

**HEAVY-DUTY ENGINE OIL CLASSIFICATION PANEL
OF
ASTM D02.B0.02
July 11, 2001**

Holiday Inn – O’Hare International Hotel, Rosemont, IL

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ACTION ITEMS

- | | |
|---|----------------------------|
| 1. Issue “Exit” ballots | J. McGeehan |
| 2. Complete T-10 matrix statistical analysis | T-10 Task Force |
| 3. Complete M-11 EGR matrix statistical analysis | M-11 EGR Task Force |
-

MINUTES

- 1.0 Call to Order
 - 1.1 Chairman Jim McGeehan called the meeting to order at 8:30 a.m. on July 11, 2001, in the Kitty Hawk room of the Holiday Inn – O’Hare International Hotel of Rosemont, IL. There were 12 members present or represented and there were approximately 21 guests present. The attendance list is shown as Attachment 2.
- 2.0 Agenda
 - 2.1 The published agenda (Attachment 1) was reviewed and additions agreed to: Tom Cousineau on 1R; Chris May on LOTRUO; Jai Bansal on used oil viscometrics.
- 3.0 Previous Meeting Minutes
 - 3.1 Lew Williams moved and Dwayne Tharp seconded that the minutes of the June 19, 2001, meeting in San Diego be approved as distributed to the members. The motion passed unanimously. Members and guests will be notified when the minutes are posted to the TMC website.
- 4.0 Membership
 - 4.1 There were no changes in membership (Attachment 3).
- 5.0 Chairman’s Comments
 - 5.1 The next two meetings will start at 7:30 a.m. and continue to 4:30 p.m., so do not plan to leave early.

- 5.2 To facilitate presentation of the T-10 and M-11 statistical analyses, Dwayne Tharp and Jim Wells volunteered to bring digital projectors for the August meeting – after finding the hotel wanted \$500+ to rent one.
- 6.0 PC-9 Matrix Status
 - 6.1 Jeff Clark presented the PC-9 matrix test status for John Zalar (Attachment 4) and noted that all T-10 and M-11 EGR testing had completed. One of the nine first round 1R tests had suffered an operational problem during break-in and had not restarted yet, while the other eight had completed or were nearing completion. So, completion of the planned 1R matrix could not occur now until mid-Sept. at the earliest.
 - 6.2 Steve Kennedy reported that the ACC had verbally agreed to use funds originally earmarked for the 1Q matrix now for the 1R matrix. An addendum to the MOA is now circulating for signatures by all the stake-holders.
- 7.0 Mack T-10
 - 7.1 Greg Shank reported on the T-10 status (Attachment 5) and presented a “To Do” list (Attachment 6) for things that need to be done before the next HDEOCP meeting. He noted that the Task Force had agreed to start using intake and exhaust CO2 measurements to control EGR rate instead of the exhaust oxygen sensor. This should provide more consistent EGR mass flow rates between labs. The Task Force is looking for HDEOCP approval of the test at the Aug. 15th meeting, with registration hopefully to start in the 8/16 to 8/21 timeframe.
 - 7.2 Jim Rutherford presented a preliminary summary of the statistical analysis of T-10 matrix data available to date (Attachment 7). The Task Force decided to not use the data from the test on oil CMIR 38815 because of oil analysis and test result anomalies.
- 8.0 Cummins M-11 EGR
 - 8.1 Dave Stehouwer reported on the M-11 EGR and presented what Cummins is proposing as limits for the test (Attachment 8). “Beaded” (reworked) test filters must be used on all future reference tests and those tests must fall within an 8.0% to 9.0% soot window at 250 hours with a minimum of 4.6% average soot – calculated using all 13 oil samples. Future non-reference tests must achieve a minimum of 8.0% soot at 250 hours and the “average” soot must be 4.6% or higher. Dave also went over the “To Do” list (Attachment 9) of things that need to be done before the next HDEOCP meeting.
 - 8.2 Dennis Malandro reviewed the preliminary statistical analysis he had done using the available M-11 EGR matrix data (Attachment 10).
- 9.0 Caterpillar 1R
 - 9.1 Lew Williams presented an update on the 1R matrix design and test activities (Attachment 11).
 - 9.2 Ton Cousineau presented data from Ethyl (Attachment 12) illustrating their concern that using the 1P read across guidelines for the 1R may be counter productive and opposite of what is needed.
 - 9.3 Steve Kennedy remarked that he had some data that supported the Ethyl position.
 - 9.4 Tom and Steve to get their data to Ralph Cherrillo and the API BOI/VGRA Task Force will consider it and review the guidelines.
- 10.0 Mack T-8E
 - 10.1 Greg Shank restated Mack’s concern that today’s engines are more severe with regard to oil shear. Greg then made a motion that the Relative Viscosity calculation for PC-9 T-8E results be changed to use 100% of the D6278 shear value and the RV limit be set to 1.7.

Ralph Cherrillo seconded the motion. In the ensuing discussion, the motion was withdrawn and never voted on. It was decided to include this item as an "exit" ballot to the HDEOCP members.

11.0 High Temperature, High Shear

- 11.1 Greg Shank opened the discussion with previously used slides of EMA's recommendation for a HTHS limit and wear versus HTHS (Attachment 13).
- 11.2 Steve Kennedy presented an ExxonMobil perspective on a 3.5 cP HTHS limit (Attachment 14) and how it would constrict the basestock / additive blend window. They would prefer the 3.5 limit, if adopted, be made a "non-critical" limit.
- 11.3 Pat Fetterman presented Infineum concerns with any proposed HTHS limit above 3.0 cP (Attachment 15).
- 11.4 Jim McGeehan presented data from SAE paper 932845 showing significant reductions in ring / liner wear rates with increasing HTHS (Attachment 16).
- 11.5 Ken Chao, Dave Stehouwer and Dwayne Tharp all expressed opinions that wear protection was more important to their companies than possible fuel economy benefits of lower viscosity within a grade.
- 11.6 Bill Kleiser made a motion that HTHS be considered a "non-critical" parameter for the PC-9 specification at 3.5 cP for fresh oil. Tom Cousineau seconded the motion which passed with 9 votes for, 2 against and 1 abstain. SAE specification (?) 3244 was cited as a reference for the definition of "non-critical".

12.0 Used Oil Viscometrics

- 12.1 Chris May reported on the LOTRUO activities (Attachment 17) and asked that the remaining T-10 and M-11EGR samples be sent to him for analysis. His group recommends use of the standard MRV TP-1 test for evaluating the low temperature viscosity of used oils. SwRI is to send 75 hour T-10 samples to Chris May at Imperial Oil. There was discussion about soot dropout from samples and someone remarked they had checked samples for up to 3 years and found no dropout.
- 12.2 Jai Bansal presented data Infineum had acquired from field and engine tests of various oils (Attachment 18). They concluded that MRV TP-1 performance of a used oil is not predicted by the KV100 increase, relative viscosity or viscosity slope and that certain oils will exhibit potential for low temperature pumpability problems in the field.
- 12.3 Dave Stehouwer presented plots from a low temperature pumpability study using an operable engine and various sooted / fresh oils (Attachment 19). Based on that data, Cummins is proposing a limit of 25,000 cP at 5°C higher than the fresh oil "W" grade temperature, using the 75 hour T-10 sample (approximately 5% soot). Steve Kennedy asked that the modified MRV method be used if the sample exhibited yield stress. This proposal will be included in the upcoming "exit" ballot.

13.0 Elastomers

- 13.1 Tom Boschert reported on the status of the elastomer compatibility specification (Attachments 20, 21, 22, 23) and indicated one reference oil was not going to be sufficient for the method. He also felt a "referee" body would be needed to rule on situations that were perhaps opposite the expected trend. He presented a chart of proposed limits (Attachment 24). This proposal will also be included in the upcoming "exit" ballot.

14.0 Caterpillar 1N

- 14.1 Dwayne Tharp reported he has seen 1N data for one "PC-9" oil which had the following results:

WDN = 276	versus a CG-4 limit of	286 max.
TGF = 38	“ “ “	20 max.
TLHC= 8	“ “ “	3 max.
OC = 0.19	“ “ “	0.5 max.

This result is causing concern that oils formulated to pass the new tests may struggle to pass the 1N. Some discussion with regard to changing limits for the 1N in PC-9, but that raised backward compatibility concerns.

15.0 Next Meeting

15.1 Chairman McGeehan reviewed the next meeting times (7:30 – 4:30) and the forthcoming “exit” ballots. The “exit” ballots are to cover the carryover test items, elastomer compatibility, HTHS, T-8E and low temperature used oil viscosity.

16.0 Thanks

16.1 Appreciation was expressed to Chris May and Tom Boschert for all the work they have done on the low temperature viscosity measurement method and elastomer compatibility.

17.0 Adjournment

17.1 The meeting was adjourned at 12:49 p.m. on July 11, 2001.

Submitted by:

Jim Wells
Secretary to the HDEOCP

ASTM-HDEOCP
Holiday Inn O'Hare International
July 11th 2001
8:30 am –1:00 pm

Chairman/ Secretary: Jim Mc Geehan/Jim Wells
Purpose: PC-9
Desired Outcomes: - Matrix results in EGR tests
 Test limits on all the PC-9 tests

TOPIC	PROCESS	WHO	TIME
Agenda Review	<ul style="list-style-type: none"> • Desired Outcomes & Agenda 	Group	8:30-8:35
Minutes Approval	<ul style="list-style-type: none"> • June 19th 2001 	Group	8:35-8:40
Membership	<ul style="list-style-type: none"> • Changes • Chairman's comments 	Group Jim Mc Geehan	8:40-8:45
Matrix Status	<ul style="list-style-type: none"> • Mack T-10; Cummins M11-ERG; Cat 1R • Time line for PC-9 	John Zalar	8:45-8:55
Cat 1R Approval	<ul style="list-style-type: none"> • Stack-holder approval of Cat 1R 	Steve Kennedy	8:55-9:00
Mack T-10 Up-date: new data	<ul style="list-style-type: none"> • All Matrix results" • Ring-Liner wear • Bearing wt loss and lead increase • Oil consumption • IR oxidation of used oil • Statistical analysis of data • Discussion 	Greg Shank Jim Rutherford	9:00-9:30
Cummins M11 EGR	<ul style="list-style-type: none"> • All Matrix results • Cross-head, injector screw and top-ring wear • Filter delta p • Sludge • New filter results • Statistical analysis of data • Discussion 	Dave Stehouwer Dennis Malandro	9:30-10:00
Caterpillar 1R	<ul style="list-style-type: none"> • Matrix design • Matrix status • Timing of completion 	Dwayne Tharp Don Marn	10:00-10:15-
Coffee break			10:15-10:30

TOPIC	PROCESS	WHO	TIME
Mack T-8E	<ul style="list-style-type: none"> • 100% DIN shear of VI improver • Support data • Proposed limits for exit ballot 	Greg Shank	10:30-11:00
SAE 10W-30 HT/HS limits for PC-9	<ul style="list-style-type: none"> • Data to support a 3.5 HT/HS limit • Mack T-9/Cummins M11 ring wear/Navistar bearings • Global DHD-1 limits • Discussion 	Greg Shank	11:00-11:30
Used oil viscometrics	<ul style="list-style-type: none"> • Cummins M11 pumping times with high soot oils compared to fresh oils. • Proposed test and limits for exit ballot 	Dave Stehouwer	11:30-12:00
Elastomers compatibility	<ul style="list-style-type: none"> • Task-force recommendations on tests and precision • Exit ballot limits 	Tom Boschert	12:00-12:30
Cat 1N	<ul style="list-style-type: none"> • API CG-4 limits • (Cat 1N in CG-4/Cat1K in CH-4) • Feature oils performance in Cat 1N • Category oil performance 	Dwayne Tharp	12:30-12:45
Next meeting	<ul style="list-style-type: none"> • August 15th • Holiday Inn O'Hare, Chicago • Time change: 8:00 am-4:30 pm 	Jim Mc Geehan	12:45-12:50
New or Old Business	<ul style="list-style-type: none"> • Action Items: Exit Criteria ballots • E-electronic ballot or fax 	Jim Mc Geehan	12:45-1:00

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Reddy, Vijay N. Thermo Haake 149 Commonwealth Dr. (Thermal Lab) Menlo Park, CA 94025	(650) 688-7075 (650) 688-7202 vijay.reddy@thermohaake.com		
Righi, Dino Lubrizol Corp. 29400 Lakeland Blvd. Wickliffe, OH 44092	(440) 347-4436 (440) 943-9013 dwri@lubrizol.com		
Romanoschi, Ovidiu Infineum USA LP. P.O. Box 735 Linden, NJ 07036	(908) 474-3335 (908) 474-2298 ovidiu.romanoschi@infineum.com		
Rosenbaum, John Chevron Products Co. 100 Chevron Way Richmond, CA 94802-0627	(510) 242-5673 (510) 242-3758 rosj@chevron.com		
Rumford, Robert H. Haltermann Products 1201 South Sheldon Rd. Channelview, TX 77530-0429	(281) 457-2768 (281) 457-1469 rhrumford@haltermann-usa.com		
Runkle Jr., William A. Valvoline Company LA 3 South P.O. Box 14000 Lexington, KY 40512-4000	(859) 357-7686 (859) 357-3343 wrunkle@ashland.com	WAR	☺
Rutherford, Jim Chevron Oronite 100 Chevron Way Richmond, CA 94802-0627	(510) 242-3410 (510) 242-1930 jaru@chevron.com	JR	☺

ASTM**SECTION D.02.B0.02
HEAVY DUTY ENGINE OIL CLASSIFICATION PANEL****ATTENDANCE LIST****AUGUST 2001****PREVIOUS GUESTS**

	Phone No. Fax No. e-mail add.	INITIAL WHEN PRESENT	ROOM FEE
St. Germain, Bob Crompton Corp. 6847 Napier Lane Houston, TX 77069	(281) 587-2393 (281) 587-0338 robert_stgermain@cromptoncorp.com		
Sander, John Lubrication Engineers, Inc. 1919 E. Tulsa Wichita, KS 67216	(316) 529-2112 (316) 529-4654 sanderj@lubricationengineers.com		
Sarlo, Mark Southwest Research Institute 6220 Culebra Rd. San Antonio, TX 78238	(210) 522-3754 (210) 523-6919 msarlo@swri.org		
Schoppe, Dean PerkinElmer AR 5404 Bandera Rd. San Antonio, TX 78238	(210) 523-4605 (210) 523-4607 dean.schoppe@perkinelmer.com		
Schuettenburg, Alex Phillips Petroleum 148 AL, PRC Bartlesville, OK 74004	(918) 661-3863 (918) 661-8060 adschue@ppco.com		
Scinto, Phil Lubrizol 29400 Lakeland Blvd. Wickliffe, OH 44092	(440) 347-2161 (440) 347-9031 prs@lubrizol.com		
Selby, Ted Savant, Inc. 4800 James Savage Rd. Midland, MI 48642	(517) 496-2301 (517) 496-3438 tselby@savantgroup.com		
Shah, Mayur Lubrizol Corporation 29400 Lakeland Blvd. Wickliffe, OH 44092	(440) 347-1697 mpsa@lubrizol.com	MS	☺

ASTM**SECTION D.02.B0.02
HEAVY DUTY ENGINE OIL CLASSIFICATION PANEL****ATTENDANCE LIST****AUGUST 2001****PREVIOUS GUESTS**

	Phone No. Fax No. e-mail add.	INITIAL WHEN PRESENT	ROOM FEE
Strigner, Paul 31 Seguin St. Ottawa, Ontario Canada K1J 6P2	(613) 746-0647 (613) 746-9292		
Sutherland, Robert Pennzoil-Quaker State 1520 Lake Front Circle The Woodlands, TX 77380	(281) 363-8029 (281) 363-8002 RobertSutherland@pzlqs.com		
Sztenderowicz, Mark Chevron Products Co. 100 Chevron Way Richmond, CA 94802-0627	(510) 242-1022 (510) 242-3758 mlsz@chevron.com		
Tarbox, Steven R. 76 Lubricants Company 1920 E. Deere Avenue Santa Ana, CA 92705	(714) 428-7400 (714) 428-7498 starbox@tosco.com		
Tharby, Ron Tharby & Associates 273 Juniper Ave. Burlington, Ontario L7L2TS	(905) 632-1568 (905) 333-8194		
Tucker, Richard Shell International Petroleum Co. P.O. Box 1380 Houston, TX 77251-1380	(281) 544-8354 (281) 544-6196 rtucker@shellus.com		
Van Dam, Wim Oronite P.O. Box 1627 Richmond, CA 94802	(510) 242-1404 (510) 242-3173 wvda@chevron.com	WvD	☺
Venier, Cliff Pennzoil-Quaker State P.O. Box 7569 The Woodlands, TX 77381-2539	(281) 363-8060 (281) 363-8002 cliffordvenier@pzlqs.com		

ASTM**SECTION D.02.B0.02
HEAVY DUTY ENGINE OIL CLASSIFICATION PANEL****ATTENDANCE LIST****AUGUST 2001****PREVIOUS GUESTS**

	Phone No. Fax No. e-mail add.	INITIAL WHEN PRESENT	ROOM FEE
Zaiontz, Michael Perkin Elmer 5404 Bandera Rd. San Antonio, TX 78238	(210) 647-9483 (210) 523-4607 mike.zaiontz@perkinelmer.com		
Zalar, John 6555 Penn Ave. ASTM TMC Pittsburgh, PA 15206	(412) 365-1005 (412) 365-1047 jlz@tmc.astm.cmri.cmu.edu		
Ziemer, Jim Chevron Products Co. 100 Chevron Way Richmond, CA 94802	(510) 242-2362 (510) 242-1156 jnzi@chevron.com		

ASTM

SECTION D.02.B0.02 HEAVY DUTY ENGINE OIL CLASSIFICATION PANEL

ATTENDANCE LIST

AUGUST 2001

GUESTS

	Phone No. Fax No. e-mail add.	ROOM FEE
Name: ___Jacobson, Mark Company: _Dupont Address: _36263 Derby Downs ___Solon, OH 44139	(440) 248-9151 (440) 248-9161 mark.s.jacobson@usa.dupont.com	☺
Name: ___Wu, Y. T. Ken Company: _Dupont Co. Address: _712 Chestnut Run ___Wilmington, DE 19880-0712	(302) 999-2481 (302) 999-4822 Yun-Tai.Wu@usa.dupont.com	☺
Name: ___Sutherland, Mark Company: _Chevron Oronite Address: _4502 Centerview Dr., Suite 210 ___San Antonio, TX 78228	(210) 731-5605 (210) 731-5699 msut@chevron.com	☺
Name: _____ Company: _____ Address: _____ _____		
Name: _____ Company: _____ Address: _____ _____		
Name: _____ Company: _____ Address: _____ _____		
Name: _____ Company: _____ Address: _____ _____		
Name: _____ Company: _____ Address: _____ _____		

HDEOCP Voting Members

Balance Between OEM's and Oil Companies and Additive Suppliers

- ◆ **G. Shank**
 - **Mack Trucks**
- ◆ **D. Stehouwer**
 - **Cummins Engine Company**
- ◆ **M. (Mesfin) Belay**
 - **Detroit Diesel Corp.**
- ◆ **K. Chao**
 - **John Deere**
- ◆ **F. Bondarowicz**
 - **Internal Truck and Engine Corp.**
- ◆ **R. Stockwell**
 - **GM Powertrain**
- ◆ **D. Tharp**
 - **Caterpillar Inc.**
- ◆ **J. Mc Geehan**
 - **Chevron Products**
- ◆ **S. Kennedy**
 - **ExxonMobil**
- ◆ **A. Huang**
 - **Equilon Enterprises**
- ◆ **T. Cousineau**
 - **Ethyl Corp.**
- ◆ **W. Kleiser**
 - **Oronite**
- ◆ **P. Fetterman**
 - **Infineum USA LP**
- ◆ **L. Williams**
 - **Lubrizol Corp.**

Status of PC-9 Matrix Testing

Presented to HDEOCP

July 11, 2001

Jeff Clark

Presenting for John L. Zalar

T-10

- **Planned Tests: 28**
- **Total Starts: 30**
- **Completed Tests**
 - **Verified / posted on TMC web site: 27**
 - **EOT and being reviewed/verified: 1**
 - **Aborted/Invalid: 3**
- **Last Matrix Test EOT'd: 7/3/01**

M11-EGR

- **Planned Tests: 26**
- **Total Starts: 28**
- **Completed Tests**
 - **Verified and posted on TMC web site:25**
 - **EOT and being reviewed/verified: 1??**
 - **Aborted/Invalid: 2**
- **Last Matrix Test EOT'd: 6/23/01**

1R

- **Planned Tests: 18**
- **Total Starts: 9**
- **Completed Tests**
 - **Verified and posted on TMC web site: 0**
 - **EOT and being reviewed/verified: 0**
 - **Aborted/Invalid: 0**
- **Tests Currently Running: 9**
- **Earliest EOT for Last Matrix Test: 8/10/01**

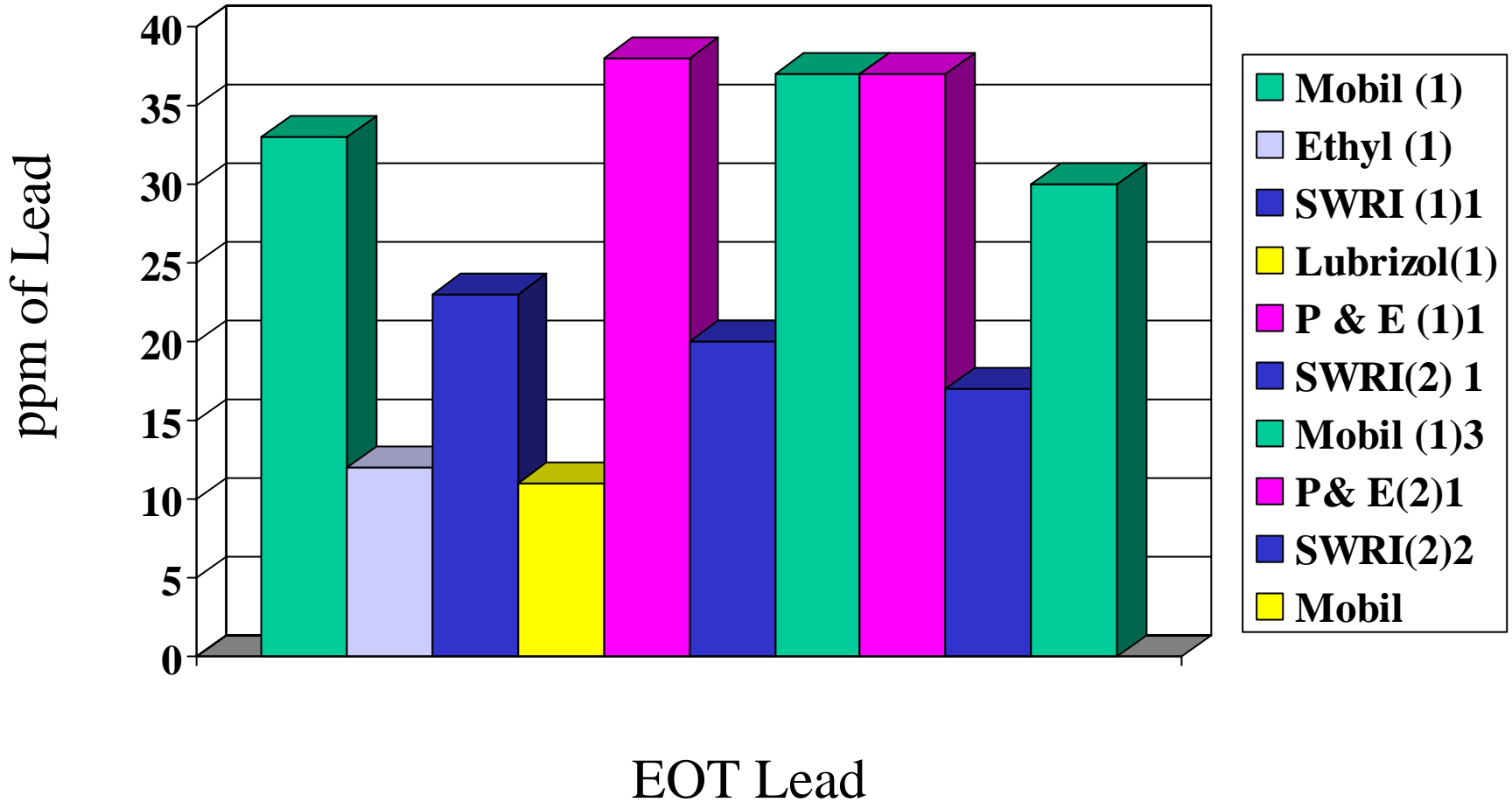


Mack T10 Status

- 31 Test Completed
- Task Force Meeting's in March, April, June & July 10 (Columbus)
- Issues: Oxidation, Oil Consumption, Deposits & EOT pb variability
- Estimated Data Analysis Completion - July 27

