- **Phosphorus: 17 for –and one abstain**
- **SASH: 18 for –no negatives or abstains**



# Engine test status for PC-10

- **Cummins ISB cam and tappet test shown “proof of performance”. Ready for discrimination matrix with 15 ppm fuel sulfur**
- **Cummins ISM demonstrated wear separation for reference oils at 5.5% soot. Matrix work at 6.0% soot planned to increase separation of reference oils for wear and filter delta P, with PC-9 fuel.**
- **Caterpillar C13 oil consumption, piston deposits and blow-by with CCV. Matrix study planned for August 04 with 15 ppm fuel sulfur**
- **Mack T-12 power-cylinder wear and oil oxidation, based on Mack T-10 and Mack T-11 under development at high EGR rates with hardware at labs by 3Q04 and discrimination matrix 4Q04**

# Matrix Funding for C13/ISB/T-12

- API and ACC have agreed to match up to \$1MM. EMA cash and in-kind contributions for PC-10 precision and BOI matrix.
- \$3MM from the trade associations will not fully fund the PC-10 matrix
- Precision only=\$4.6 MM (18 tests)
- Precision and BOI= \$7.1 MM (28 tests)

**Next step is to establish the EMA funding**

# PC-10 Timing

- **Chemical limits: June 2004**
- **Matrix oils selection: October 2004**
- **Engine tests discrimination: December 2004**
- **Matrix Testing: January to June 2005**
- **Acceptance HDEOCP: June 2005**
- **Technology demonstration: June to Dec 2005**
- **Approve Limits: Dec 2005**
- **Minimum qualification interval: Jan to June 2006**
- **API License: June 2006**
- **Oils in market place by: 3Q2006**

# Summary of PC-10

- **Currently on the planned timing schedule, with all programs, including engine tests, matrix funding, matrix design.**
- **Congratulations and appreciation to the following committees: Fuel task-force, Chemical limits task force; Funding and matrix design task-force and each of the engine task-forces**