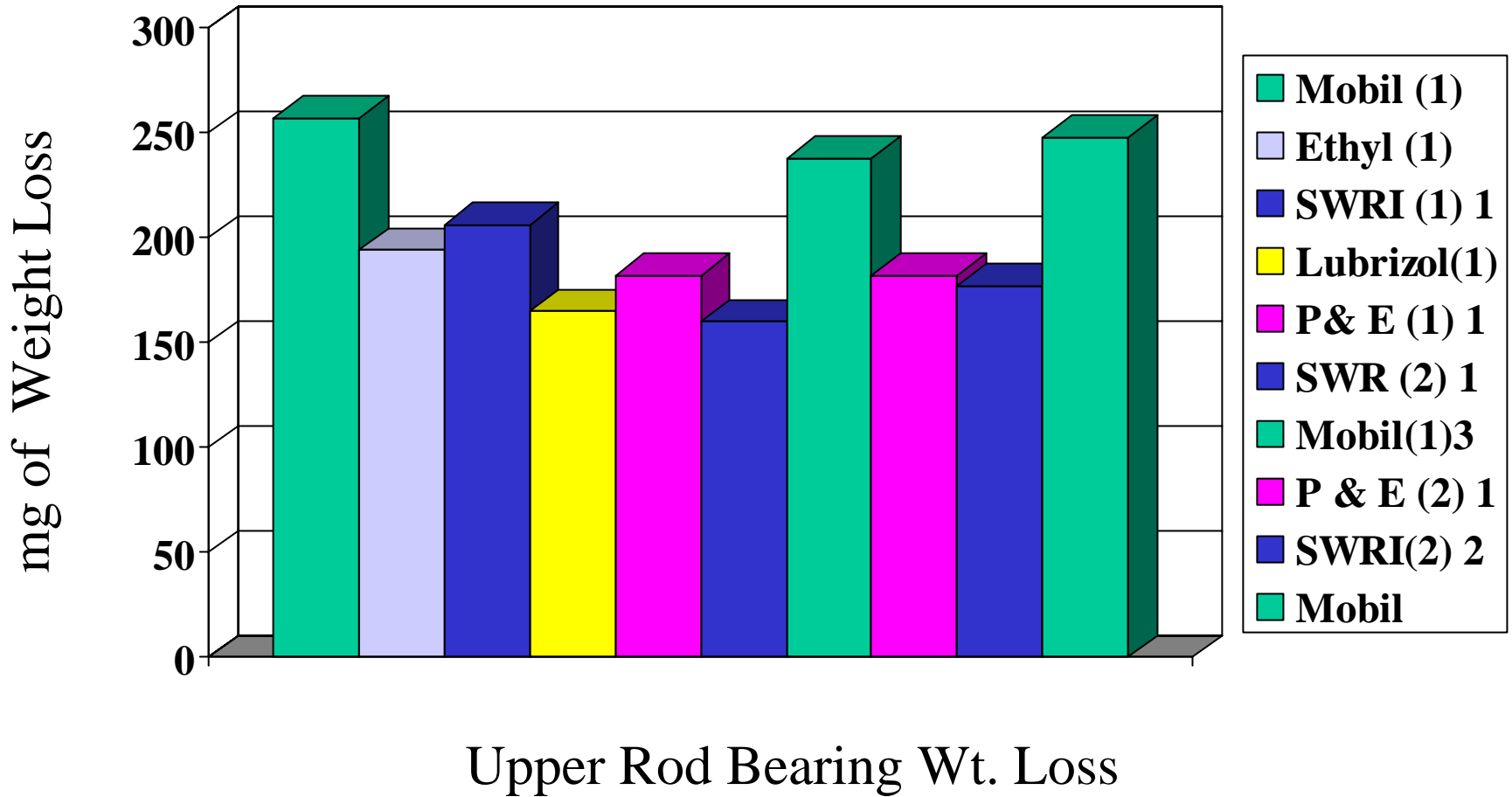
T10 Matrix Data

Oil A



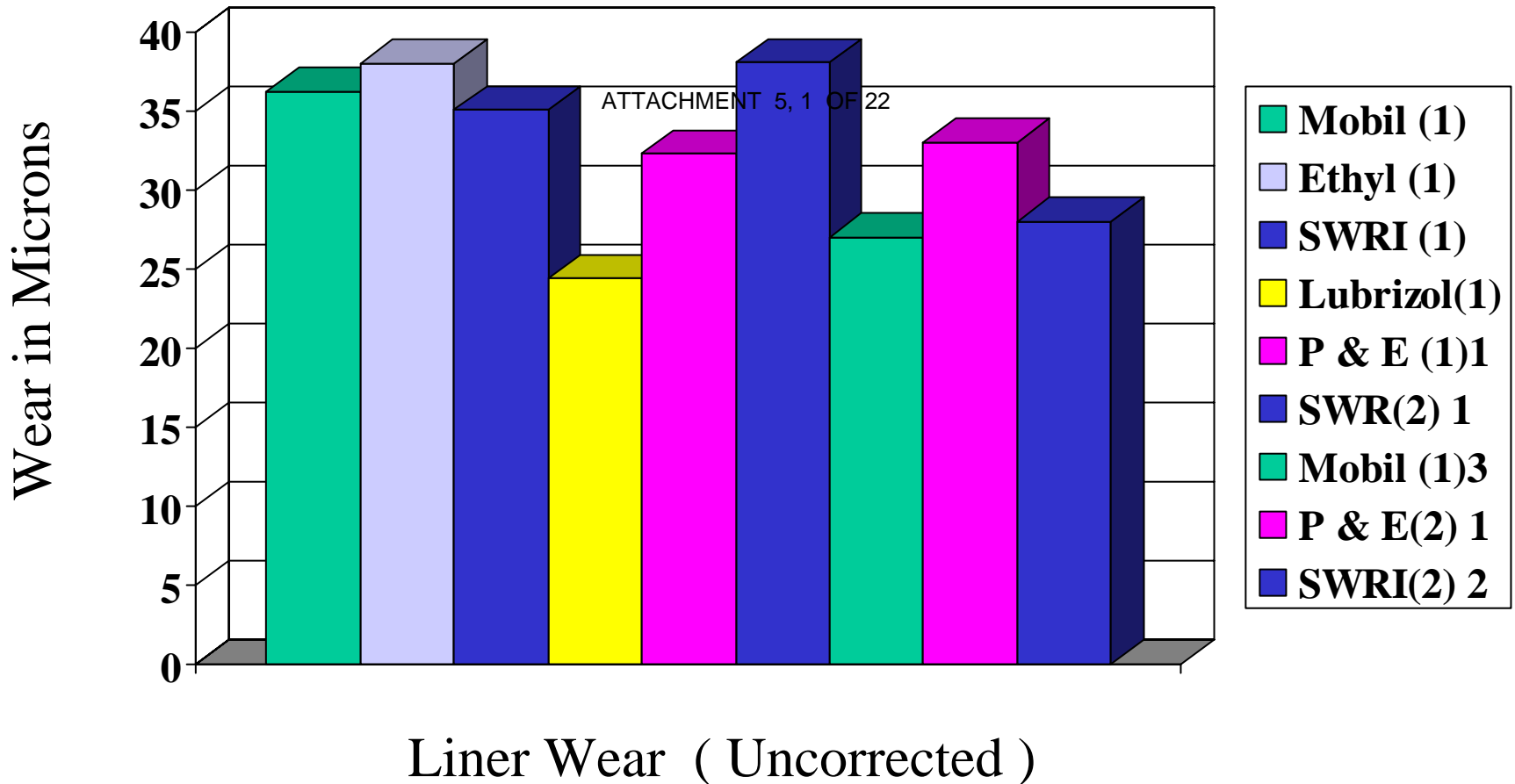
T10 Matrix Data

Oil A



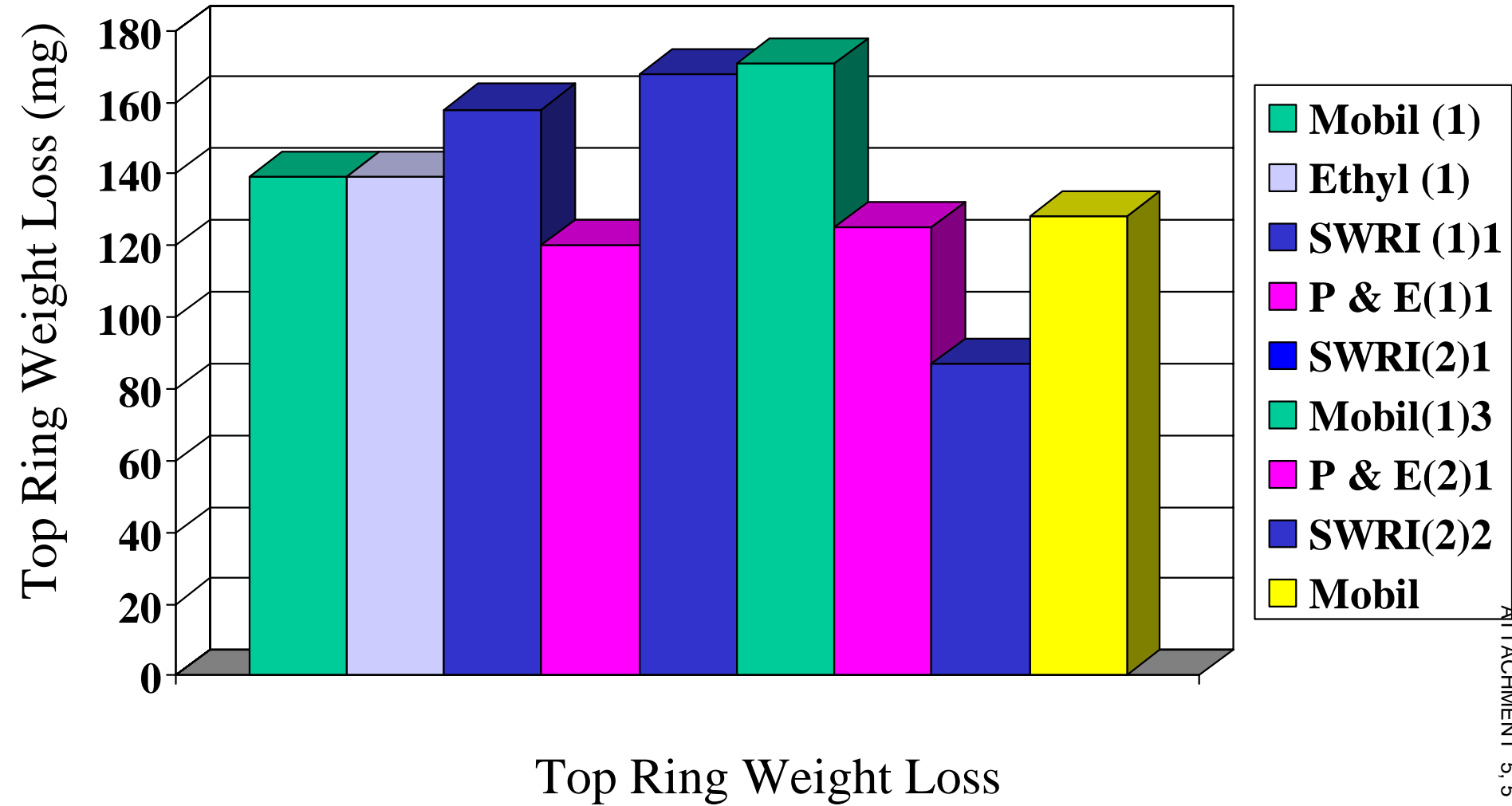
T10 Matrix Data

Oil A



T10 Matrix Data

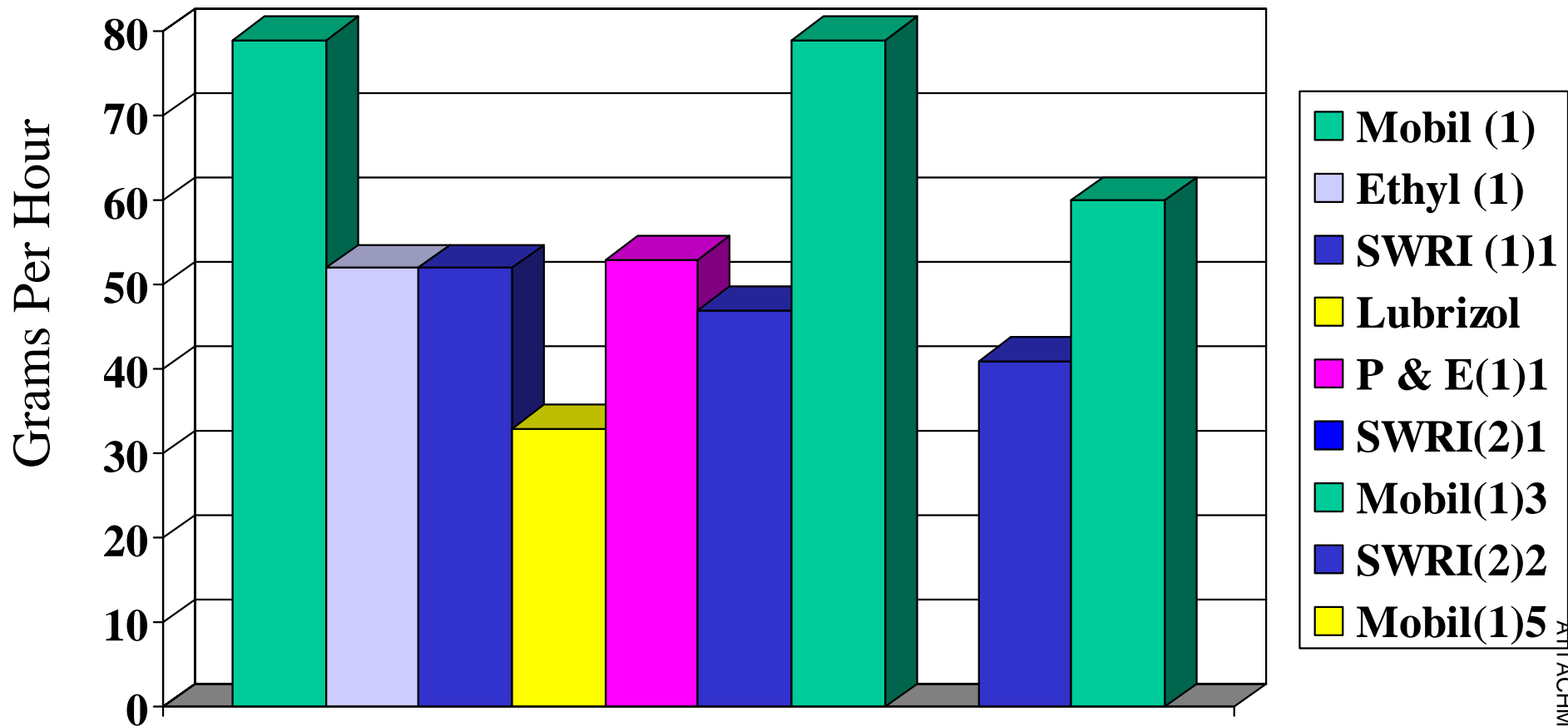
Oil A



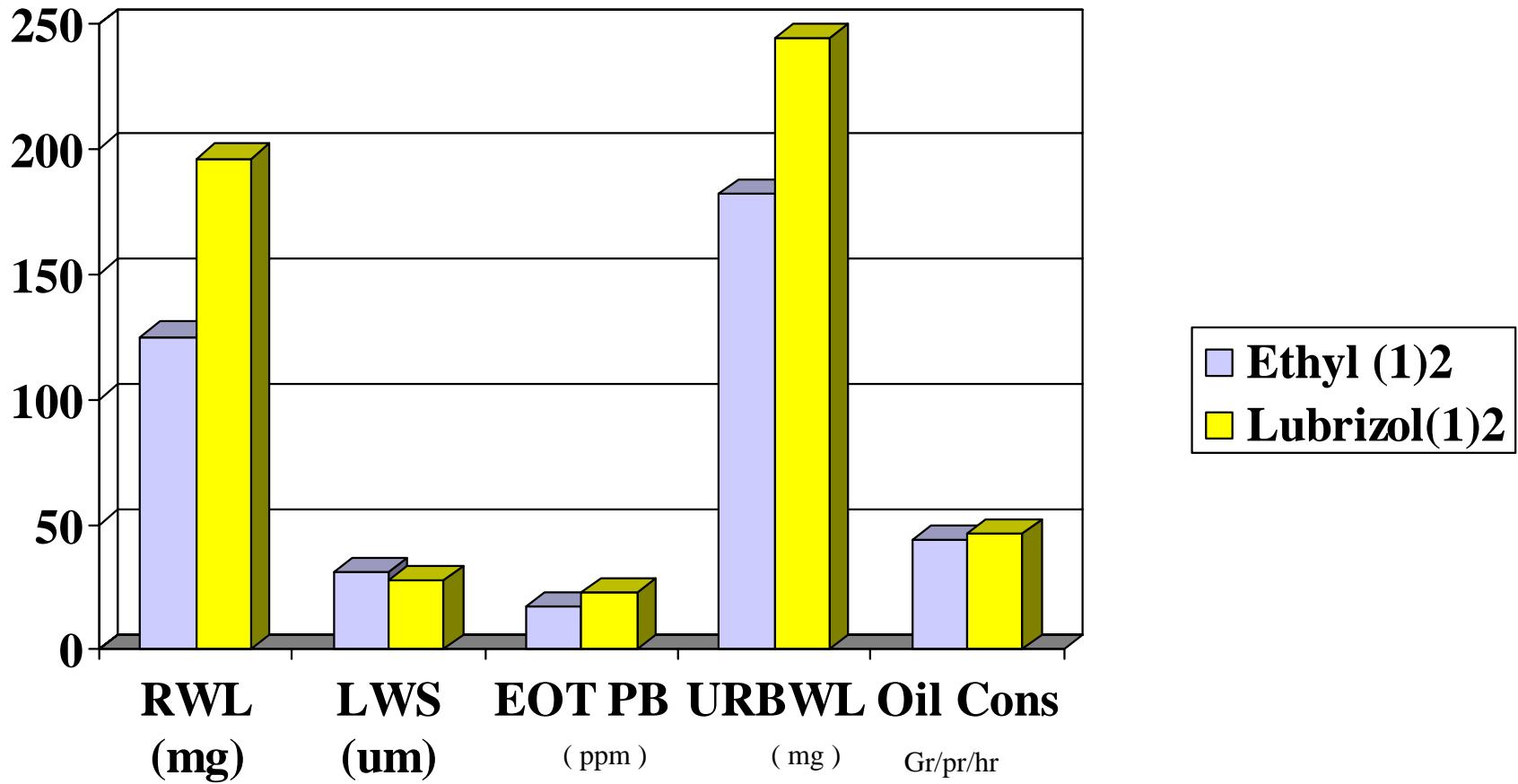


T10 Matrix Data

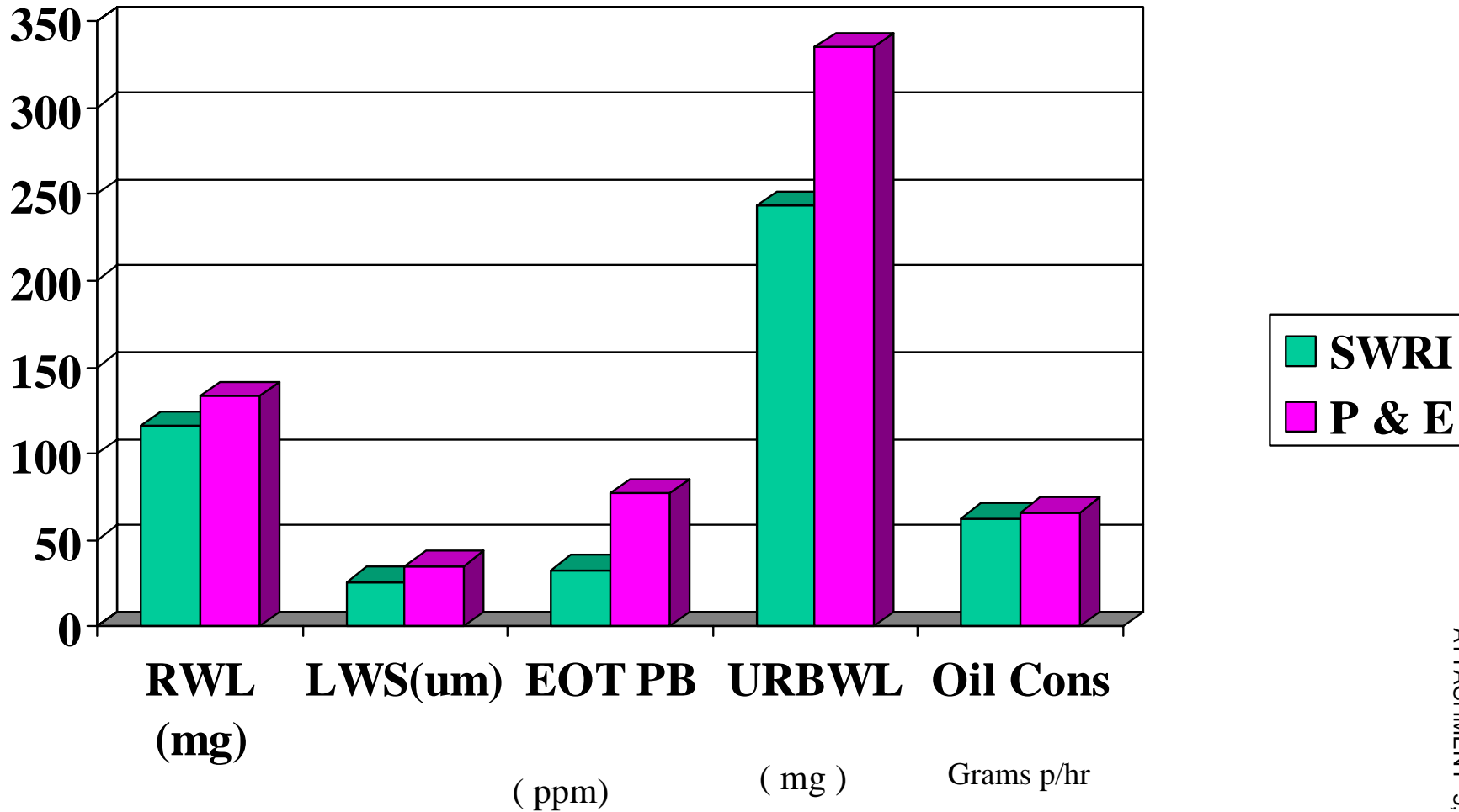
Average Oil Consumption Oil A



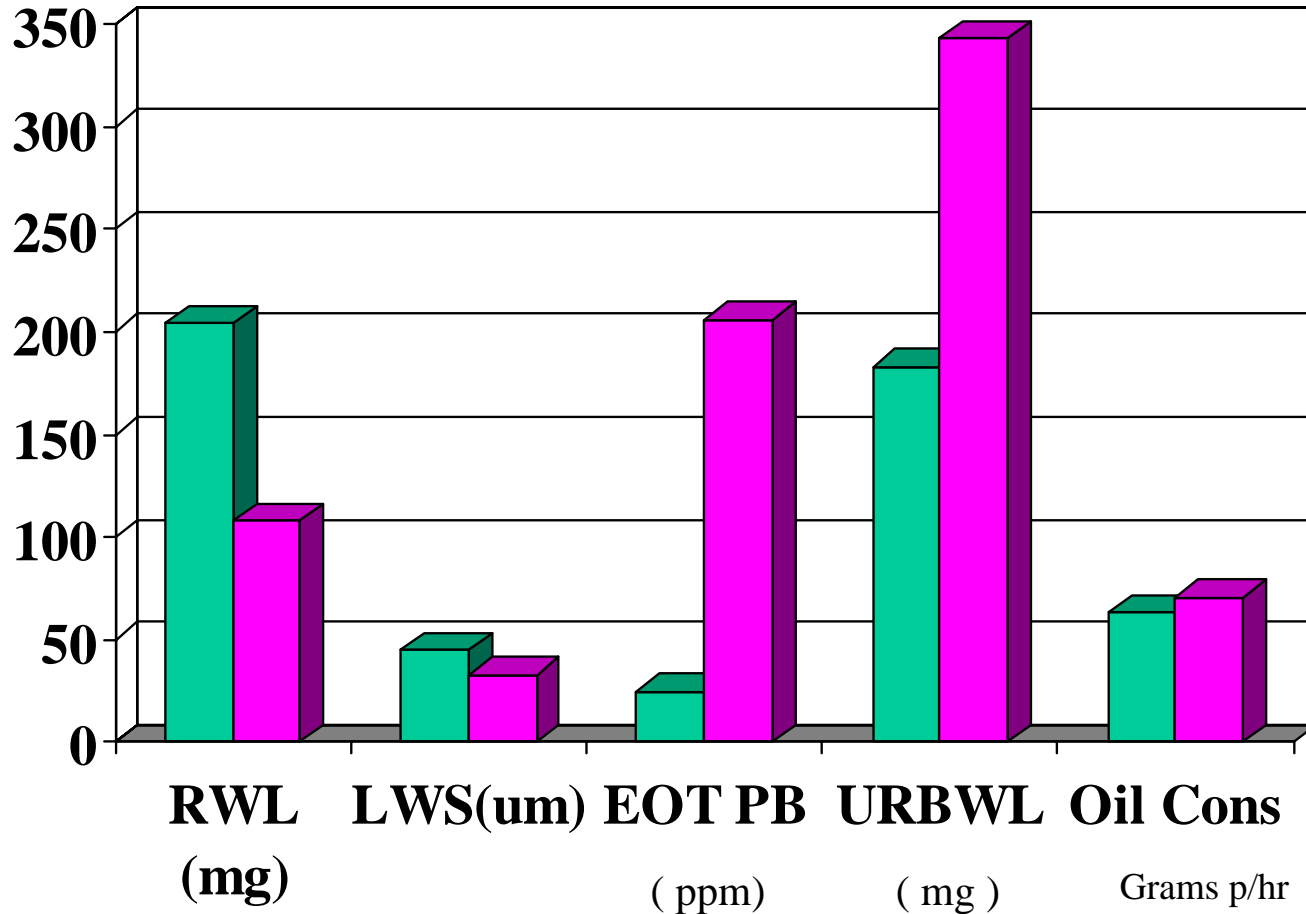
T 10 Matrix Oil B



T 10 Matrix Oil C

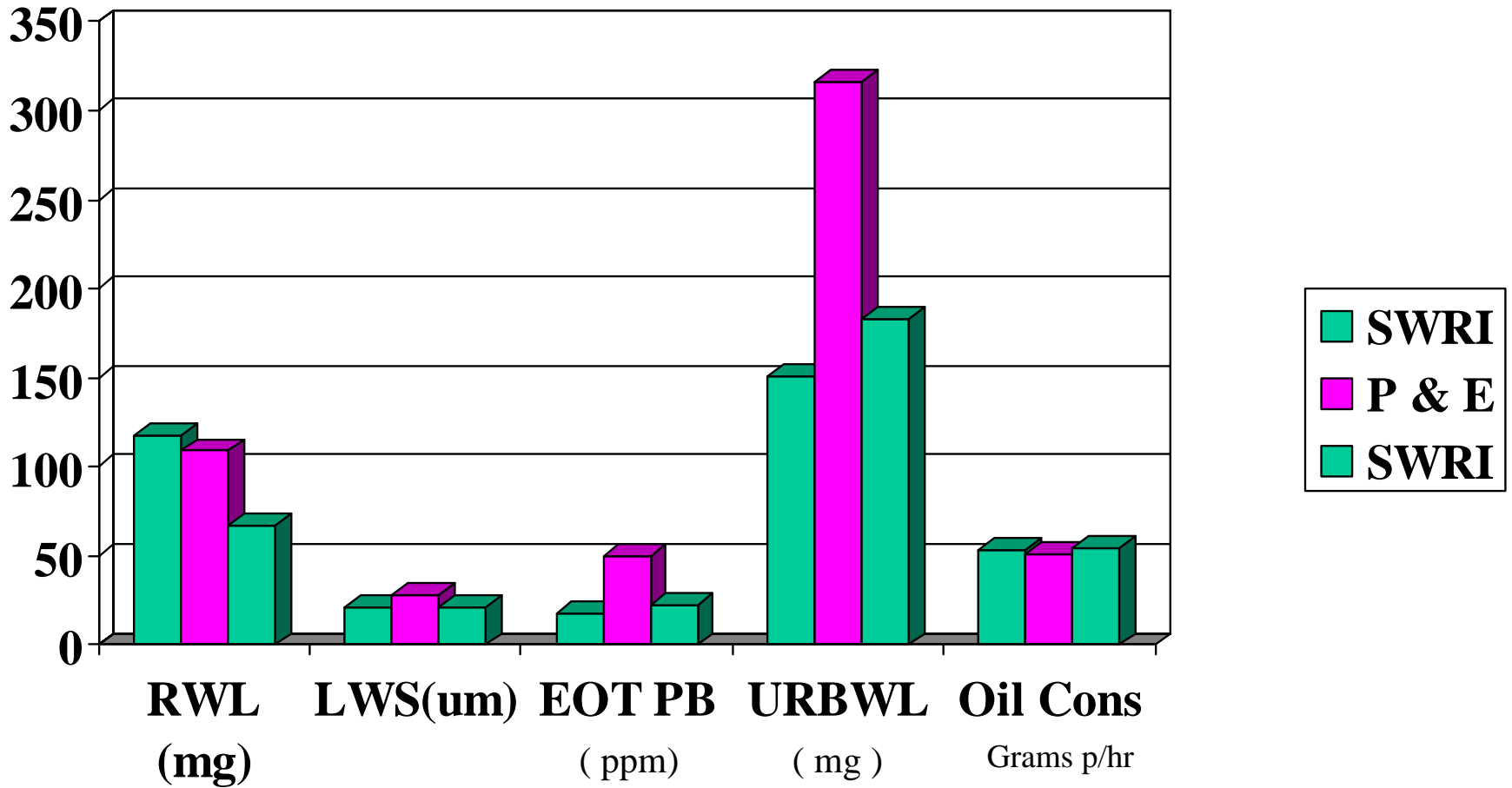


T 10 Matrix Oil D

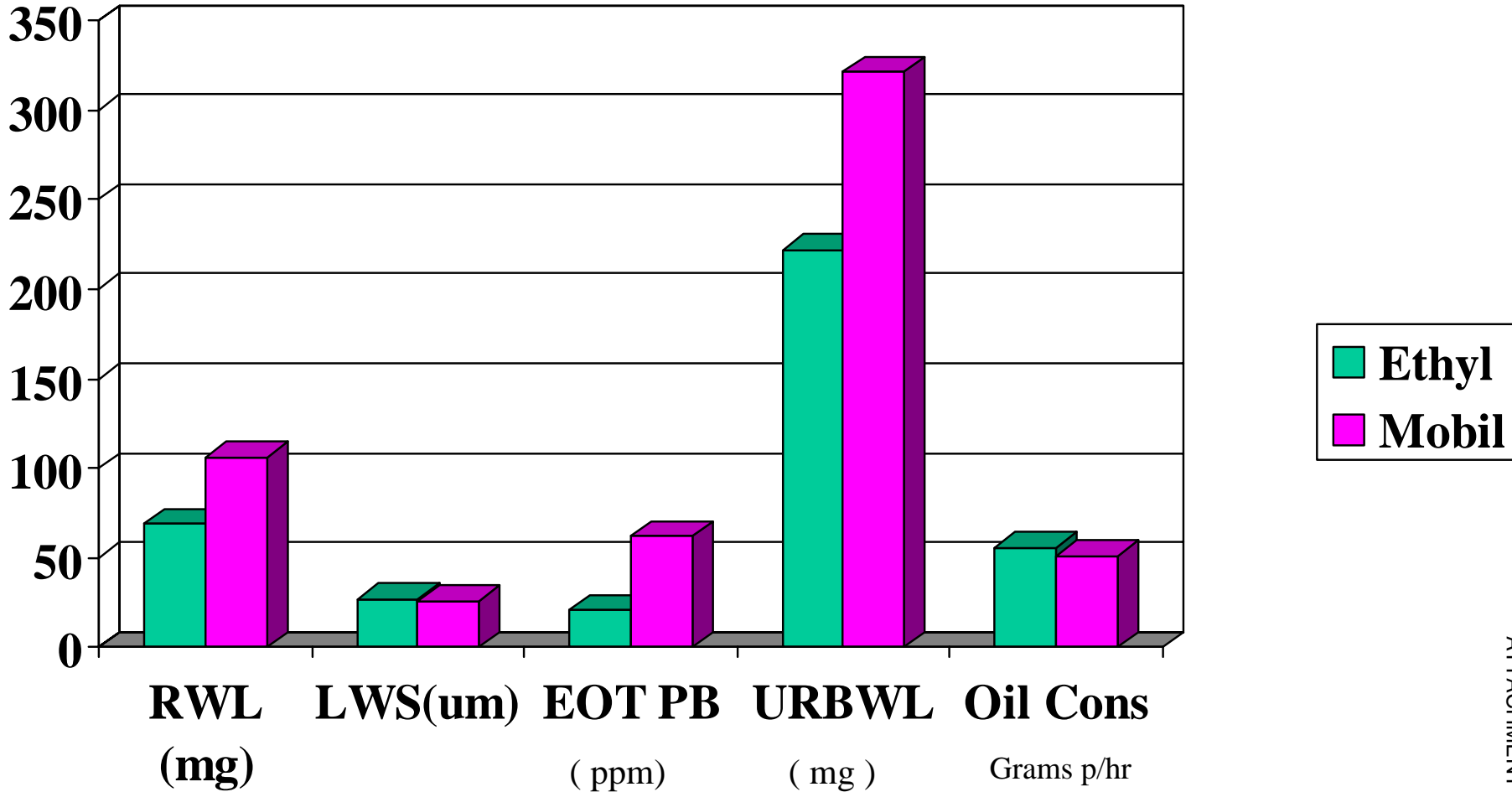


June 15 01

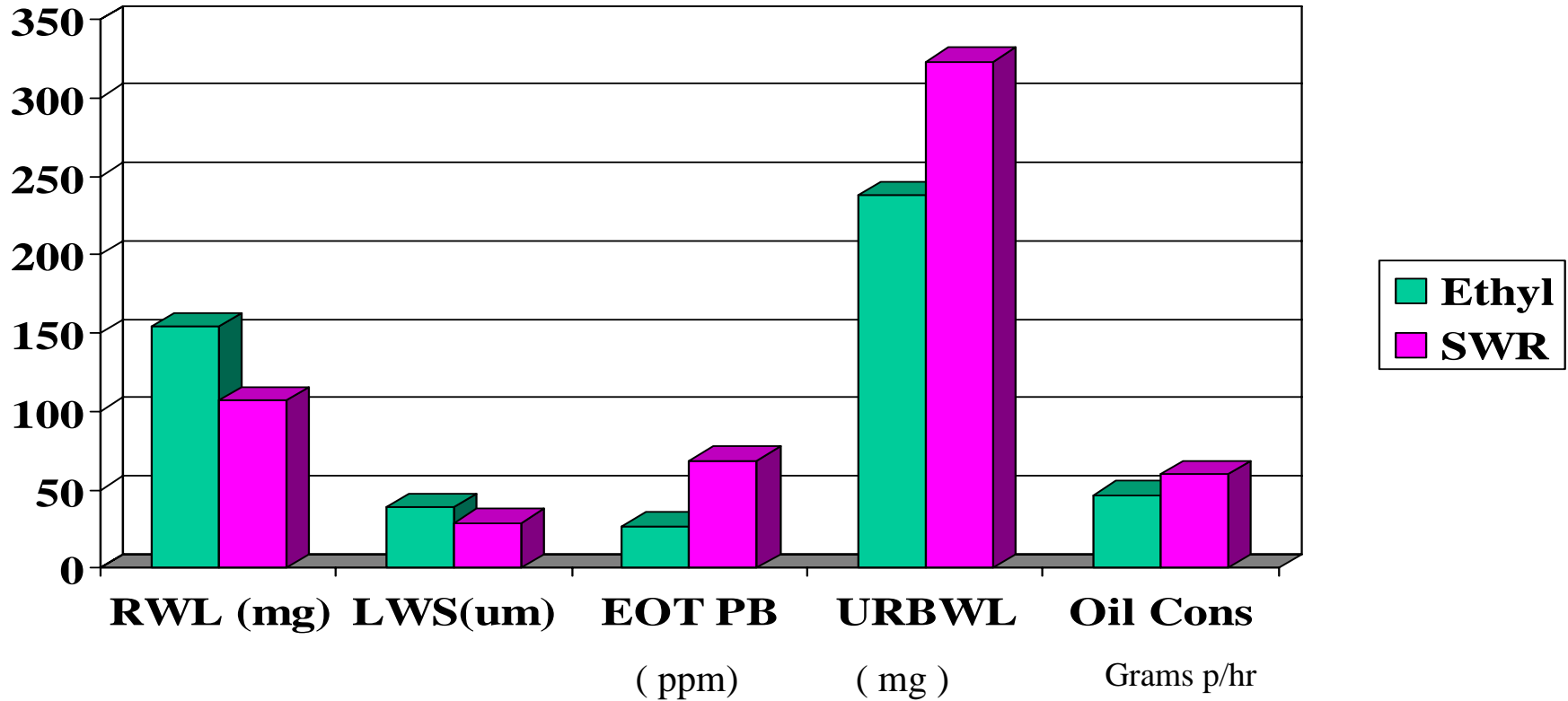
T 10 Matrix Oil E



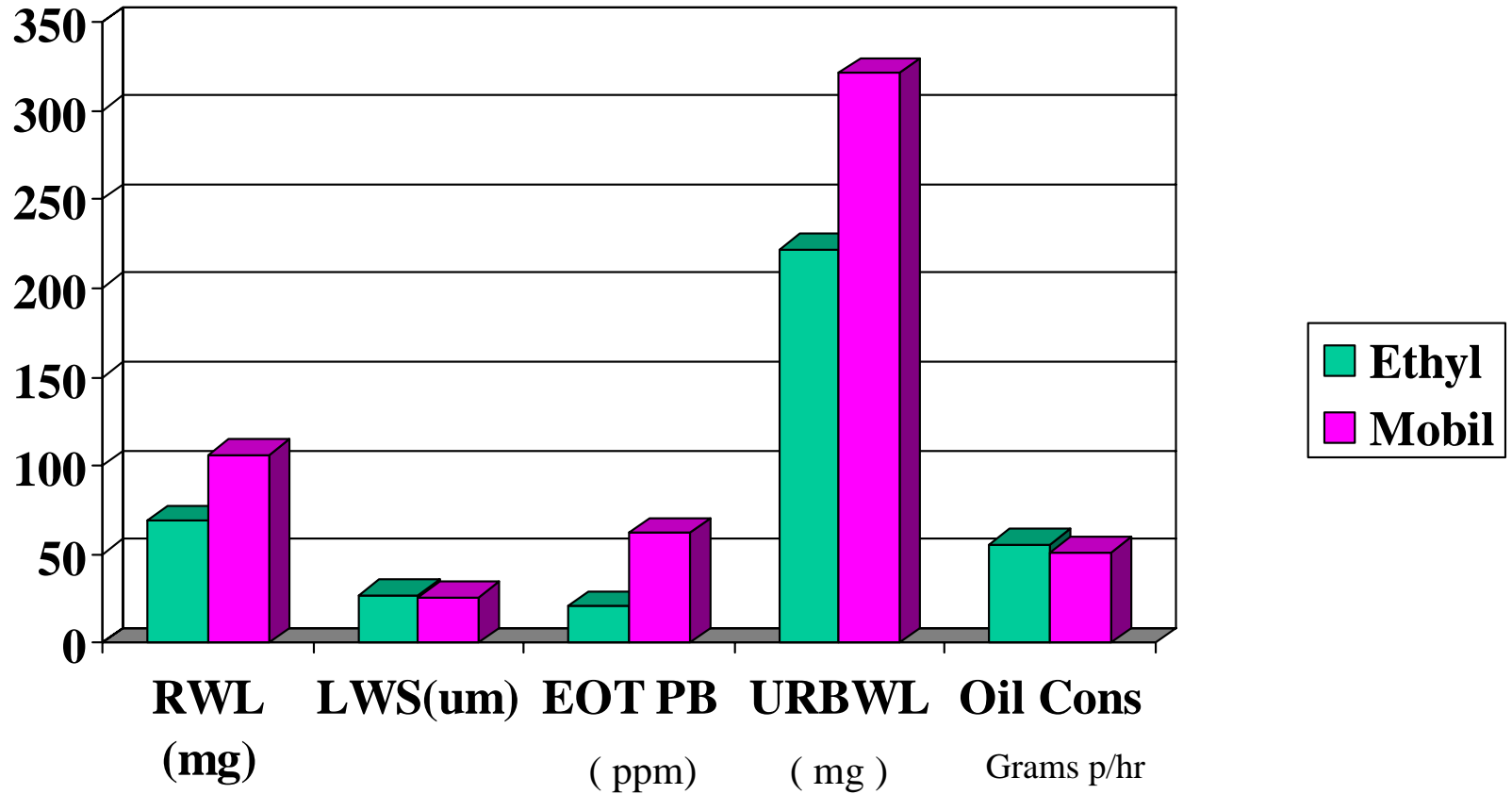
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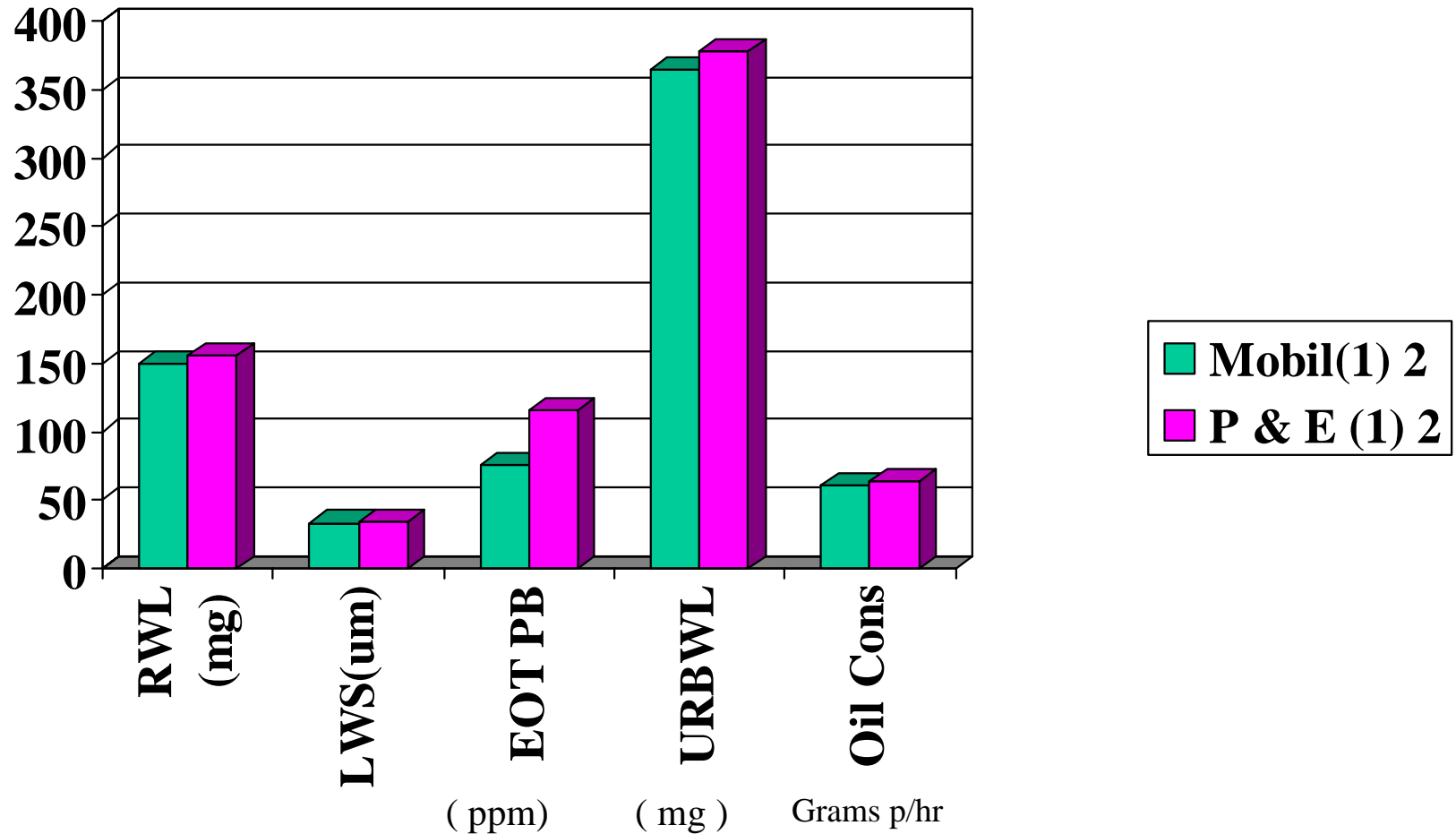
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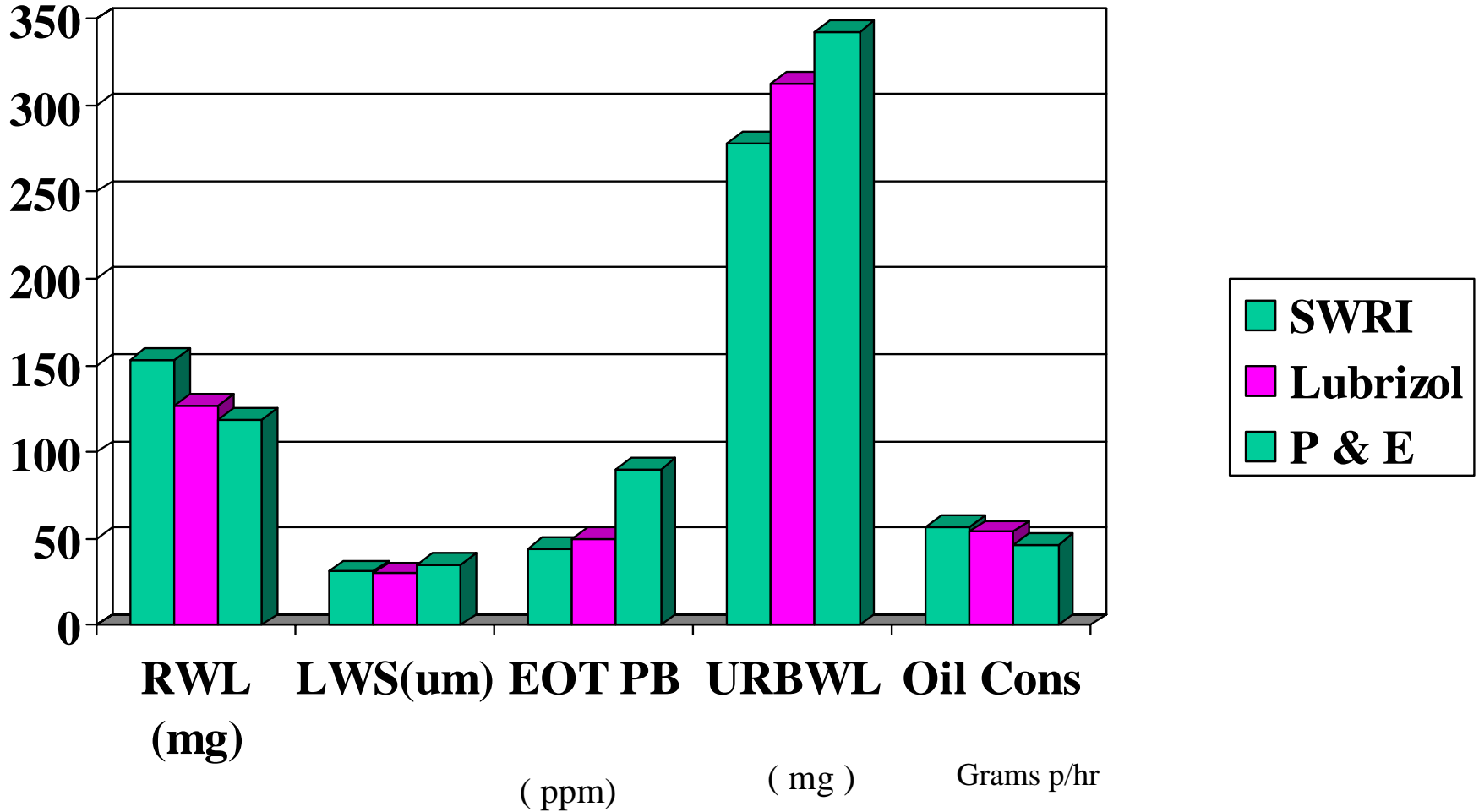
T 10 Matrix Oil F



T 10 Matrix Oil H



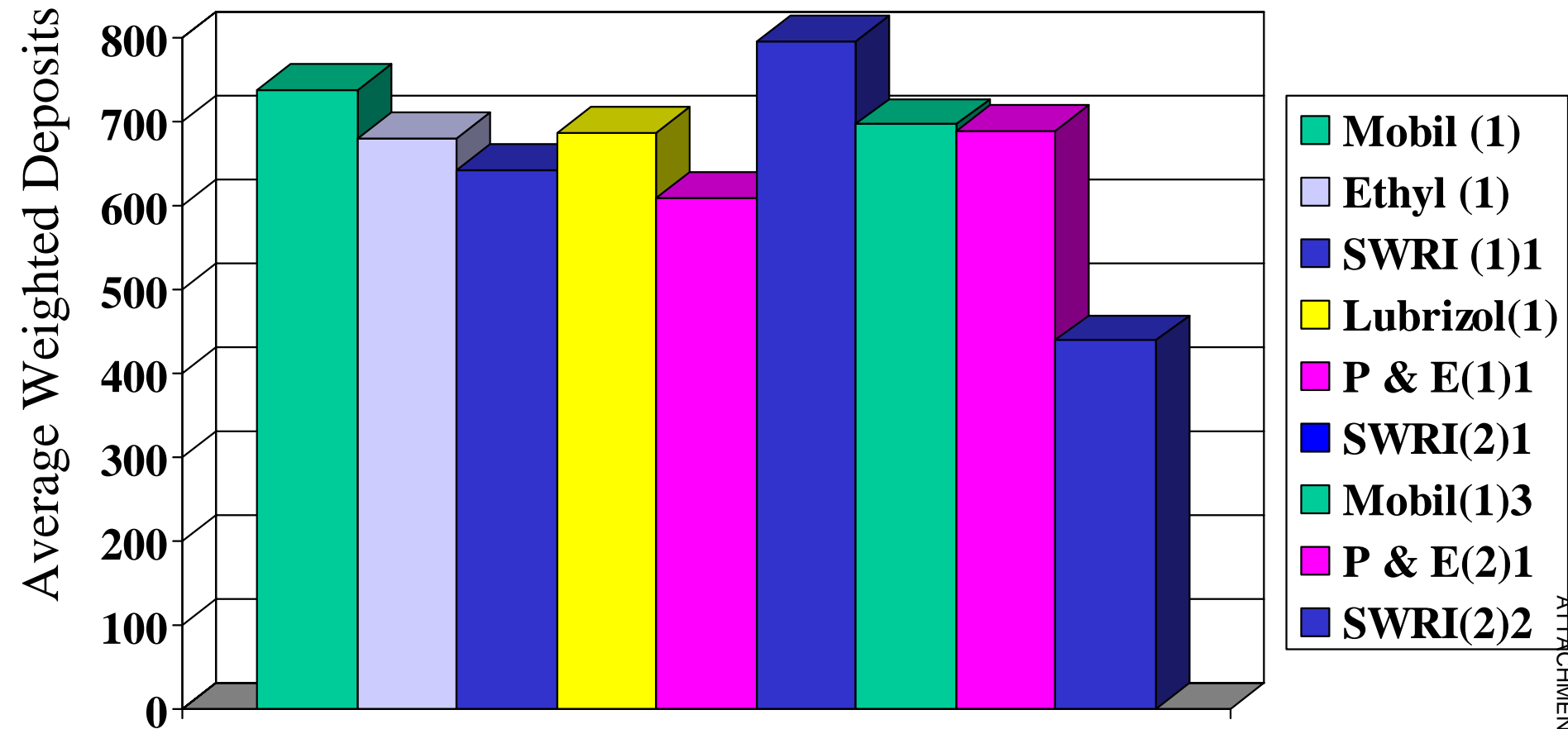
T 10 Matrix Oil J





T10 Matrix Data

Average Weighted Deposits Oil A

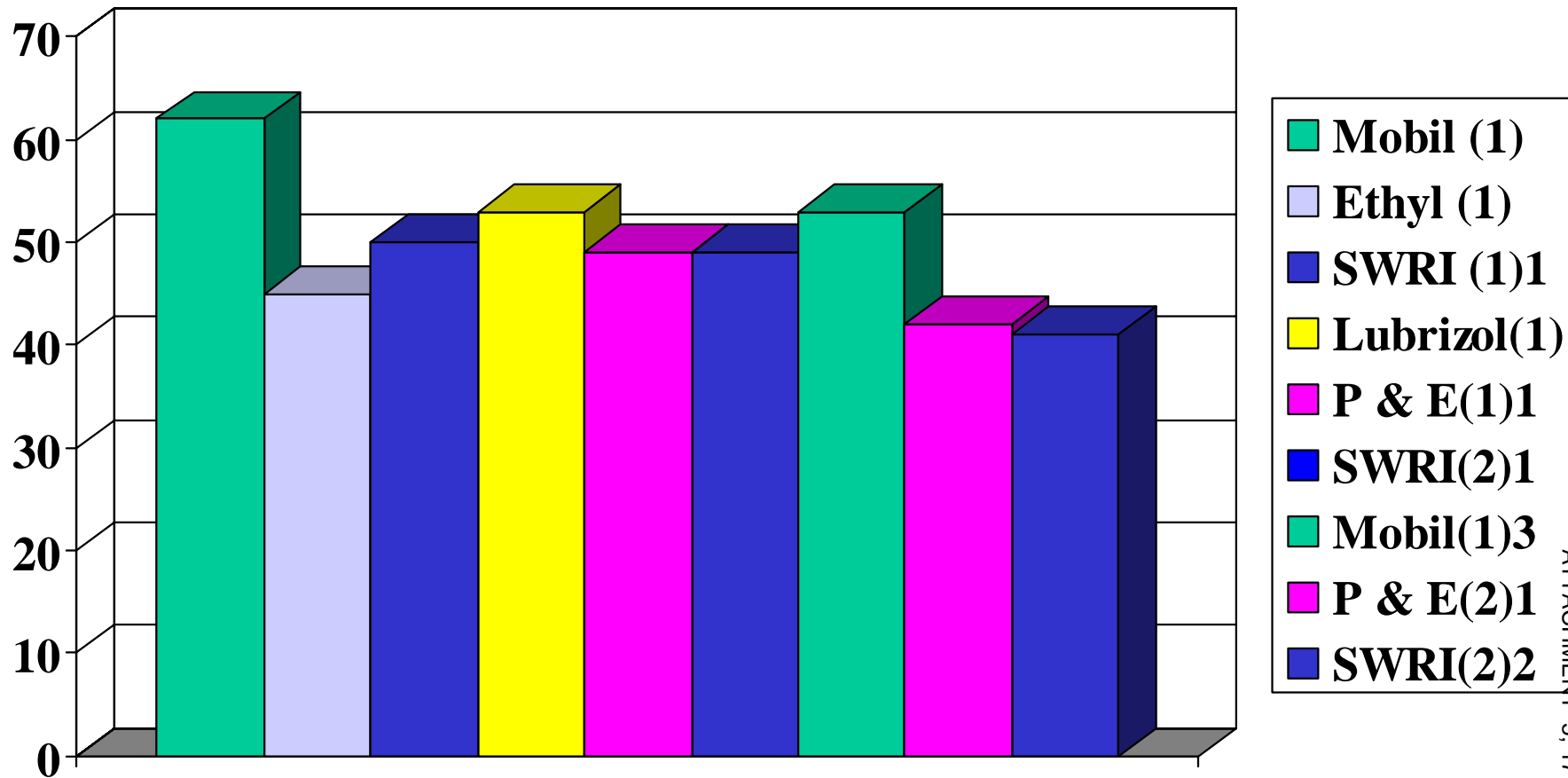




T10 Matrix Data

Top Groove Carbon Oil A

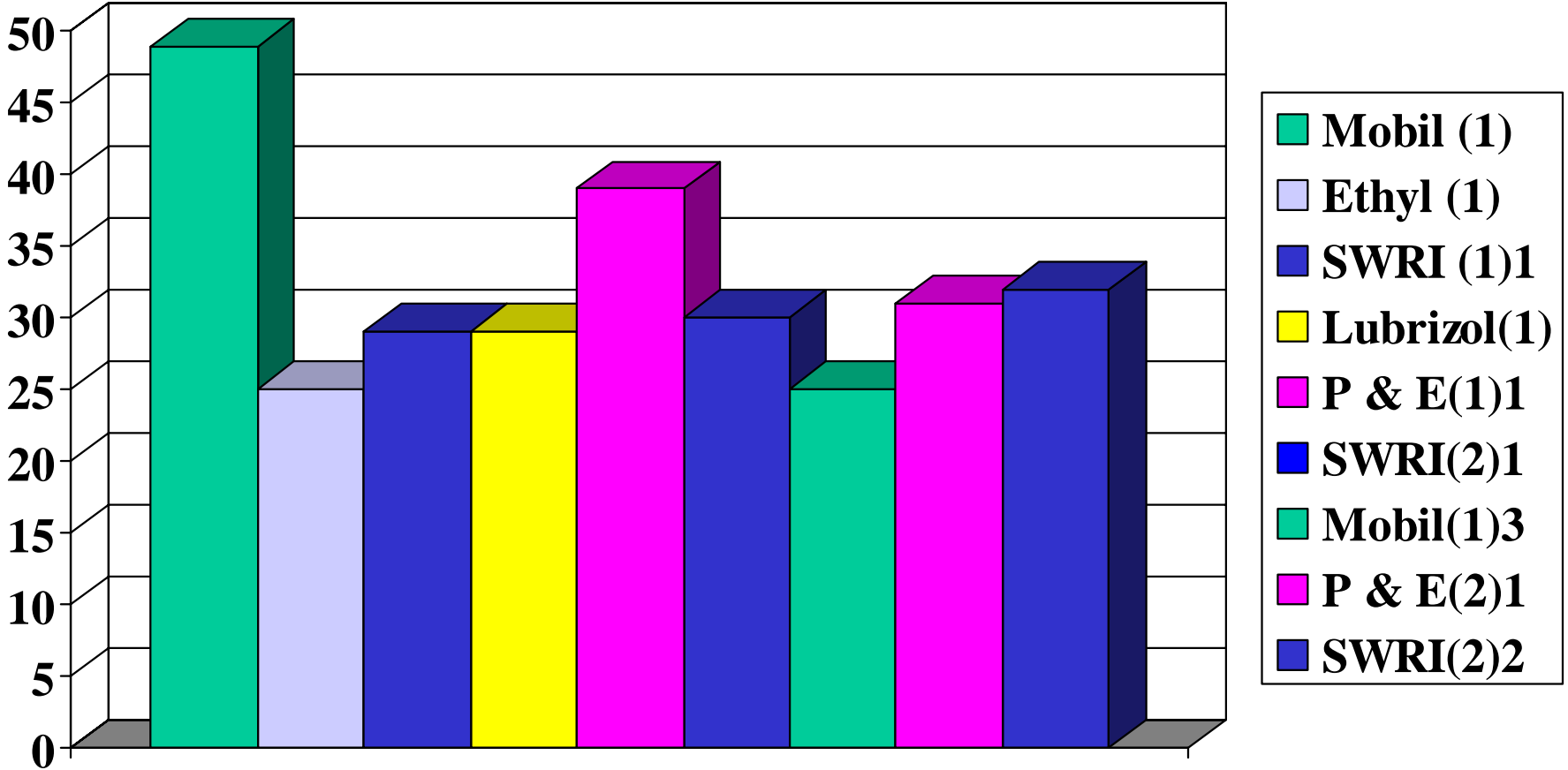
Average Top Groove Carbon





T10 Matrix Data

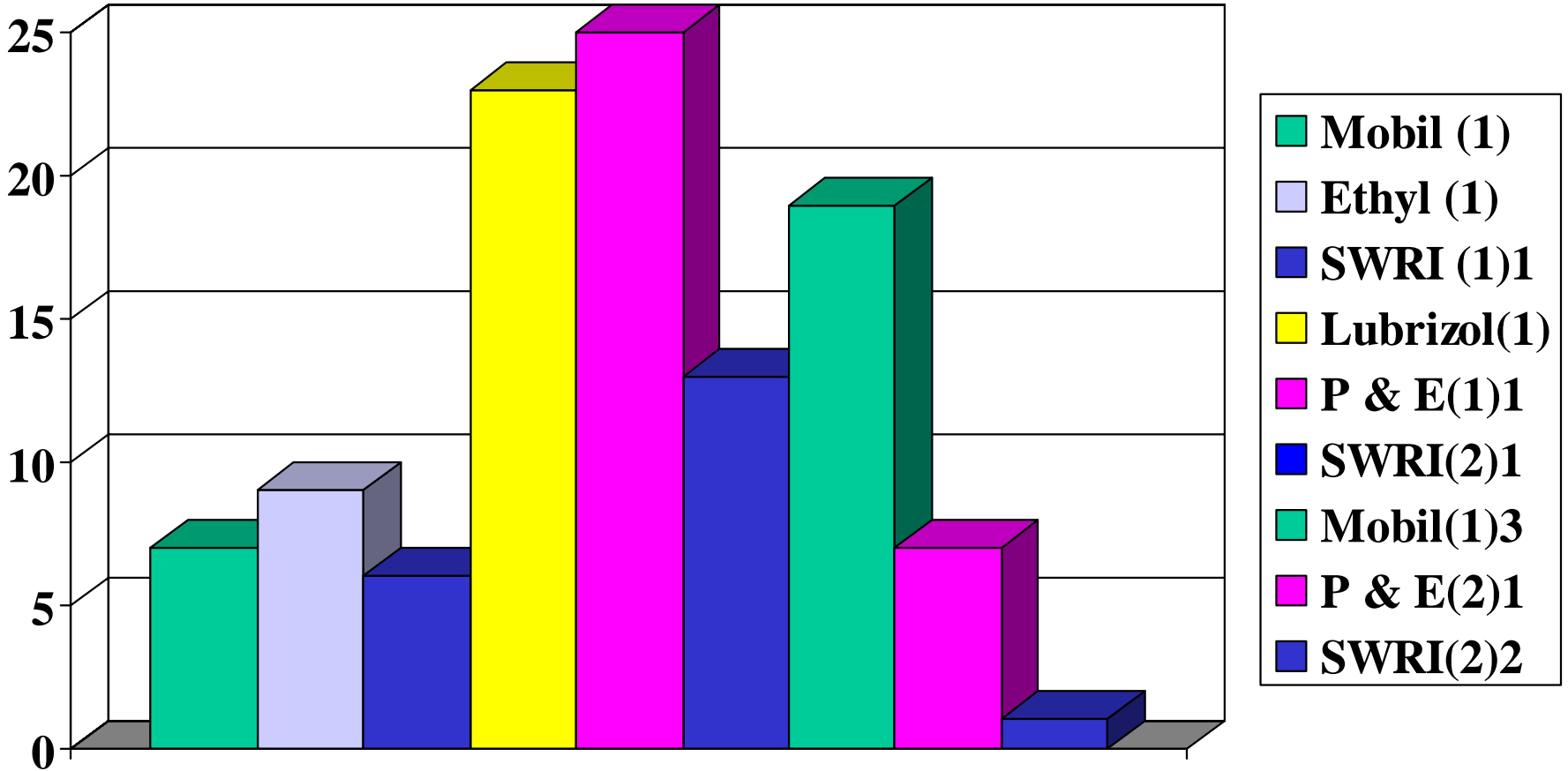
Top Land Carbon Oil A



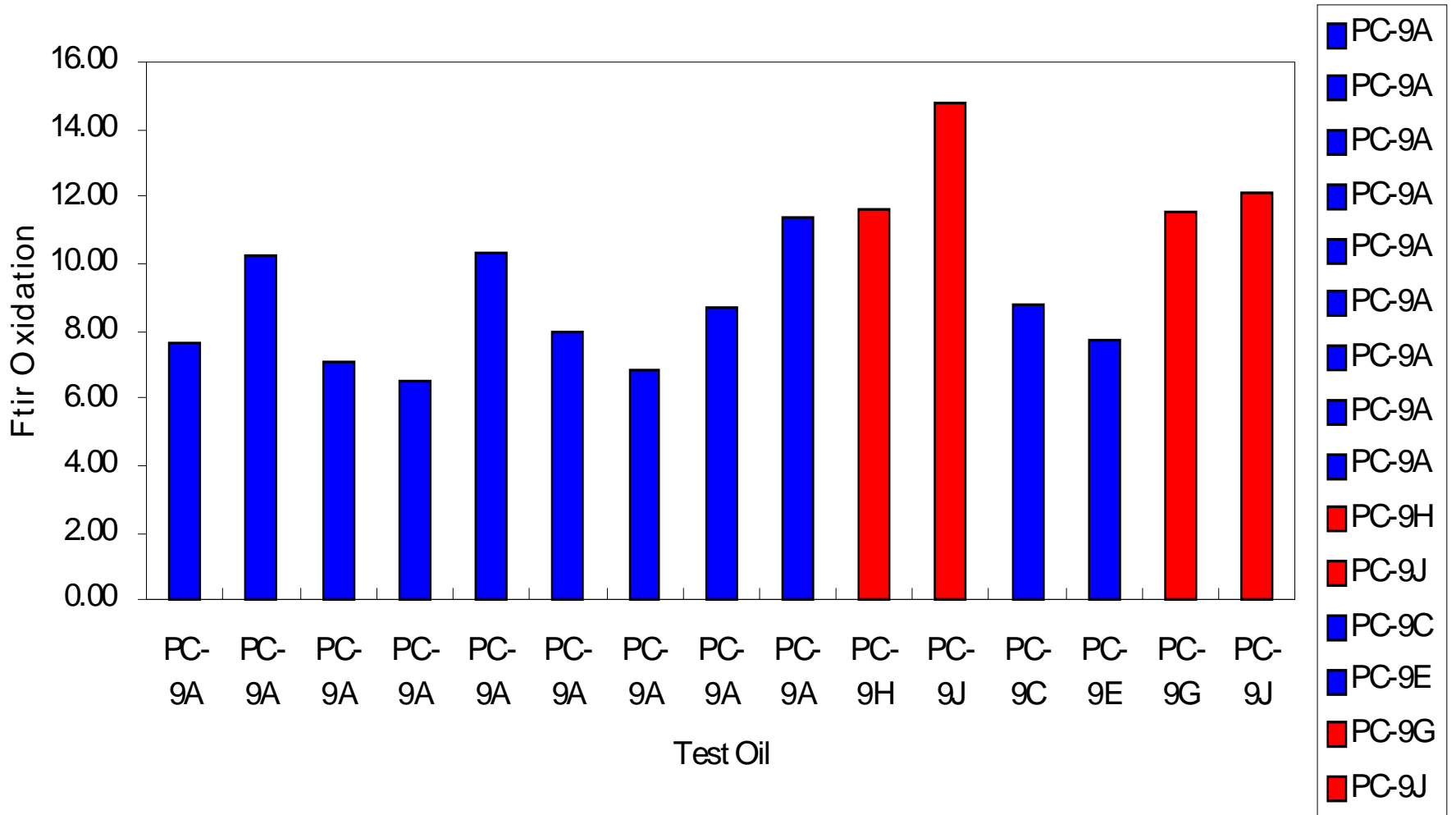


T10 Matrix Data

Average Undercrown Deposit Oil A

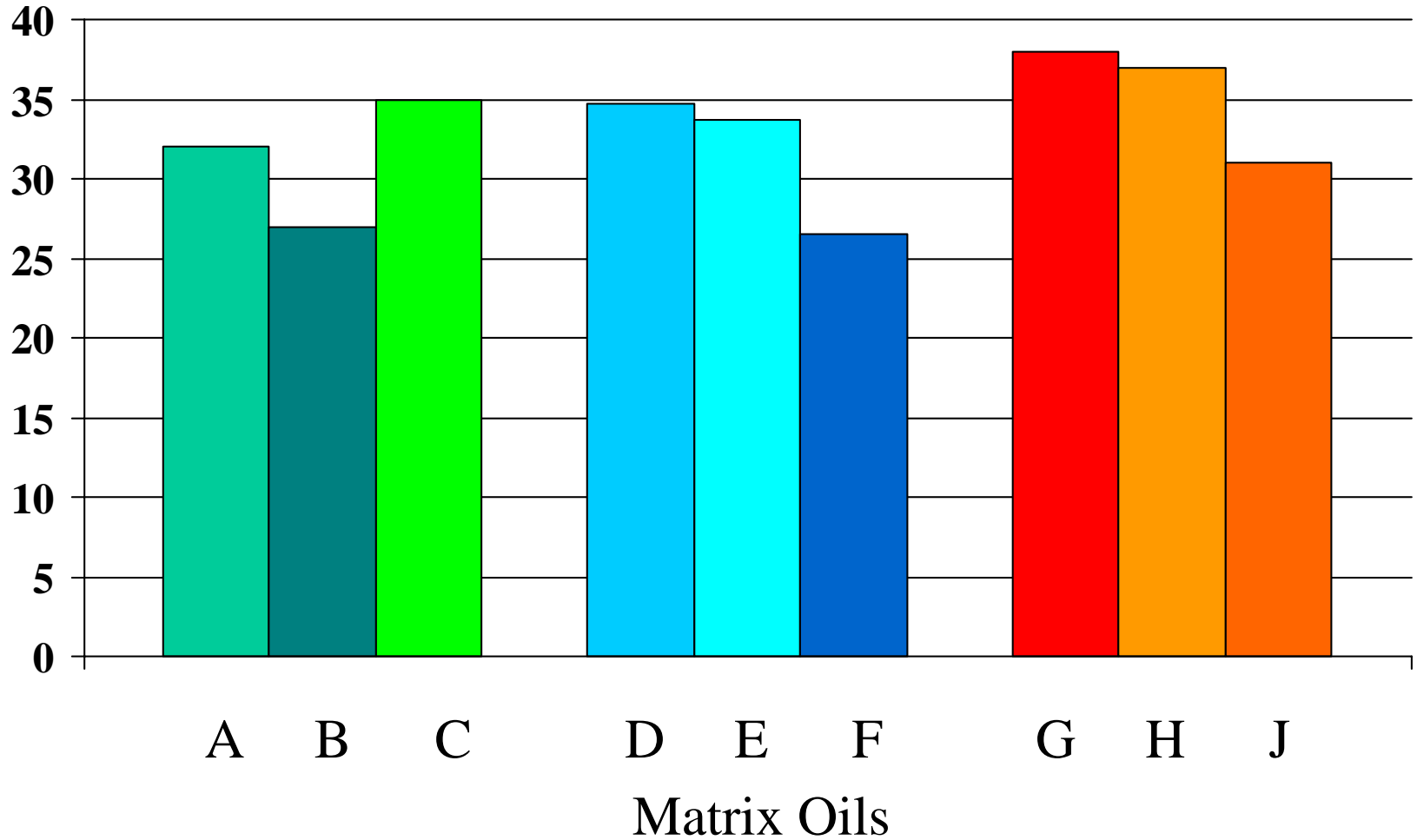


Mack T-10 Method 2
FTIR Oxidation





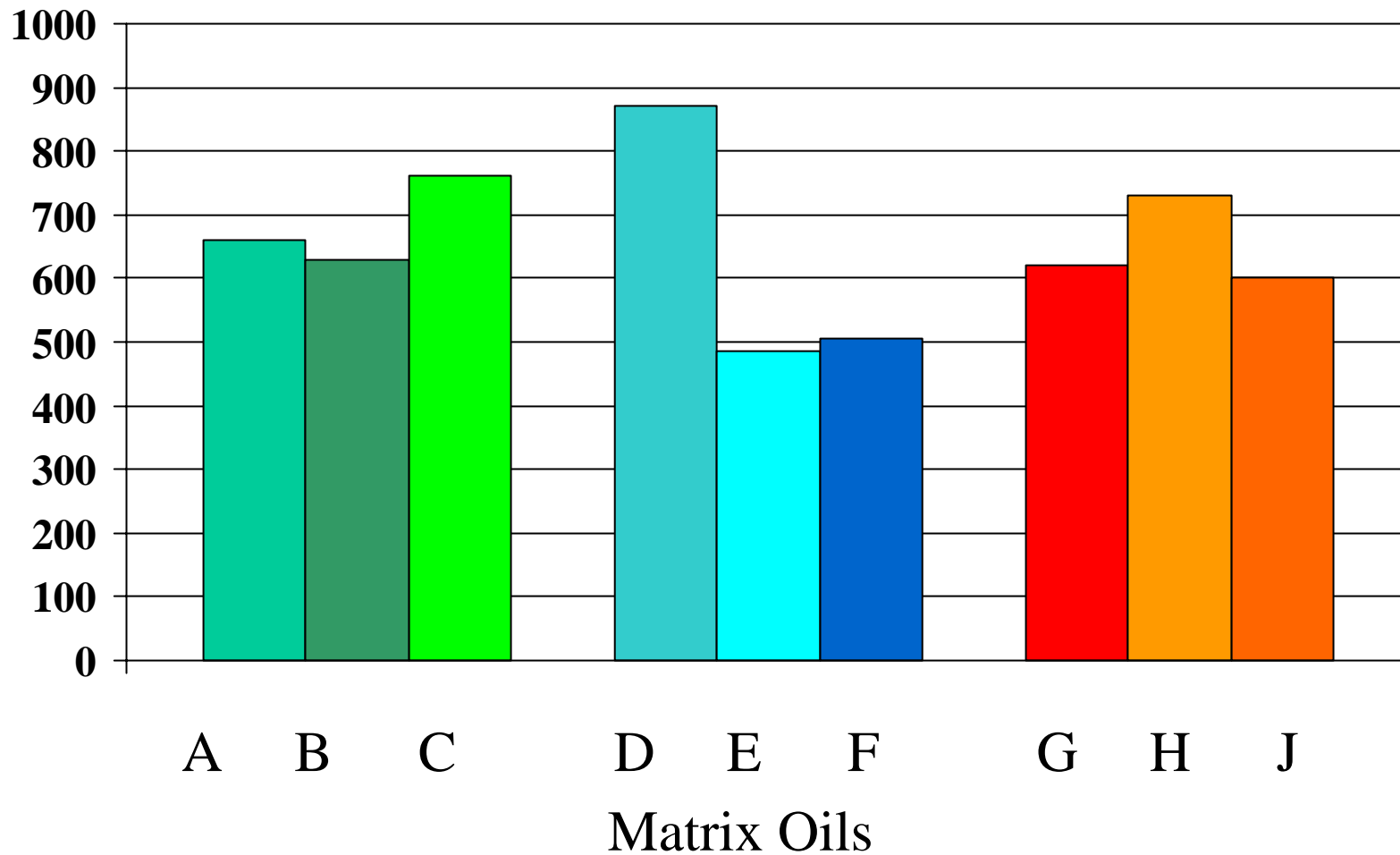
T 10 Matrix Top Land Carbon





T 10 Matrix

Average Weighted Deposits



T10 “to do” List

1. Complete missing data – **July 13th** (Matrix Labs A, B, D, F, G)
 - a. Oil consumption based on final eight 25 hour periods
 - b. Any remaining drains to SwRI for oxidation analysis
 - c. Samples to Chris May for LOTRUO.
2. Update database with all parameters – **July 16th** (Jeff C)
3. Complete statistical work – **July 23rd** (Jim R)
4. Statisticians meet or conference call – **July 25th** (Jim R / Dennis M / Phil S)
5. Complete work required to resolve lab severity differences already identified. – **Teleconference week of July 16th**
Control CO2 Intake and Exhaust for both Phase I & II (Task Force O&H)
6. Complete template & MAD surveys – **July 31st** (Jim R)
(ACC Members)

7. Test / Template approval
 - a. ACC Approve Template – **August 8th & 9th** (ACC)
 - b. Task force approve test - **August 9th** (Task Force)
 - c. HDEOCP approve test & Discuss limits
& Issue exit ballots - **August 15th** (HDEOCP)
 - d. TMC approve test – Upon approval of HDEOCP (TMC)
8. ACC approve registration - **August 16th - 22nd** (ACC)
9. RSI commences test registration - **Upon ACC approval** (RSI)
10. HDEOCP sets limits and prepares ballot - **September 5th** (HDEOCP)
11. HDEOCP issues “B” Ballot - **September 10th**

Preliminary Summary of the Mack T10 Precision/**BOI** Matrix

jar for the PC-9 Statistical Task Group
Presented to the HDEOCP July 11, 2001

Changes due to deletion of CMIR 38815 or other correction noted

Summary

- This is a very preliminary summary. One more test will enter the data set. Others might leave. When the Task Force approves the data set, the statistical task group will reach consensus analysis.
- Delta lead {DELETED "and top ring weight loss"} benefits from natural log transformations.
- No other transformations seem necessary.
- The matrix data were not evaluated for ACC precision requirements or for determination of LTMS parameters.

Summary

(continued)

- Labs had significant effects for delta lead, upper bearing weight loss, and oil consumption.
- The Technology by Base Oil interaction was significant for delta lead.
- Technology had a significant effect for delta lead and upper bearing weight loss.
- Base Oil had a significant effect for cylinder liner wear.
- Observations with large Studentized residuals were seen for delta lead, top ring weight loss, and oil consumption.

Table 1. Mack T10 Precision Matrix Plan

Base Oil	Technology		
	X	Y	Z
Base Oil 1	PC-9A	PC-9D	PC-9G
Base Oil 2	PC-9B	PC-9E	PC-9H
Base Oil 3	PC-9C	PC-9F	PC-9J

Lab/Stand						
Lab 1		Lab 2	Lab 3		Lab 4	Lab 5
1	2	3	4	5	6	7
A	A	A	A	A	A	A
G	A	G	D	A	A	D
E	E	B	H	E	H	B
C	J	F	C	J	F	J

Table 2. Mack T10 Precision Matrix Data

from TMC 07/05/01

Obs	CMIR	Lab	Stand	EOT Date	Oil	Tech	Base Oil	DPBFNL	ABWLU	ATRWLFNL	CLWLFNL	OILCON
1	38809	A	1	20001219	A	X	1	23	206	158	33	52
2	38810	A	2	20010313	A	X	1	19	159	168	38	46
3	38942	A	2	20010408	A	X	1	16	182	87	27	41
* 4	38815	B	4	20001231	A	X	4	11	165	349	24	32
4	41410	B	1	20010618	A	X	1	34	229	140	35	42
5	38811	D	1	20001224	A	X	1	12	195	139	38	52
6	38814	F	1	20001211	A	X	1	33	257	139	36	79
7	41135	F	1	20010611	A	X	1	28	248	128	26	60
8	38951	G	2	20010330	A	X	1	37	218	125	33	53
9	40230	G	2	20010602	A	X	1	25	197	108	34	48
10	40919	B	1	20010529	B	X	2	34	234	121	24	54
11	38943	D	1	20010401	B	X	2	17	182	125	31	44
12	38939	A	1	20010305	C	X	3	33	243	116	25	63
13	38949	G	1	20010420	C	X	3	77	336	133	35	66
14	38957	B	1	20010403	D	Y	1	25	183	204	46	54
15	38946	G	1	20010517	D	Y	1	206	344	108	33	71
16	38937	A	1	20010329	E	Y	2	18	151	118	21	53
17	38940	A	2	20010528	E	Y	2	22	184	67	20	45
18	38950	G	2	20010512	E	Y	2	52	317	109	28	55
19	38945	D	1	20010215	F	Y	3	21	222	69	27	56
20	38952	F	1	20010419	F	Y	3	62	321	106	26	51
21	38941	A	1	20010422	G	Z	1	71	324	107	29	52
22	38944	D	1	20010504	G	Z	1	27	238	154	39	47
23	38953	F	1	20010217	H	Z	2	73	364	150	33	61
24	38947	G	1	20010318	H	Z	2	115	378	156	34	64
25	38938	A	2	20010504	J	Z	3	44	278	153	31	58
26	38956	B	1	20010509	J	Z	3	50	314	127	30	35
27	38948	G	2	20010419	J	Z	3	90	343	119	35	47

Transformations

- Box-Cox procedure was applied using all matrix data.
- Delta lead {DELETD "and top ring weight loss"} benefits from a natural logarithm transformation.
- No data transformations are indicated for other responses analyzed.

Precision Estimates

- Ln(delta lead): $s_{pp} = 0.35$; $df = 14$
- Upper bearing weight loss: $s_{pp} = 38$; $df = 14$
- Top ring weight loss: $s_{pp} = 29$; $df = 14$
- Cylinder liner wear: $s_{pp} = 4$; $df = 14$
- Oil consumption: $s_{pp} = 9$; $df = 14$

Where

- s_{pp} = Pooled standard deviation (Root MSE from the fitted model) assuming that lab differences are minimized by an LTMS severity adjustment system.
- df = Degrees of freedom.

Ln(Delta Lead)

Summary of Model Fit

- Model factors include Laboratory (A,B,D,F,G), Technology (X,Y,Z), Base Oil (1,2,3) and Technology by Base Oil interaction.
- Technology, Technology by Base Oil interaction, and Lab were significant.
 - Root MSE from the model was 0.36 (14 df).
 - The R^2 for the model was 0.85.
 - Figure 1 illustrates the least squares means by oil.
 - Figure 2 shows the least squares means for labs.
 - From residual analyses:
 - Log transformation was appropriate.
 - The two observations with Oil D had large Studentized residuals.

Figure 1
Least Squares Means for Oils

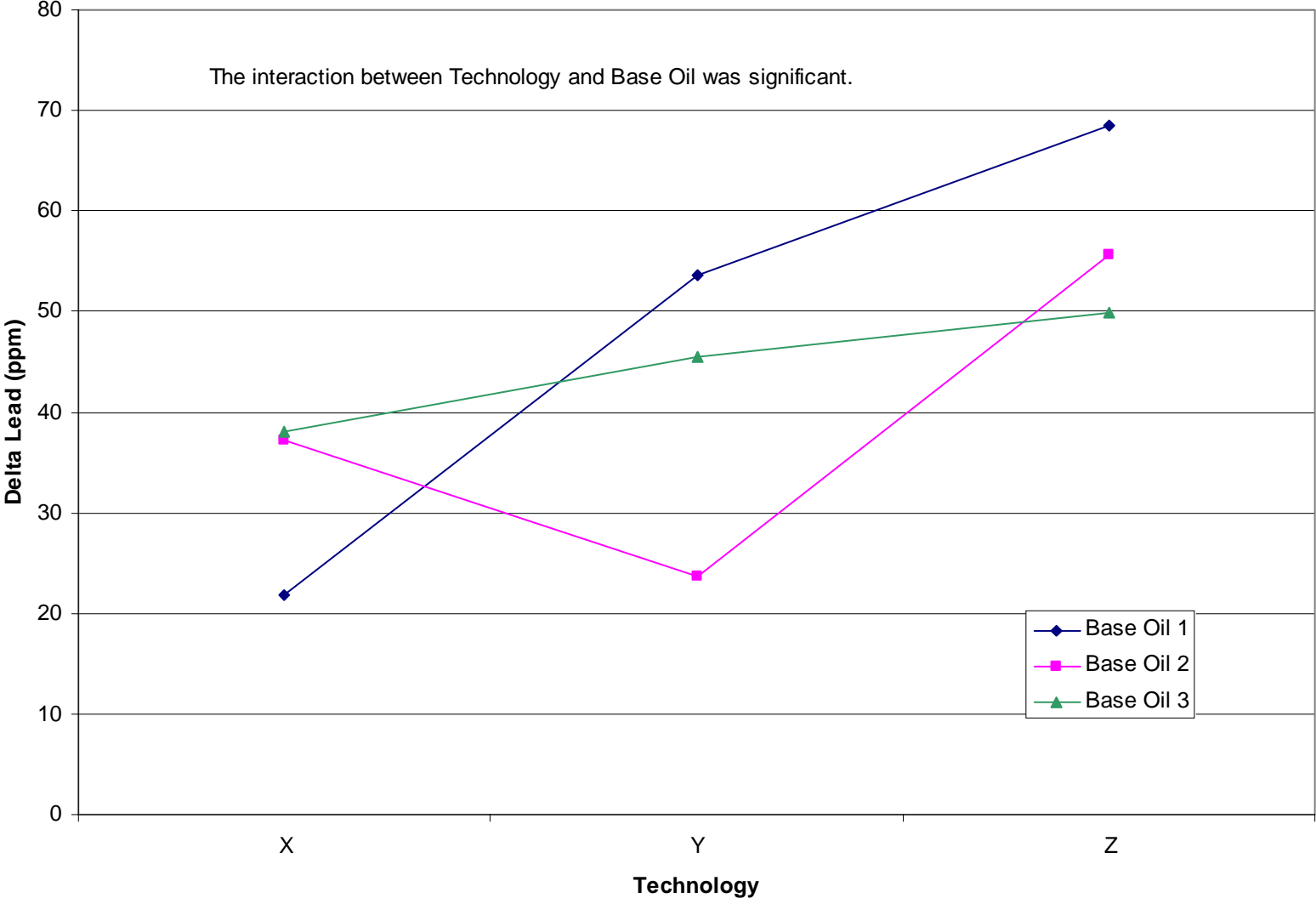
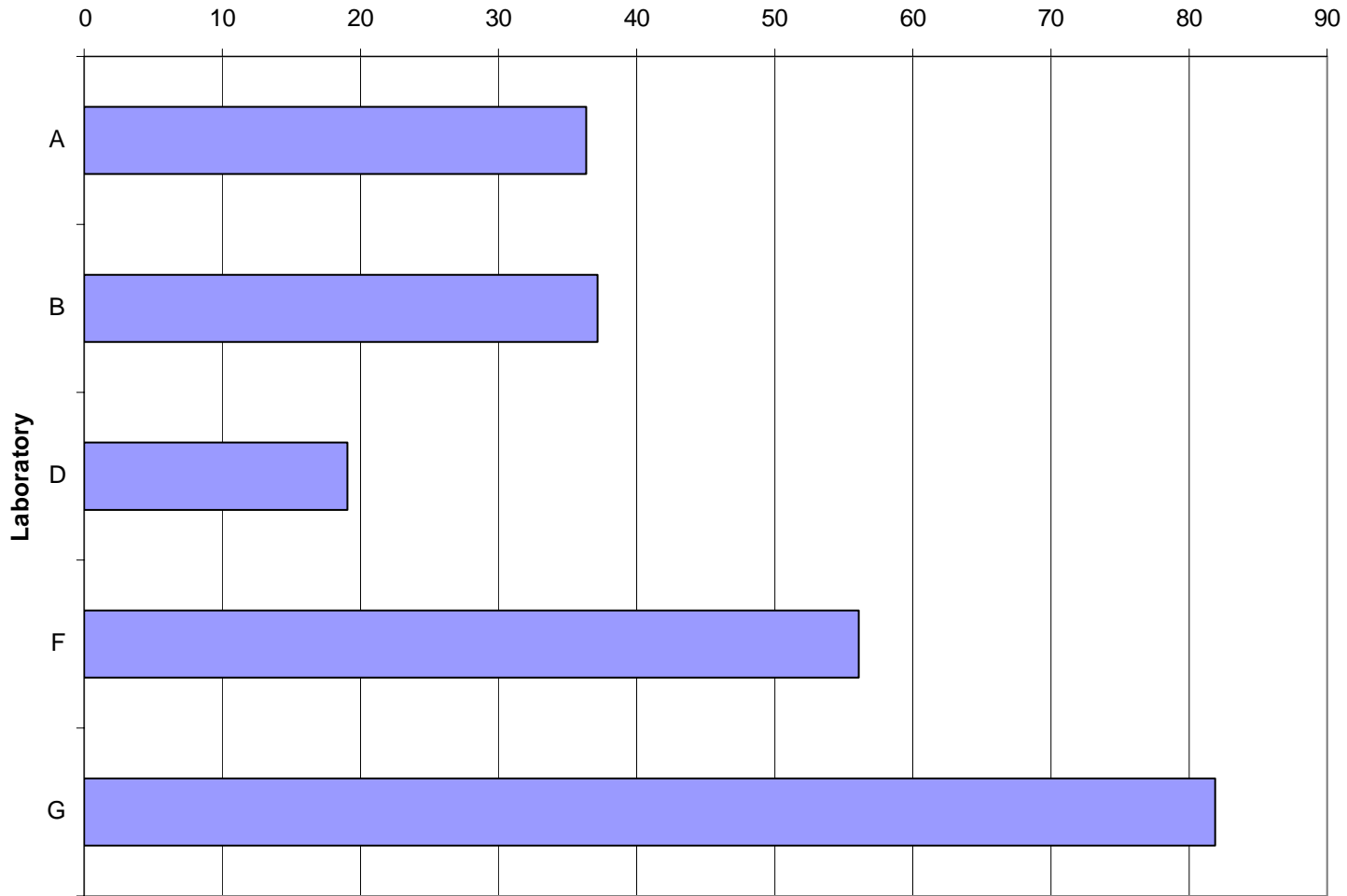


Figure 2
Least Squares Means for Labs
Delta Lead (ppm)



Upper Bearing Weight Loss

Summary of Model Fit

- Model factors include Laboratory (A,B,D,F,G), Technology (X,Y,Z), Base Oil (1,2,3) and Technology by Base Oil interaction.
- Technology and Lab were significant.
 - Root MSE from the model was 38 (14 df).
 - The R^2 for the model was 0.83.
 - Figure 3 illustrates the least squares means by oil.
 - Figure 4 shows the least squares means for labs.
 - From residual analyses:
 - No observations had large Studentized residuals.

Figure 3
Least Squares Means for Oils

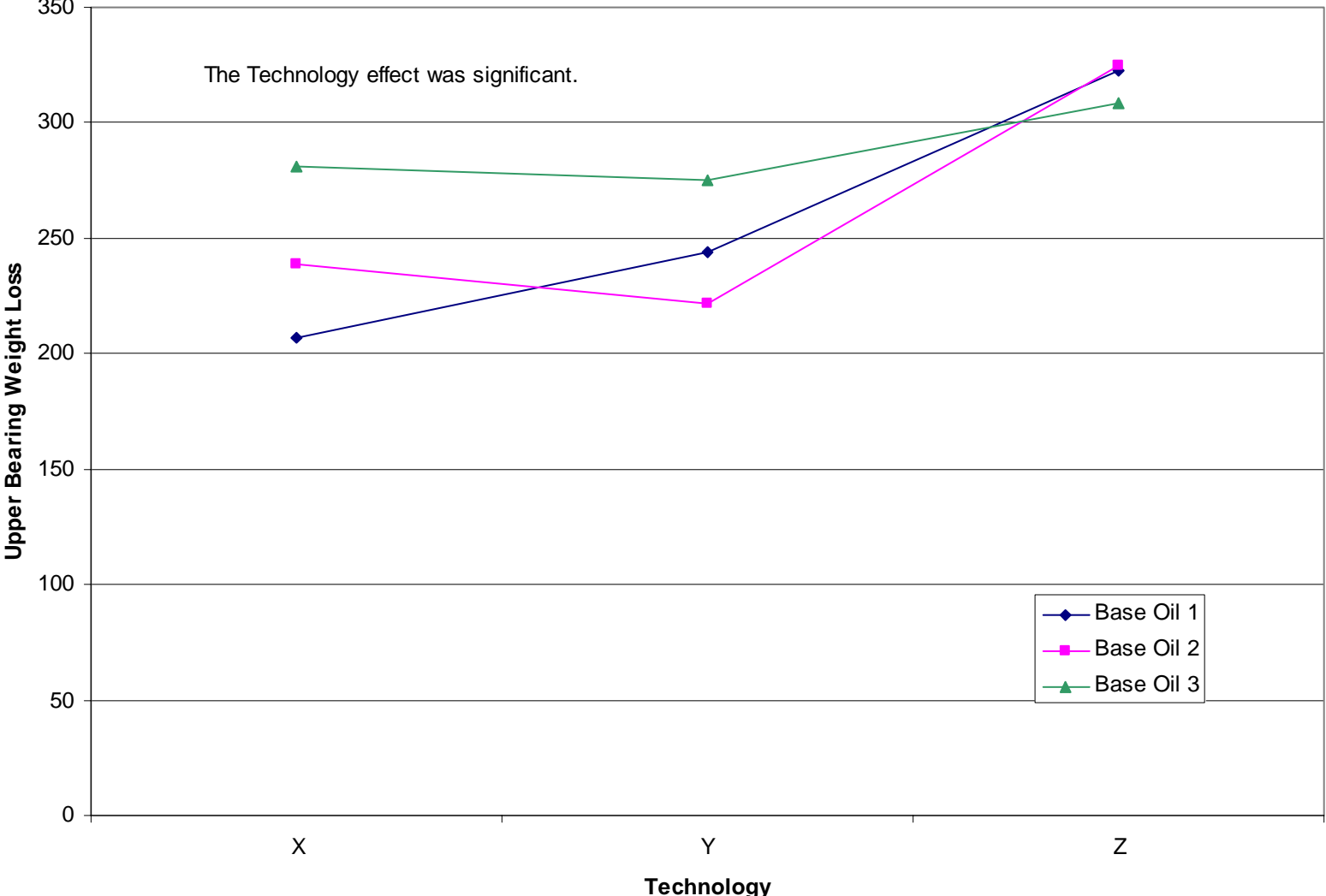
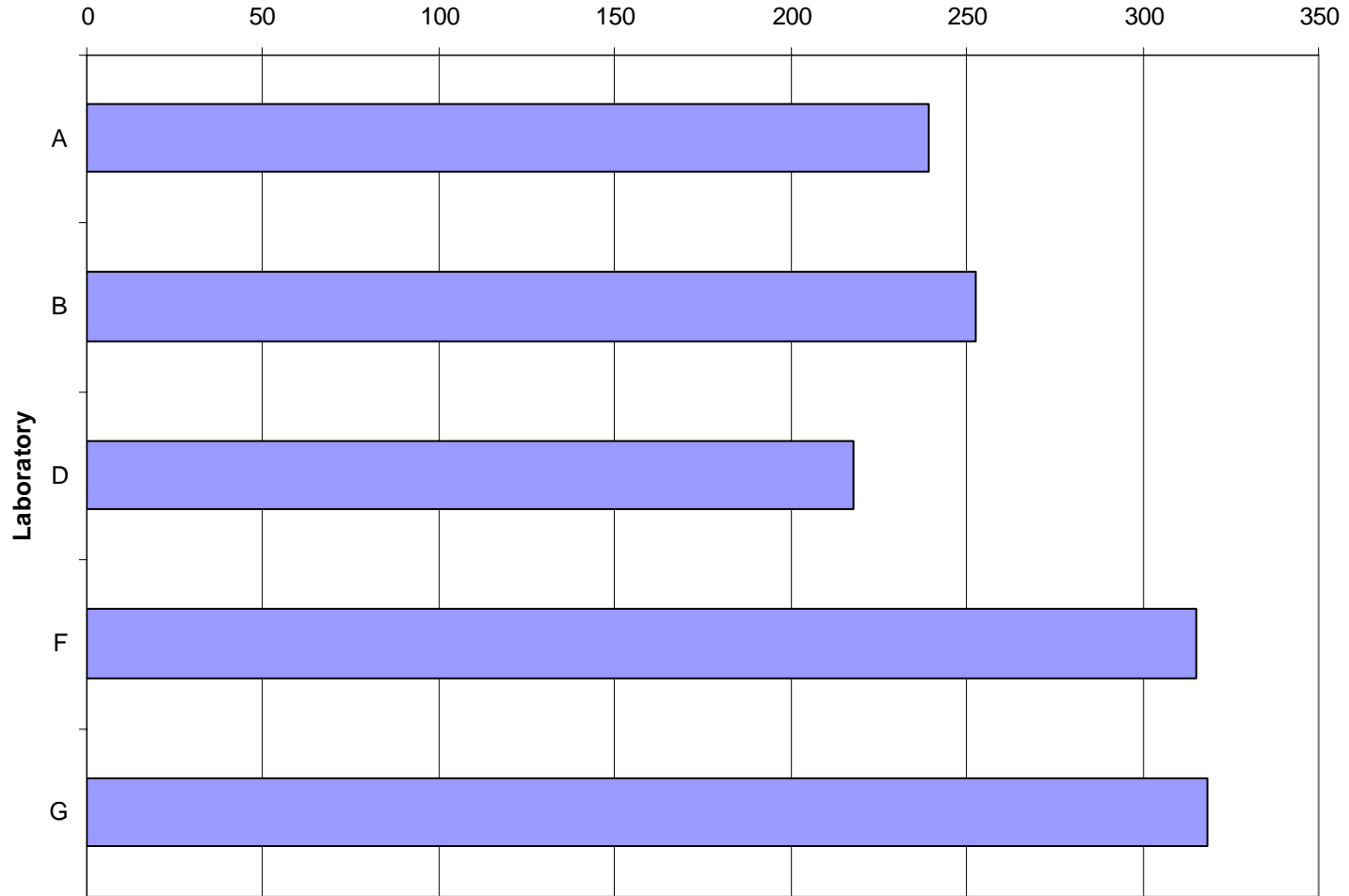


Figure 4
Least Squares Means for Labs
Upper Bearing Weight Loss

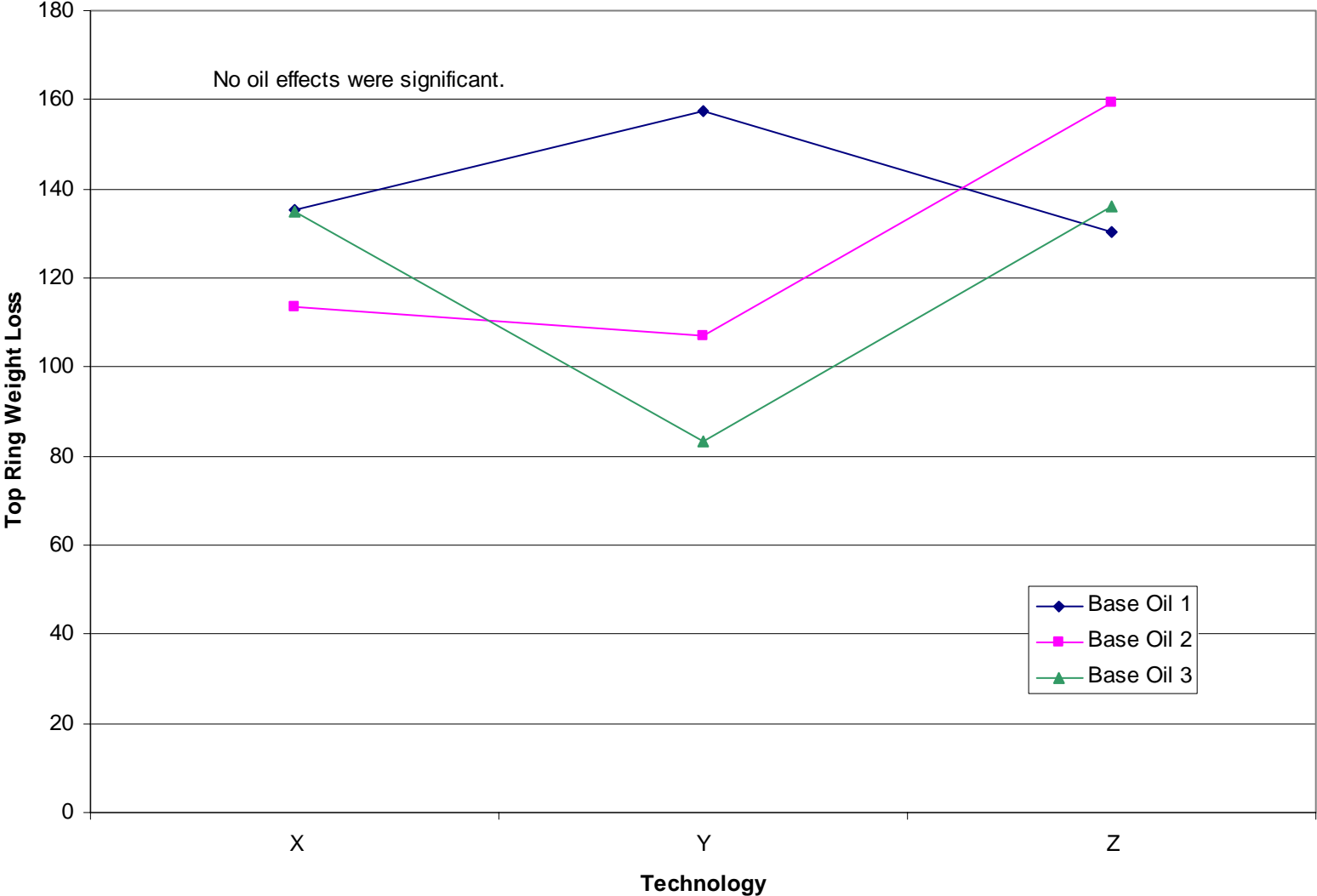


Top Ring Weight Loss

Summary of Model Fit

- Model factors include Laboratory (A,B,D,F,G), Technology (X,Y,Z), Base Oil (1,2,3) and Technology by Base Oil interaction.
- No effects were significant.
 - Root MSE from the model was 29 (14 df).
 - The R² for the model was 0.49.
 - Figure 5 illustrates the least squares means by oil.
 - From residual analyses:
 - There were no large Studentized residuals.

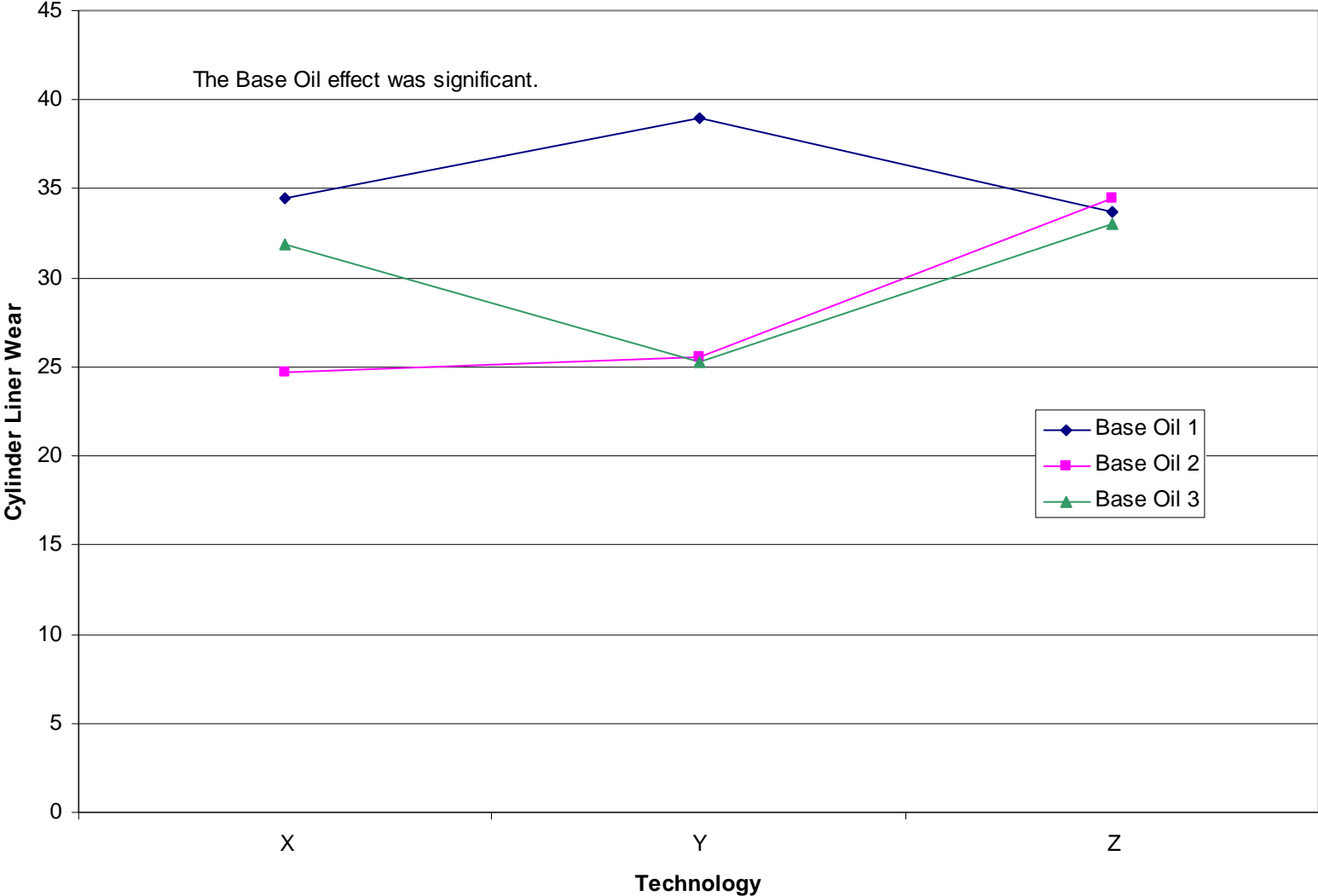
Figure 5
Least Squares Means for Oils



Cylinder Liner Wear Summary of Model Fit

- Model factors include Laboratory (A,B,D,F,G), Technology (X,Y,Z), Base Oil (1,2,3) and Technology by Base Oil interaction.
- The Base Oil effect was significant.
 - Root MSE from the model was 4 (14 df).
 - The R^2 for the model was 0.69.
 - Figure 6 illustrates the least squares means by oil.
 - From residual analyses:
 - There were no large Studentized residuals.

Figure 6
Least Squares Means for Oils



Oil Consumption Summary of Model Fit

- Model factors include Laboratory (A,B,D,F,G), Technology (X,Y,Z), Base Oil (1,2,3) and Technology by Base Oil interaction.
- **No effects were significant.**
 - Root MSE from the model was 9 (14 df).
 - The R^2 for the model was 0.58.
 - Figure 7 illustrates the least squares means by oil.
 - Figure 8 shows the least squares means by Lab.
 - From residual analyses:
 - The first test on Oil A at Lab F had a large Studentized residual.

Figure 7
Least Squares Means for Oils

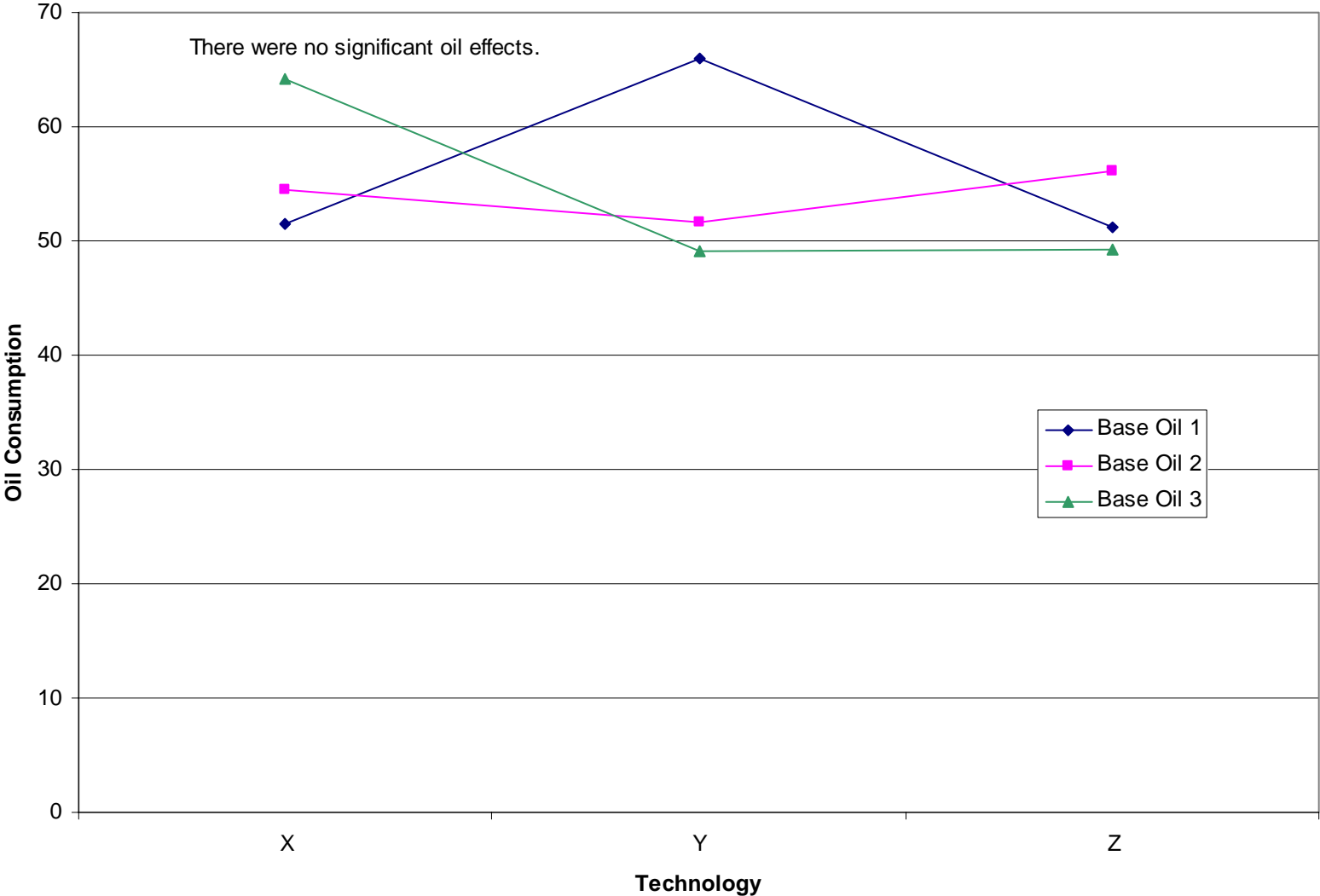
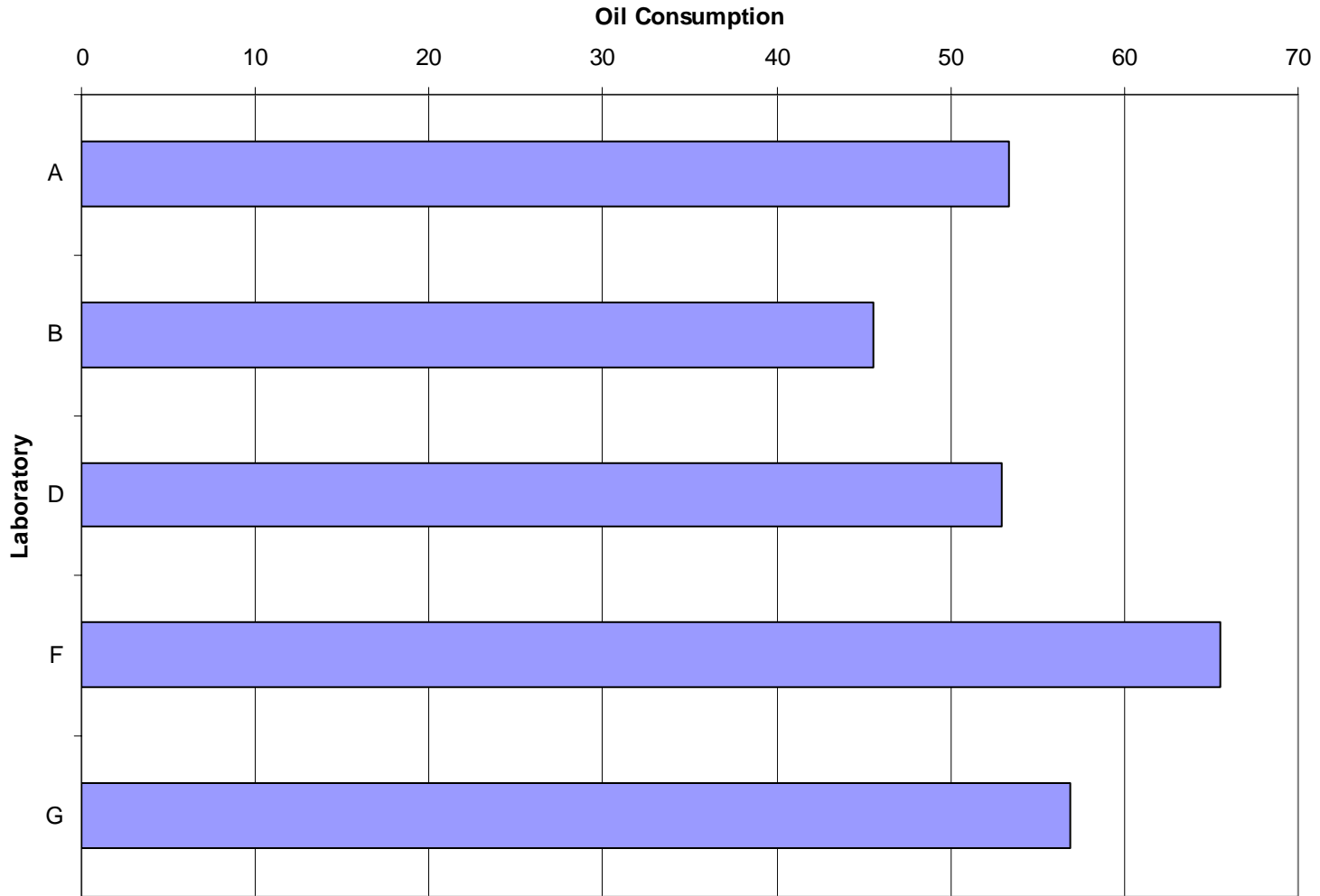


Figure 8
Least Squares Means for Labs





M11 EGR Test Matrix Status

**Presentation to
HDEOCP
July 11, 2001
David M Stehouwer**

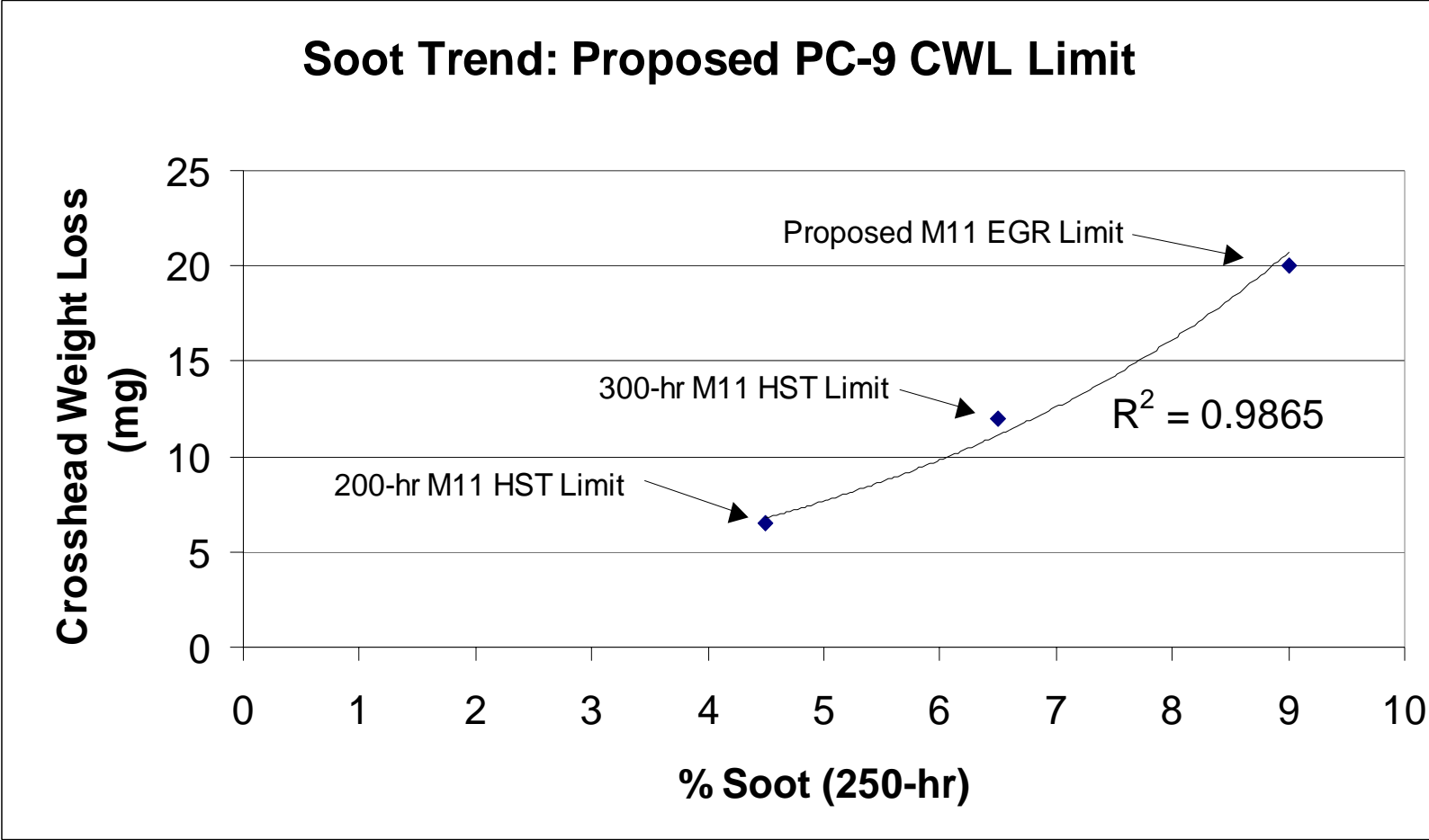
M-11 EGR Test Status

- **M11EGR Task Group met July 9, 10**
- **Agreed to Data Set**
 - ✓ **Removed CWL for soft rocker pads**
 - ✓ **One run on Oil C accepted for OFDP only.**
- **Proposed Limits**
 - ✓ **CWL 20 mg**
 - ✓ **TWL 175 mg (requested ring gap data)**
 - ✓ **OFDP 275 kPa @ 250 hrs**
 - ✓ **ASR 8.0**
 - ✓ **BWL, IAS: Report Value**

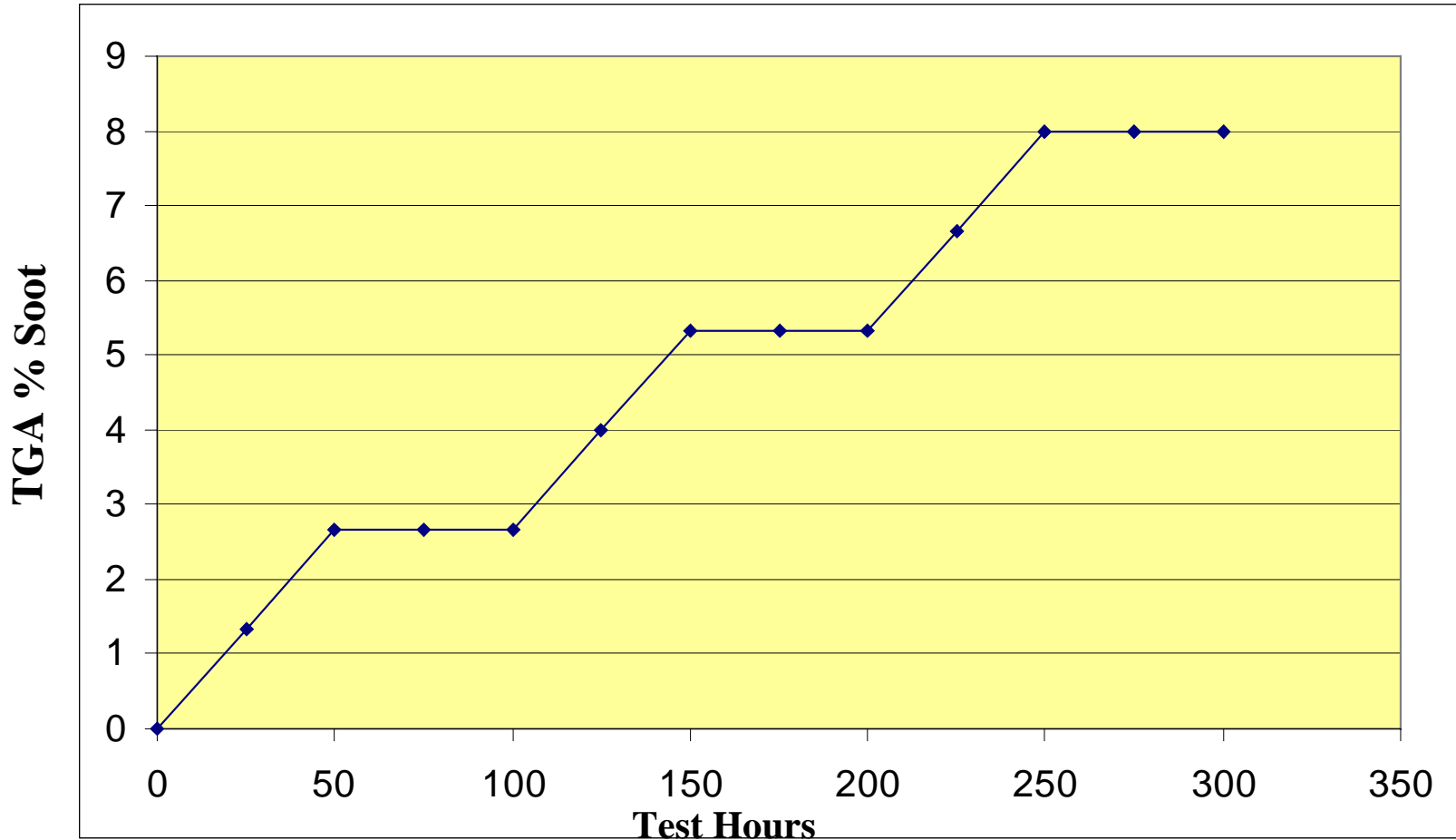
M11 EGR Test Status (Continued)

- **Beaded Filters must be used for future reference runs**
- **Soot Targets:**
 - ✓ **8.5 +/- 0.5 and 4.6 average soot (reference)**
 - ✓ **8.0 min, and 4.6 average soot (non-reference)**
- **Established “To Do List” to meet HDEOCP time requirements**
- **Rating Workshop planned**
- **O&H Panel meeting planned to address lab variability**

Soot vs. Wear in M11 Tests

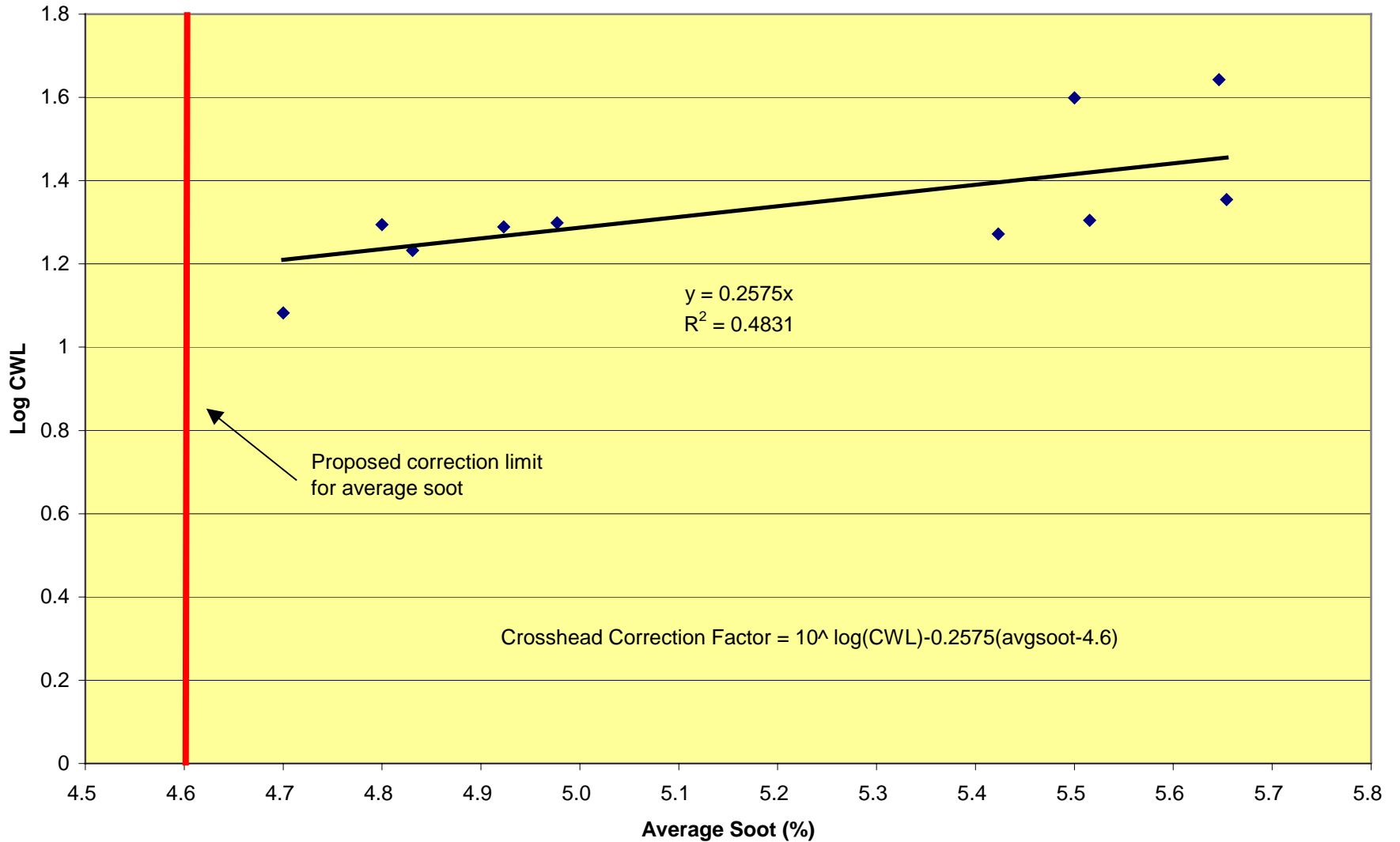


“Ideal” Soot generation Curve



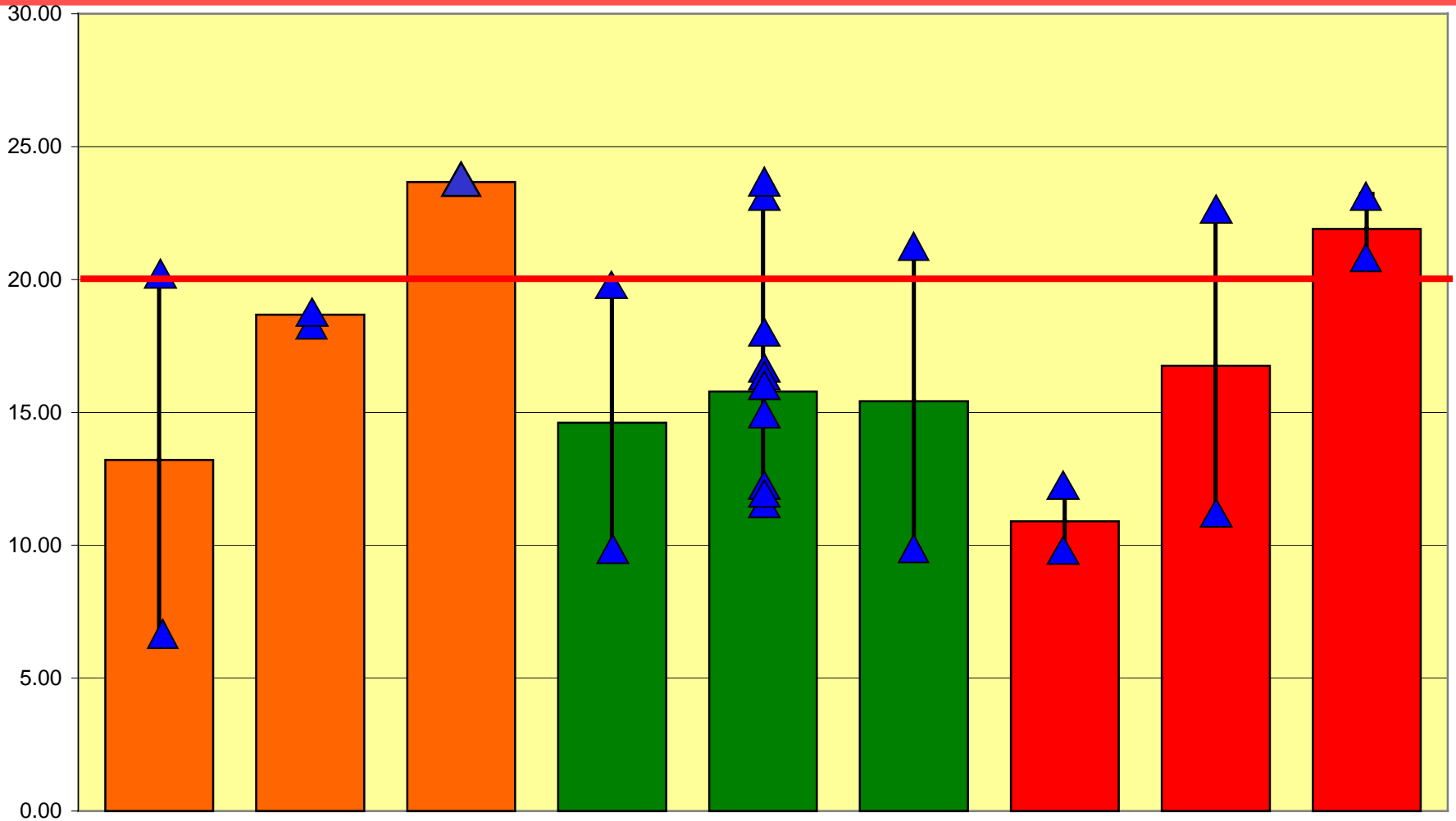
Average Soot = 4.6

Crosshead Weight Loss vs Average Soot: Oil E



M11 PC-9 Matrix: Crosshead Weight Loss

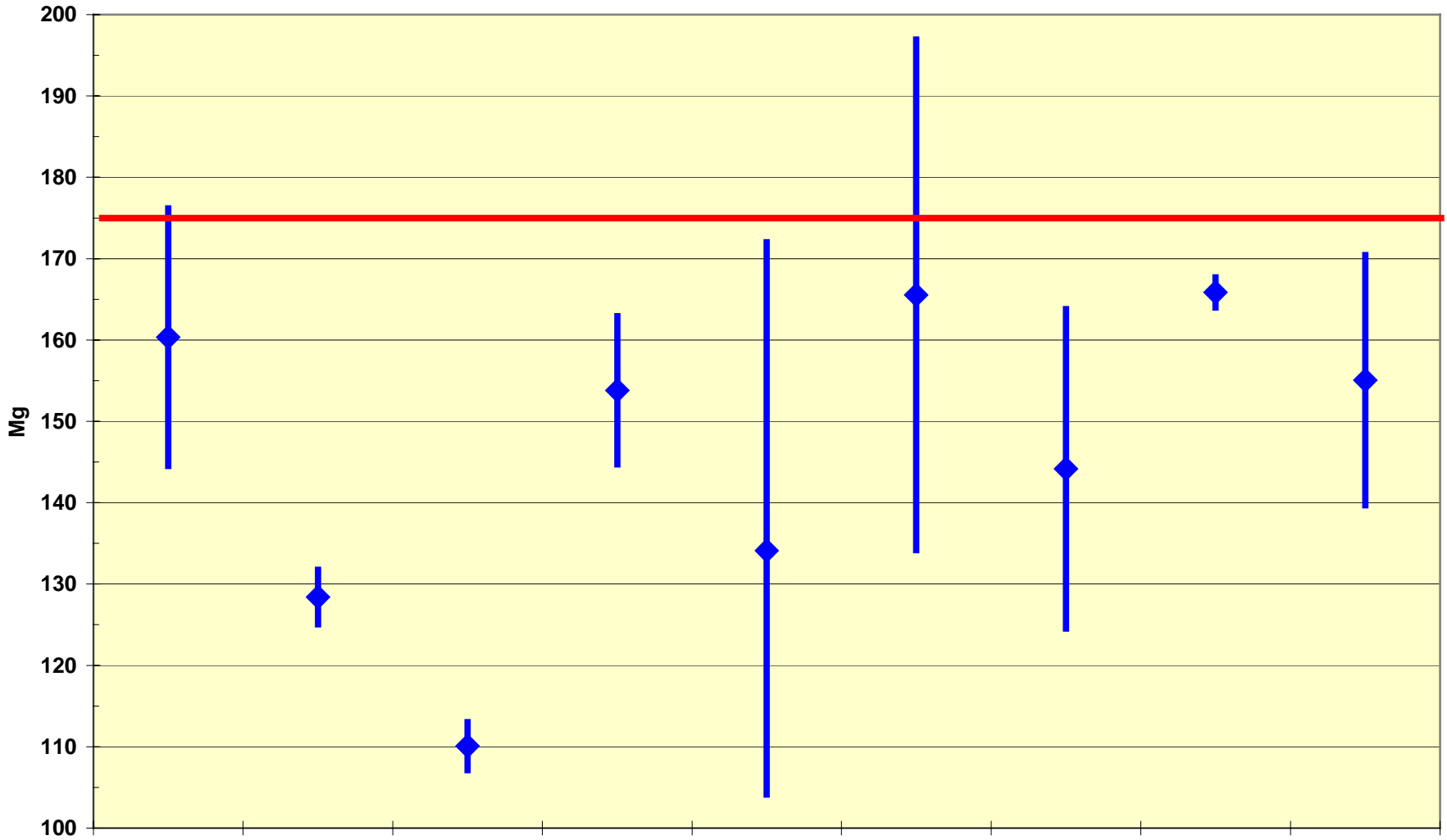
Sorted Rocker Levers, Corrected to 4.6



Oil:	A	B	C	D	E	F	G	H	J
Technology	X	X	X	Y	Y	Y	Z	Z	Z
Base Stock:	1	2	3	1	2	3	1	2	3

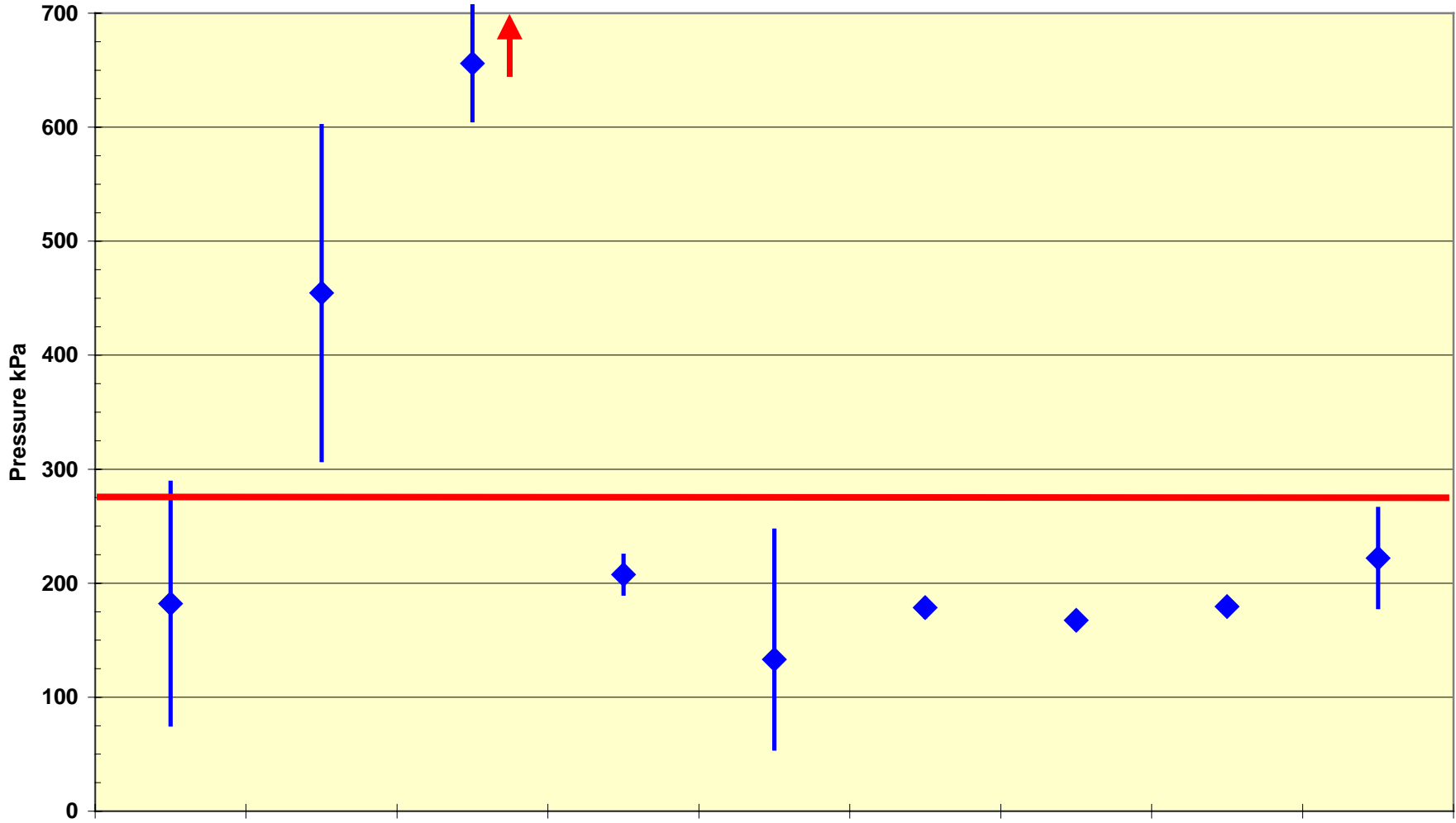
DAVID M Stenouwer, Cummins Inc.

M11 PC-9 Matrix Top Ring Weight Loss

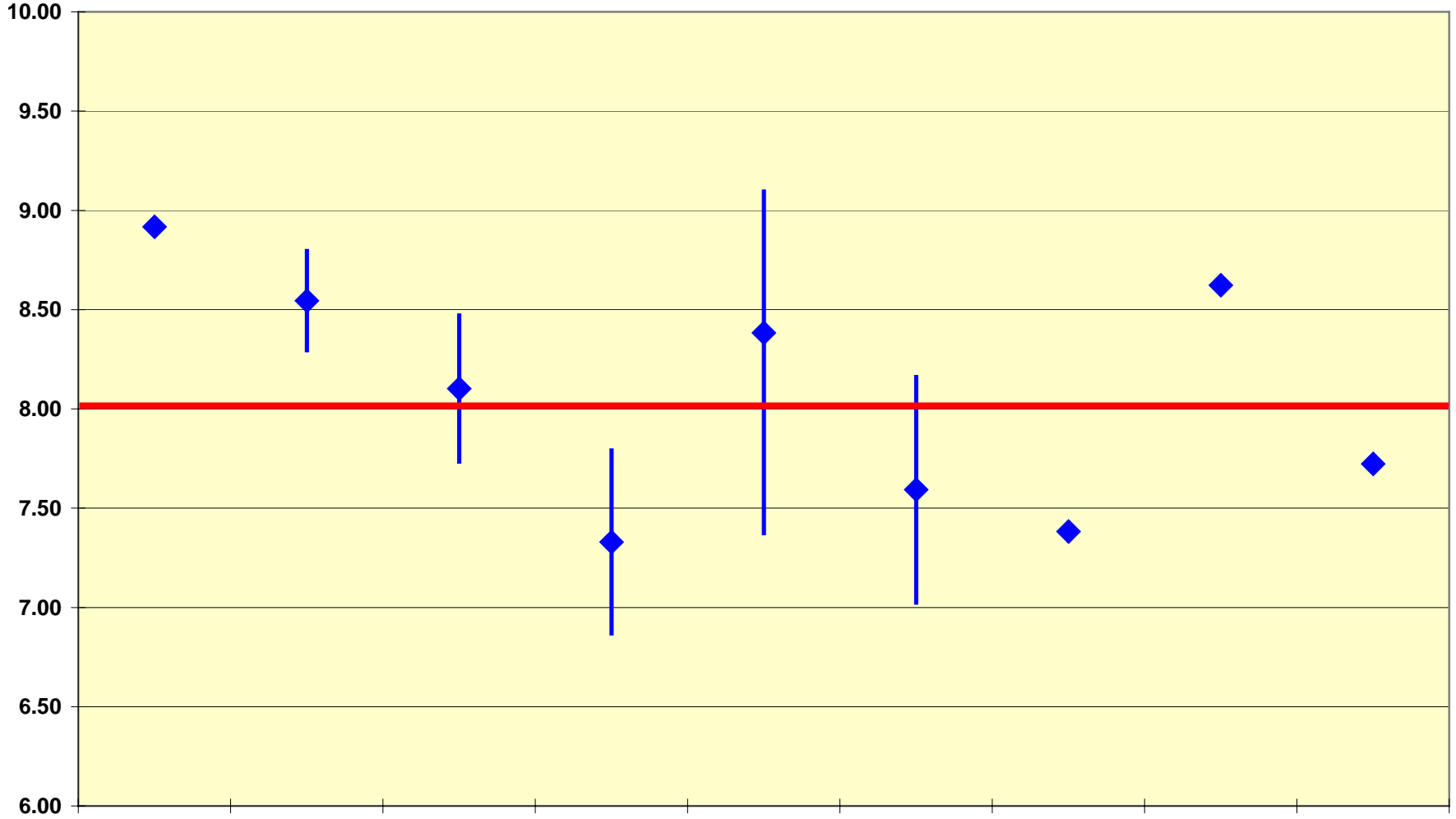


Oil:	A	B	C	D	E	F	G	H	J
Technology	X	X	X	Y	Y	Y	Z	Z	Z
Base Stock:	1	2	3	1	2	3	1	2	3

M11 PC-9 Matrix Oil Filter Delta P @ 250 Hr.

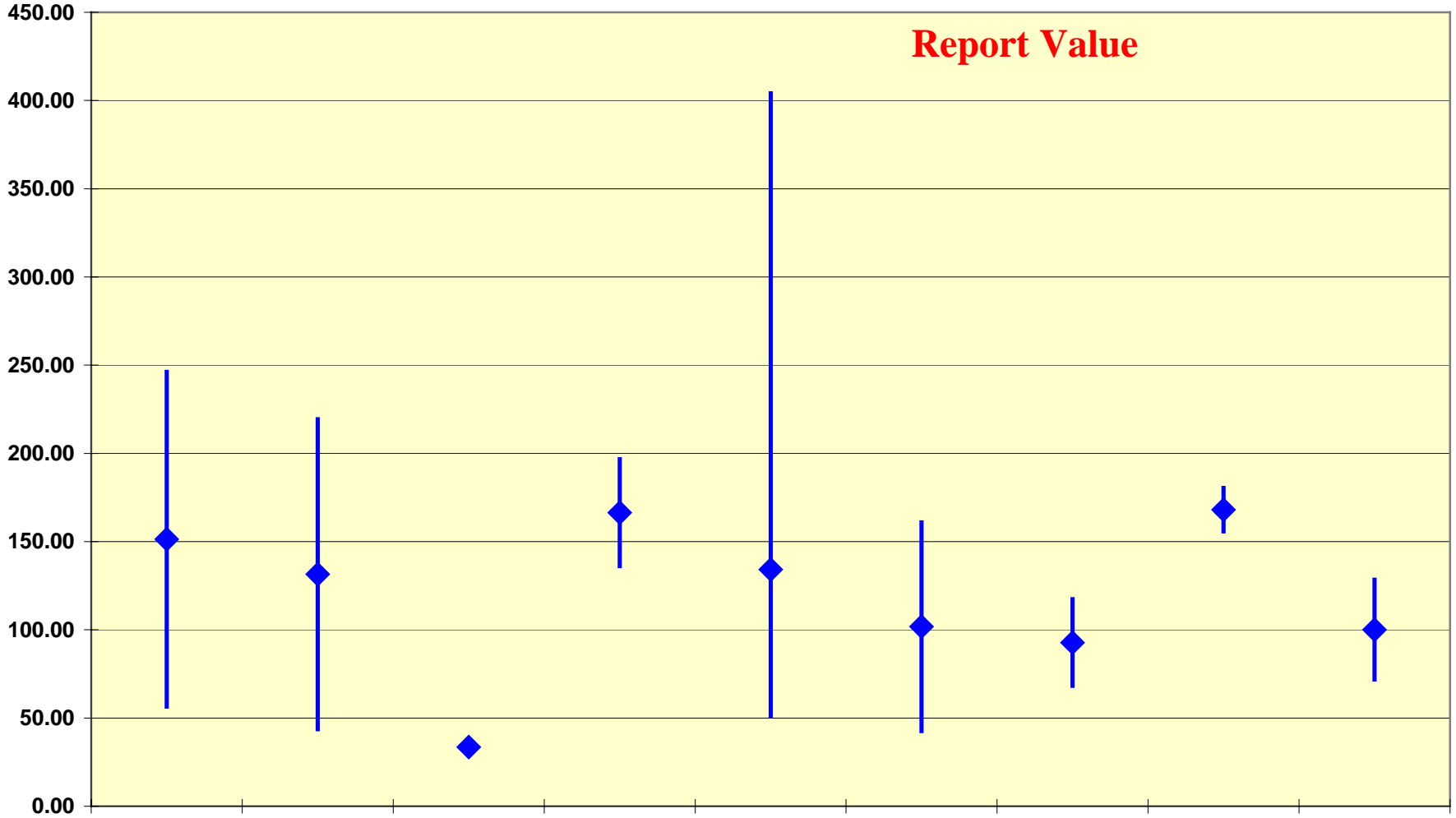


M11 PC-9 Matrix Average Sludge Rating



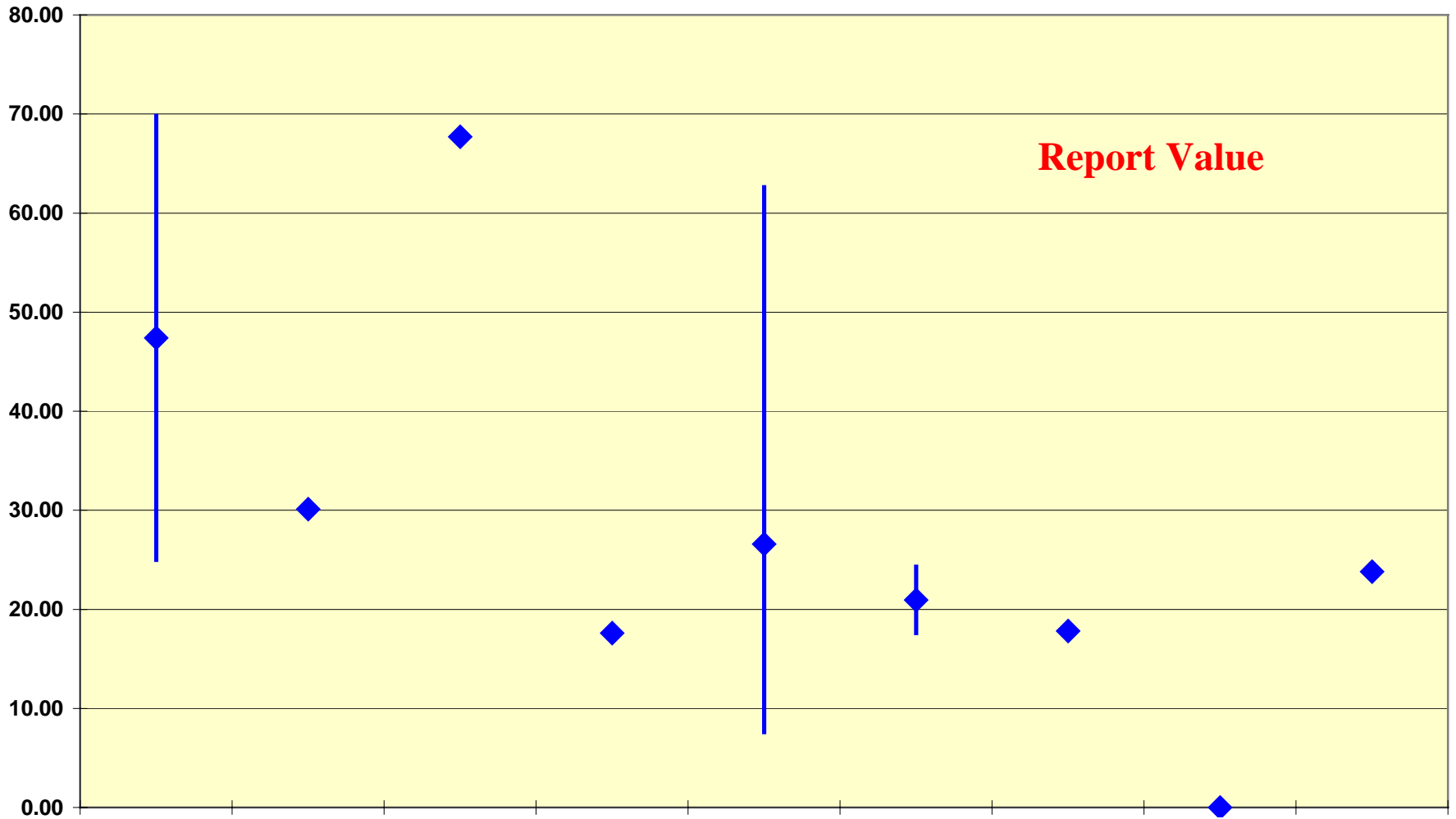
Oil:	A	B	C	D	E	F	G	H	J
Technology	X	X	X	Y	Y	Y	Z	Z	Z
Base Stock:	1	2	3	1	2	3	1	2	3

M11 PC-9 Matrix Injector Adjusting Screw Wt Loss



Oil:	A	B	C	D	E	F	G	H	J
Technology	X	X	X	Y	Y	Y	Z	Z	Z
Base Stock:	1	2	3	1	2	3	1	2	3

M11 PC-9 Matrix Bearing Wt Loss



Report Value

Oil:
Technology:
Base Stock:

A	B	C	D	E	F	G	H	J
X	X	X	Y	Y	Y	Z	Z	Z
1	2	3	1	2	3	1	2	3

M11EGR “to do” List

1. Complete missing data – **July 13th** (Individual Matrix Labs)
 - a. Labs A B D G
 - b. Ring gaps / Bearing Weight Loss / Liner Wear Step
 - c. Items identified per Dennis M presentation
2. Expedite beaded filter prod. and distr. – **August 15th** (Dave S / Ron B)
3. Update database with all parameters – **July 16th** (Jeff C)
4. Complete statistical work – **July 23rd** (Dennis M)
5. Statisticians meet or conference call – **July 25th** (Jim R/Dennis M/Phil S)
6. Complete work required to resolve lab severity differences already identified. – **Teleconference week of July 16th** (Task Force O&H)
7. Complete template & MAD surveys – **July 31st** (Dennis M)
(ACC Member Companies)
- 8.

Test / Template approval

- a. ACC Approve Template – **August 8th&9th** (ACC)
 - b. Task force approve test - **August 9th** (Task Force)
 - c. HDEOCP approve test& Discuss limits - **August 15th** (HDEOCP)
 - d. TMC monitor test – Upon approval of HDEOCP (TMC)
9. ACC approve registration - **August 16th-22nd** (ACC)
- 10.RSI commences test registration upon ACC approval (RSI)
11. Set limits HDEOCP **September 5th** – prepare ballot. (HDEOCP)

Preliminary Analysis of Cummins M11EGR Precision/BOI Matrix

Report to: M11EGR Task Force
July 9, 2001
Columbus, IN

HDEOCP
July 11, 2001
Chicago, IL

M11EGR

Executive Summary

- Top Ring Weight Loss
 - No lab, technology, or base stock effects
 - Crosshead Wear
 - Significant lab differences
 - No Technology or base stock effect
 - Soot and blowby rate effects
 - Average Engine Sludge
 - Significant lab differences
 - Technology/base stock interaction
 - Oil Filter Delta Pressure
 - No lab differences
 - Technology effect (when extreme values are included)
 - Oil Consumption
 - Significant lab differences
 - No technology or base stock effects
 - Adjusting Screw Weight Loss
 - Significant lab differences
 - No technology or base stock effect
-
- Operational differences among labs
 - Intake and Exhaust CO2
 - Number of Shutdowns
 - Torque
 - Blowby
 - Parameters are uncorrelated

Experimental design

Lab A		Lab B	Lab D	Lab G	
S1	S2	S1	S1	S1	S2
B	A	B	C ^a	A	C ^b
E	E	D	D	E	E
E	E	E	E	E	E
F	J	J	H	F	G
G				H	

^a Replaced oil filter due to low oil gallery pressure

^b Terminated at 228 hrs. due to low oil gallery pressure

		Base stock		
		1	2	3
Technology	X	A	B	C
	Y	D	E	F
	Z	G	H	J
		Group II	Group II	Group I

Outliers/missing data

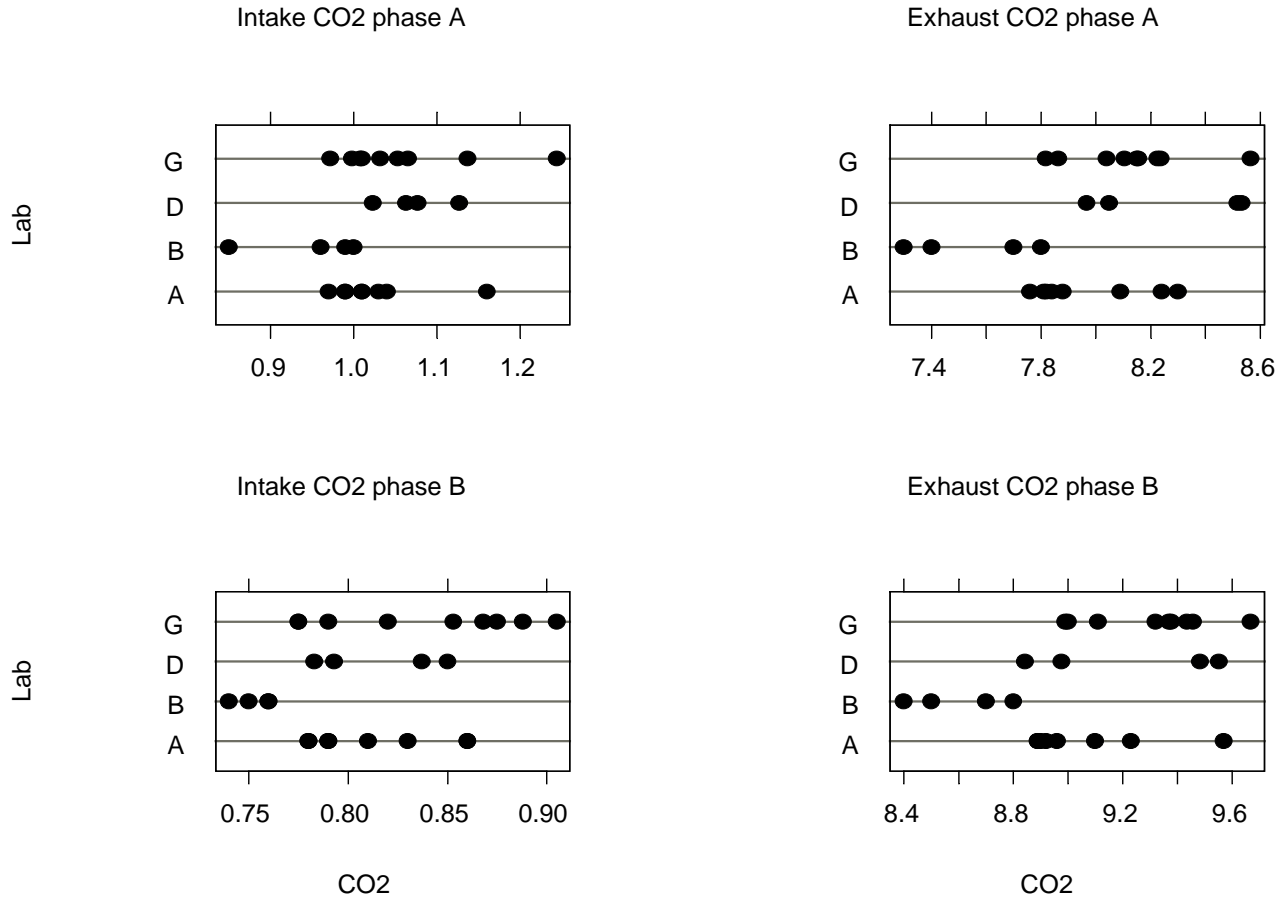
- Possible Outliers

- CMIR 38958 OC = 0 XHDW = 7.7
- CMIR 38931 TBN @ NEW = 7.7
- CMIR 38964 TBN @ NEW = 2.1
- CMIR 38929 ASWL = 404
- CMIR 38965 ASWL = 33

- Missing data

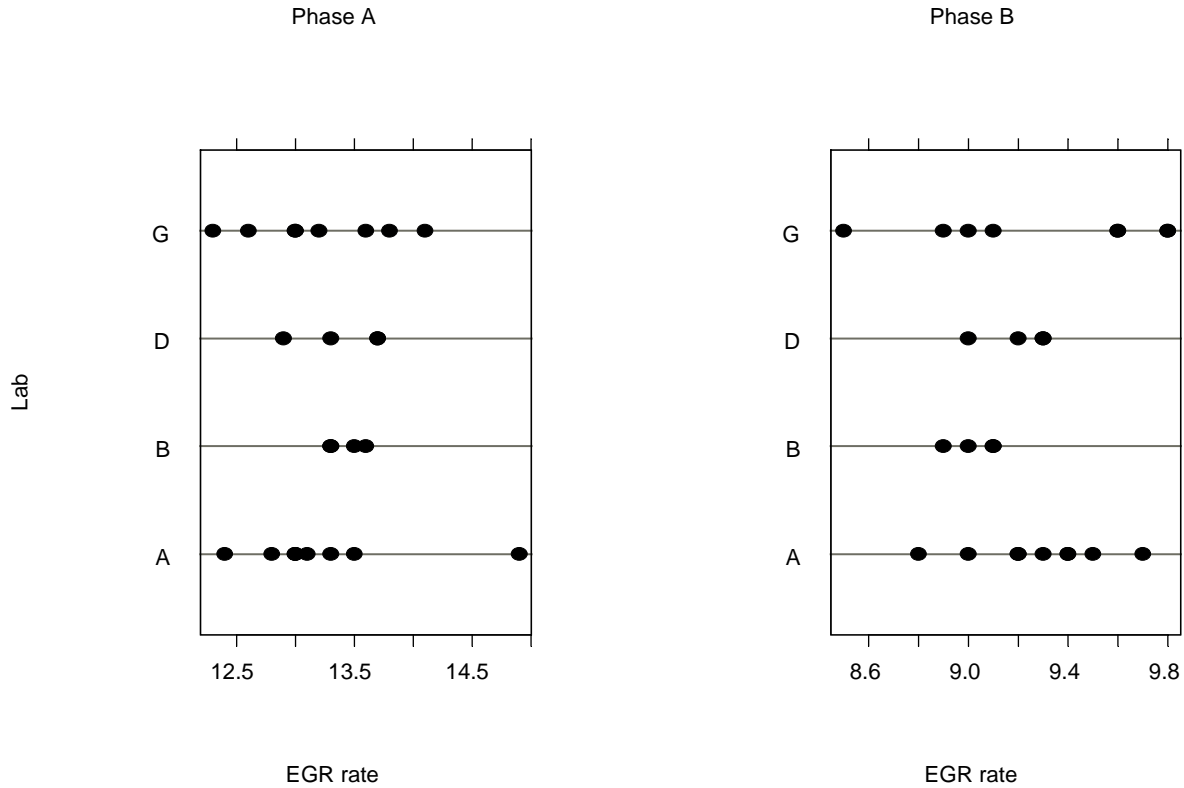
- CMIR 38963 TBN @ EOT,
- CMIR 38930 TBN @ EOT,
- CMIR 38958 Several missing values (due to termination)
- CMIR 38960 TBN @ NEW
- CMIR 38934 CO2
- Liner Wear Step Missing for all but 2 tests

Lab differences - CO₂



- Lab B has significantly lower CO₂ (both intake and exhaust) than Labs A, D, & G

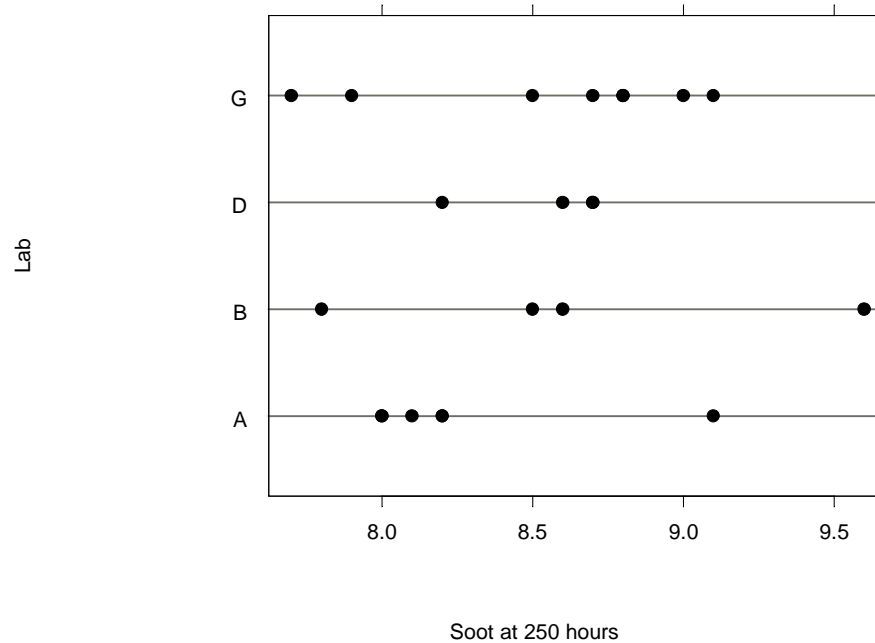
Lab differences - EGR rate



- No significant EGR rate differences by lab

M11EGR

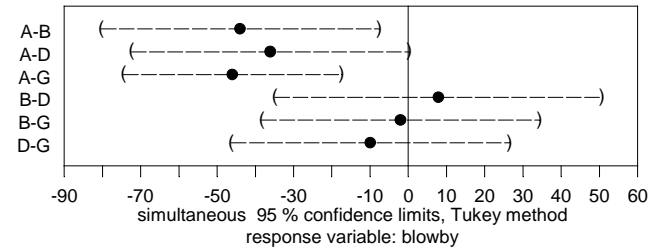
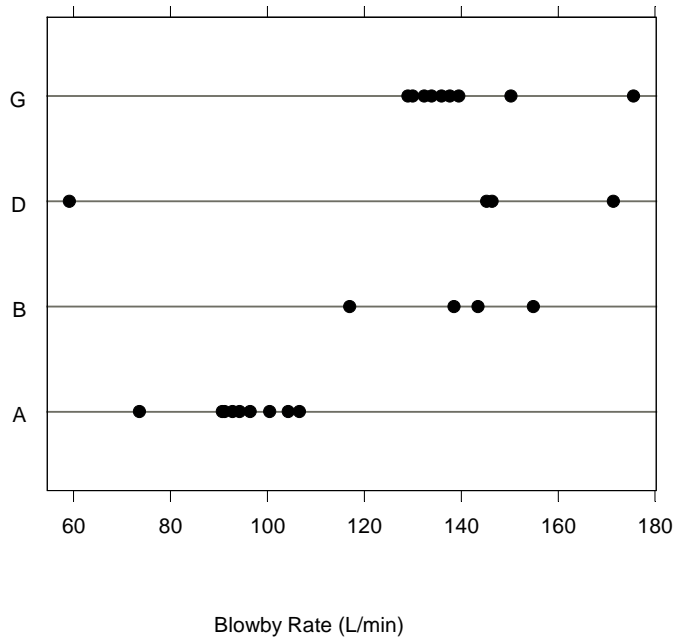
Lab differences - Soot



- No significant soot differences by lab

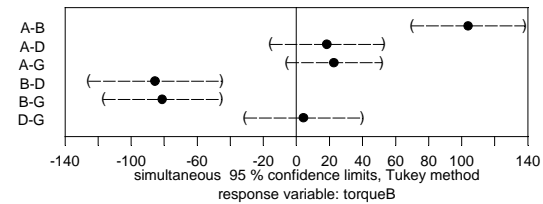
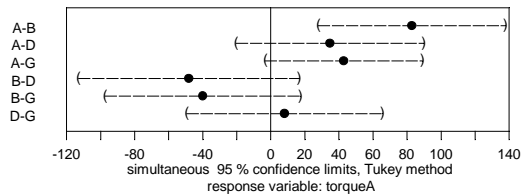
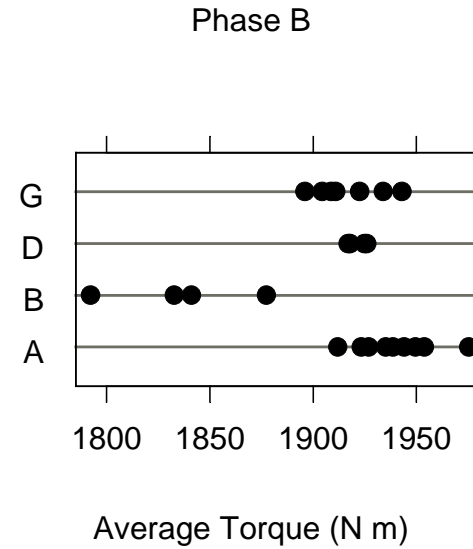
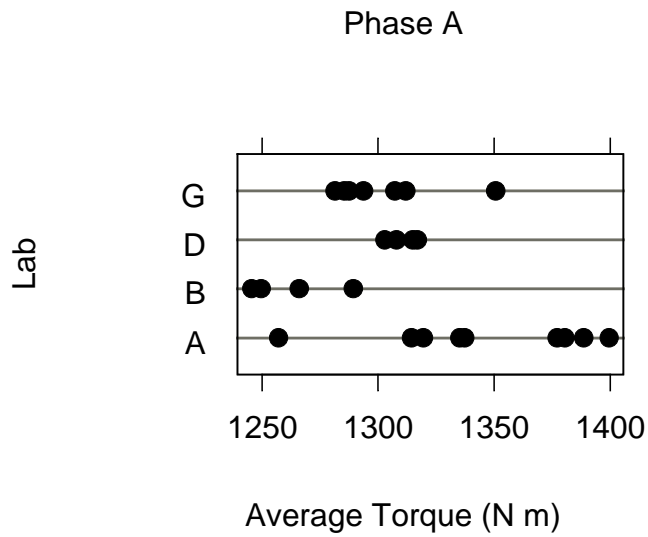
M11EGR

Lab differences - Blowby



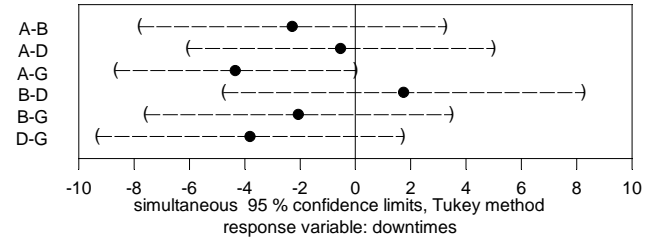
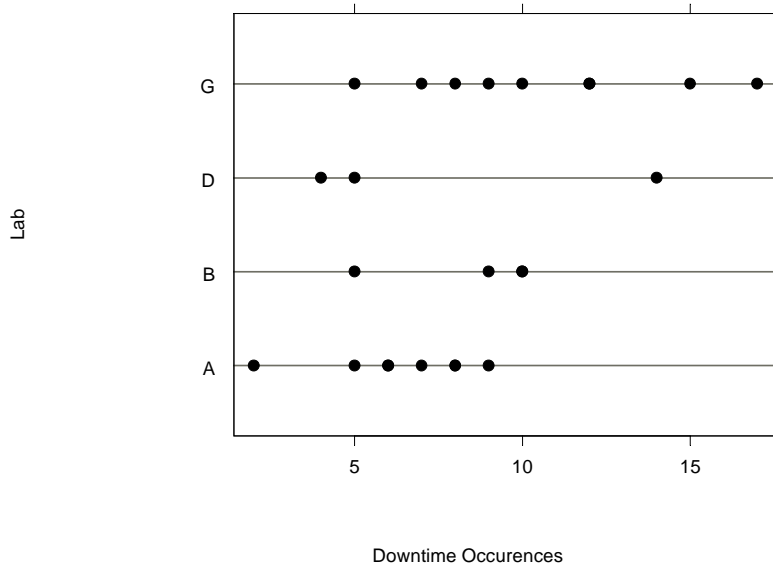
- Lab A has a significantly lower blowby rate than all other labs

Lab differences - Torque



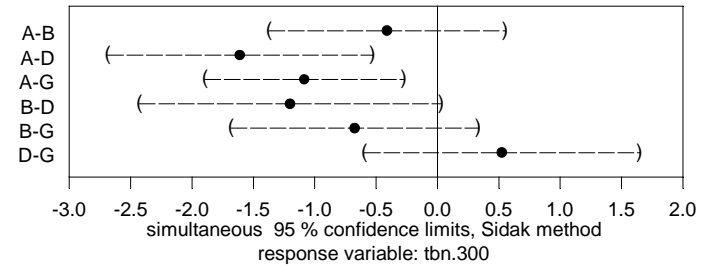
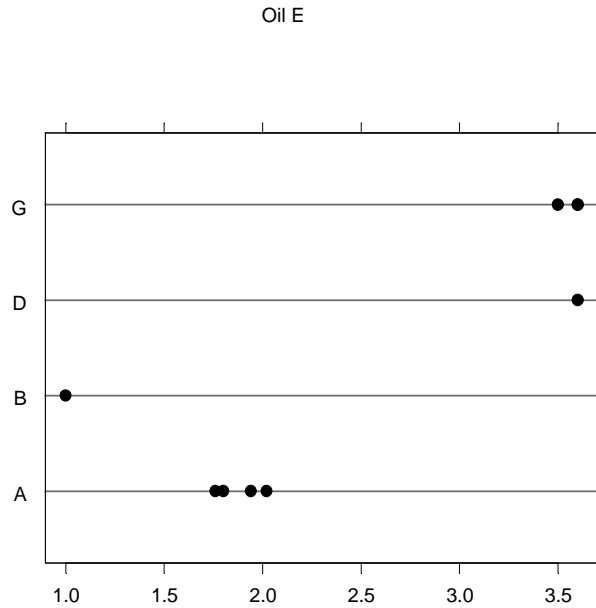
- Lab B runs at significantly lower torque than other labs

Lab differences - Shutdowns



- Lab A has fewer shutdowns than Lab G

Lab differences - EOT TBN

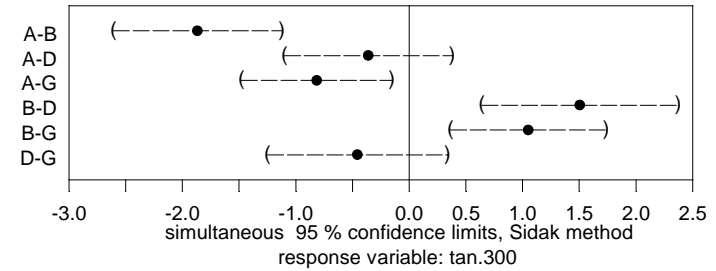
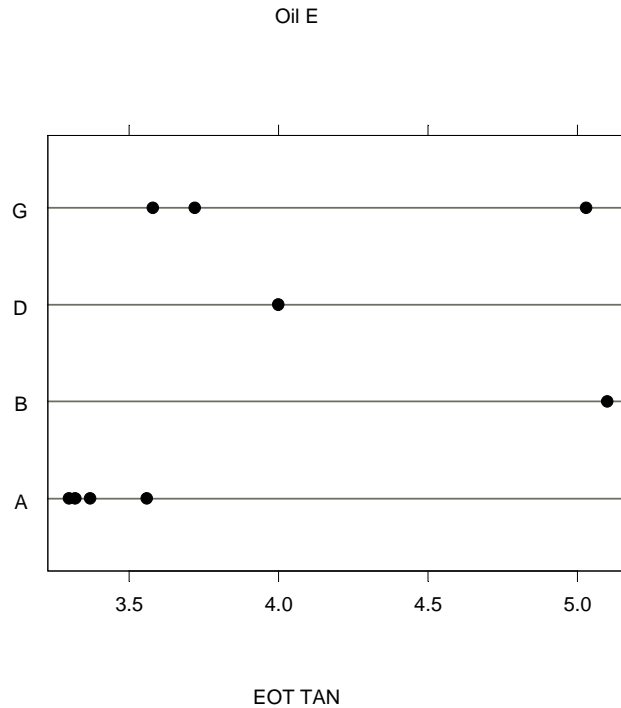


EOT TBN

- Lab A has lower EOT TBN than labs D and G
- Lab B appears not to belong to either group

M11EGR

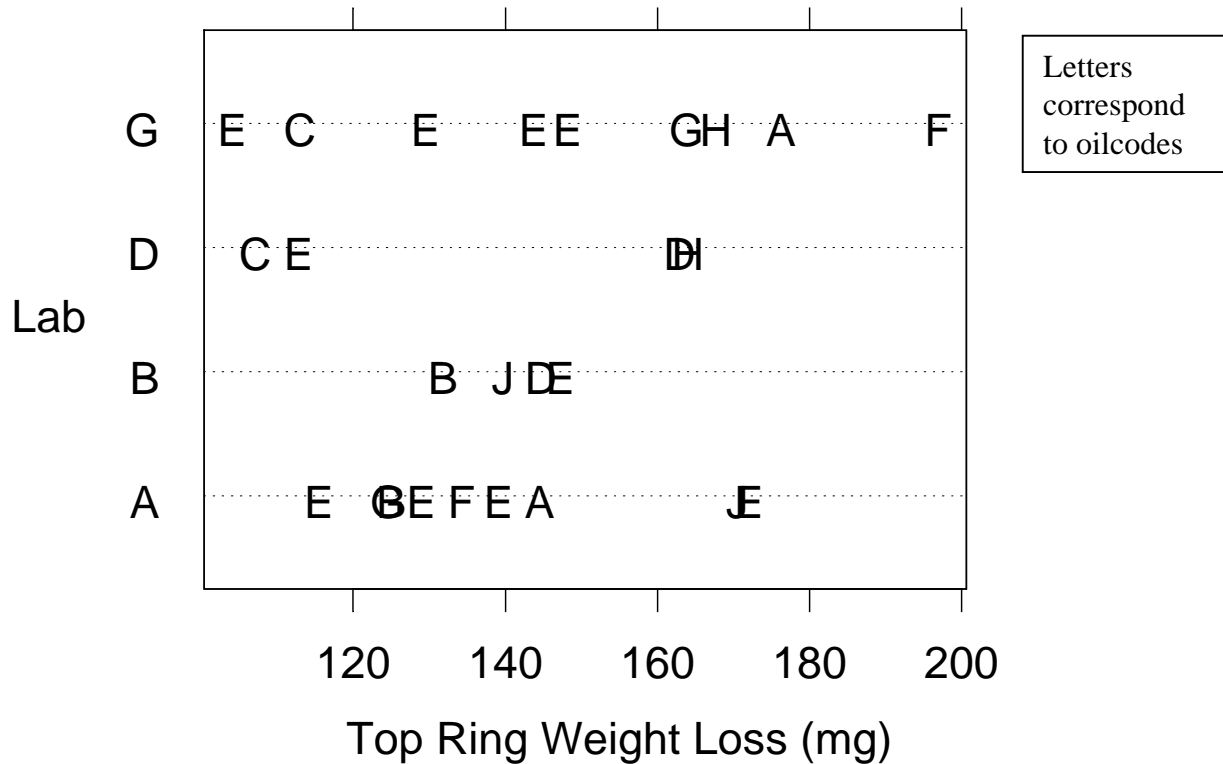
Lab differences - EOT TAN



- Lab B has higher EOT TAN than all other labs
- Lab A has lower EOT TAN than labs B and G

M11EGR

Top Ring Weight Loss

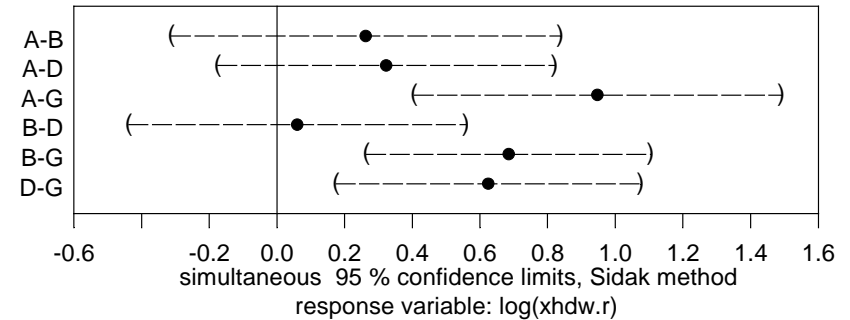
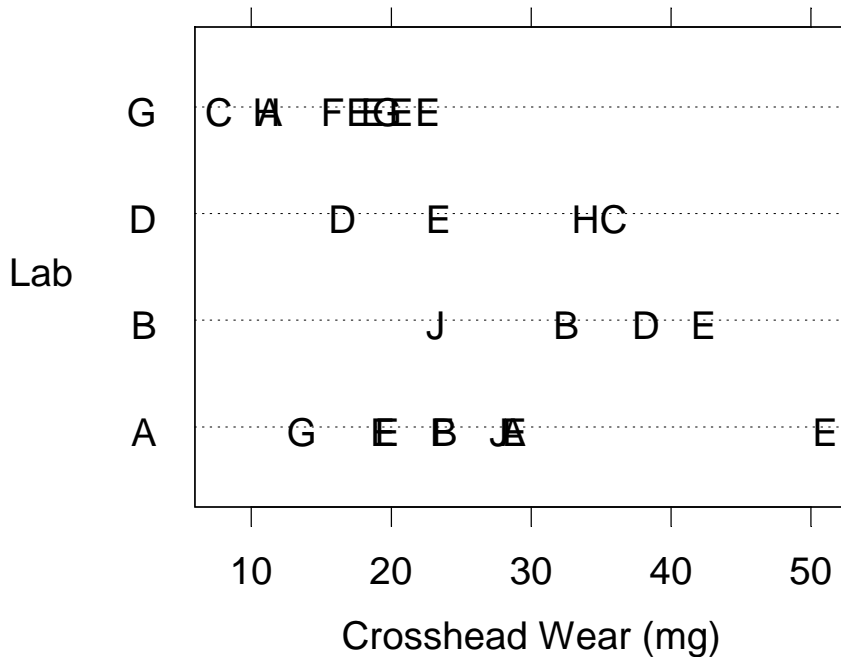


- No significant lab, base stock, or technology effect
- Excluding CMIR 38958 (TRWL=113) makes no difference

Crosshead Wear

- Significant lab effect
- Crosshead wear increases with increasing
 - Average soot (argument for soot correction)
 - Blowby rate
- No technology or base stock effect
- Log transform gives a better fit

XHDW - Lab differences



- Lab G has lower crosshead wear than the other labs
- Therefore Labs are combined into two groups: Lab G and Labs A, B, and D
- CMIR 38958 (XHDW = 7.7) and CMIR 38932 (XHDW = 51) do not effect conclusions

M11EGR

XHDW statistical model

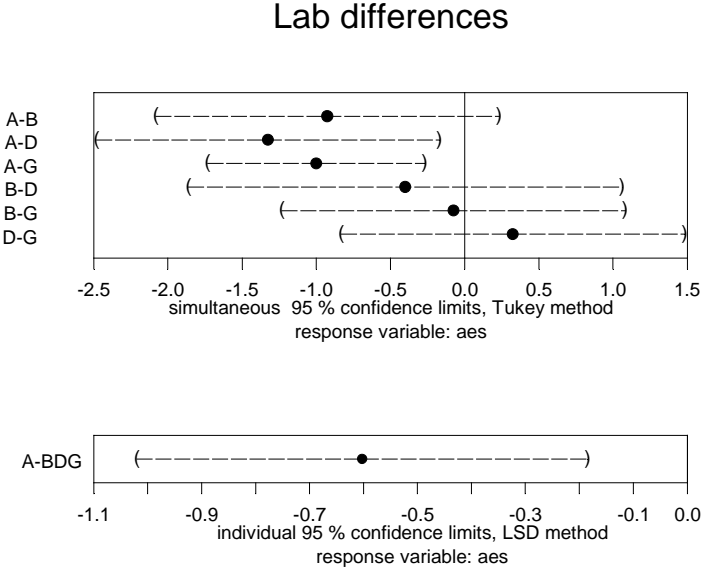
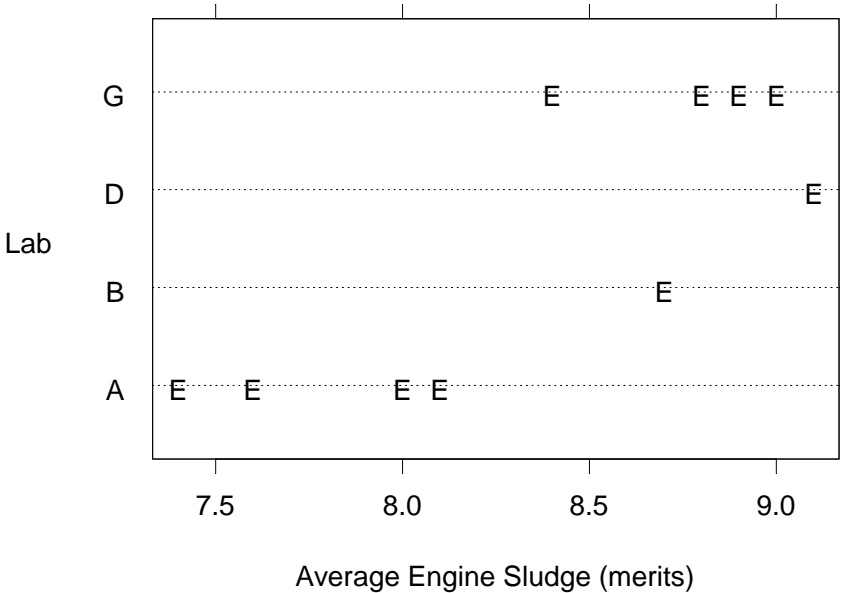
$$XHDW = e^{-0.83 LabG} e^{0.52 avSoot} e^{0.0047 Blowby}$$

- Lab G results in $e^{-0.83} = 0.43$ times as much wear as the other labs
- An increase in average soot by one TGA unit gives $e^{0.52} = 1.7$ times as much wear
- An increase in blowby rate of 10 L/min gives $e^{0.047} = 1.1$ times as much wear

Average Engine Sludge

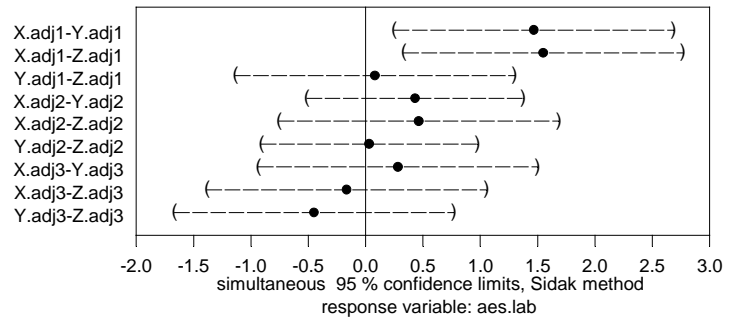
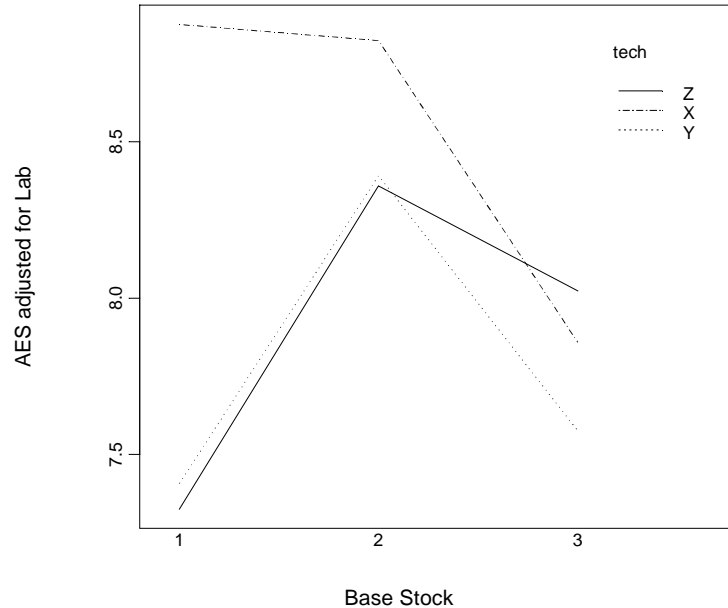
- Significant lab effect
- Two lab populations: A and BDG
- Technology / base stock interaction
- No transformation necessary
- CMIR 38958 is included (AES = 8.5)

AES - Lab differences



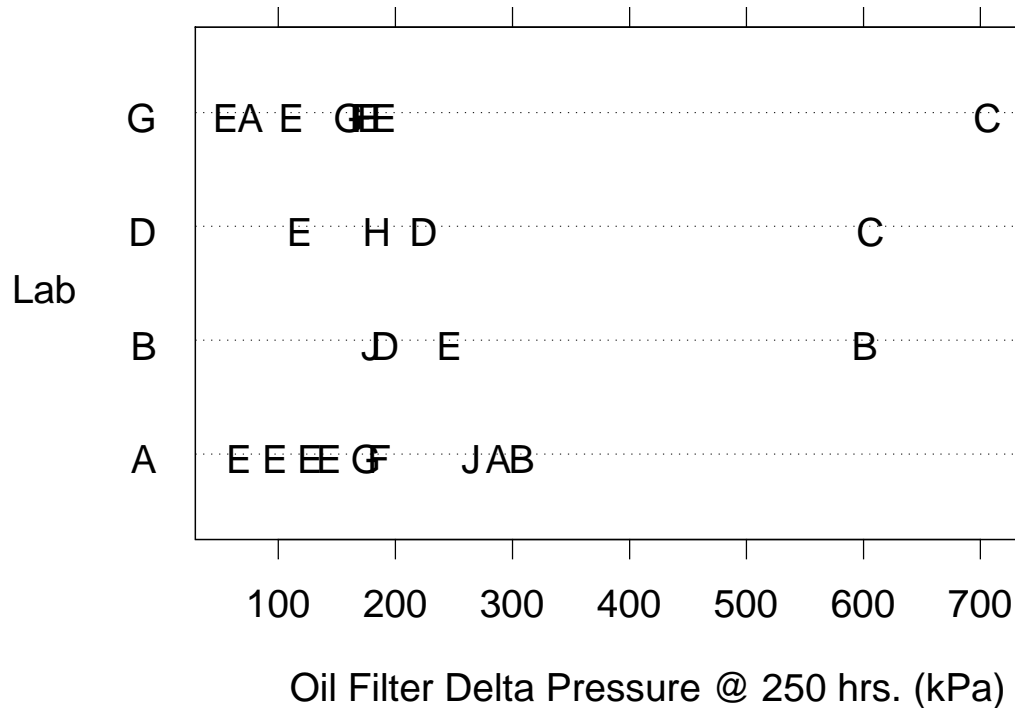
- Two lab populations: A and BDG
- CMIR 38958 is included (AES = 8.5)

AES - Base stock/Technology interaction



- Technology X has better sludge performance than technologies Y and Z, but only in base stock 1
- Base stock 2 is has better sludge performance than base stock 3 for all technologies

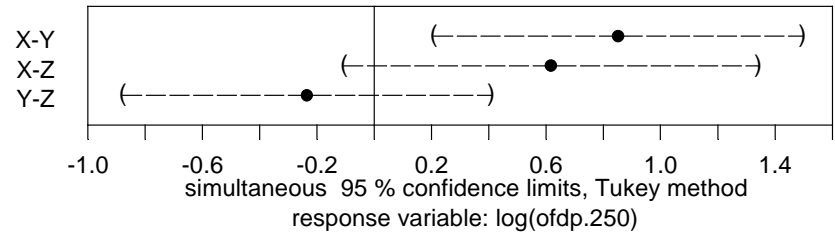
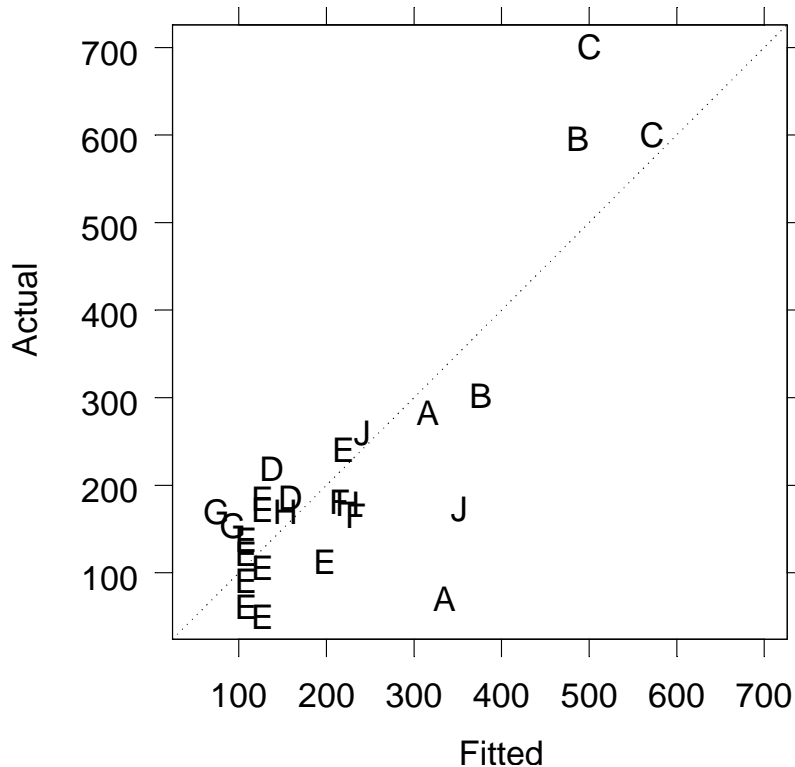
Oil Filter Delta Pressure @ 250 hrs.



- Three extreme values
- This includes CMIR 38958 OFDP = 706 @ 228 hrs.

M11EGR

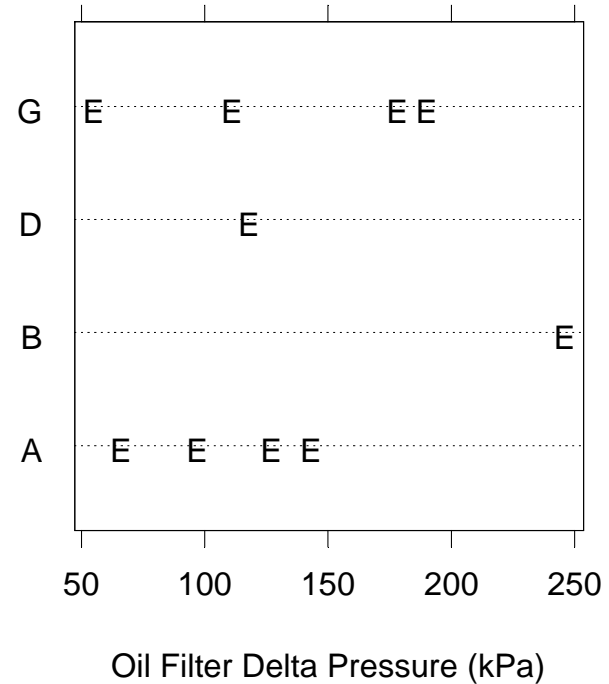
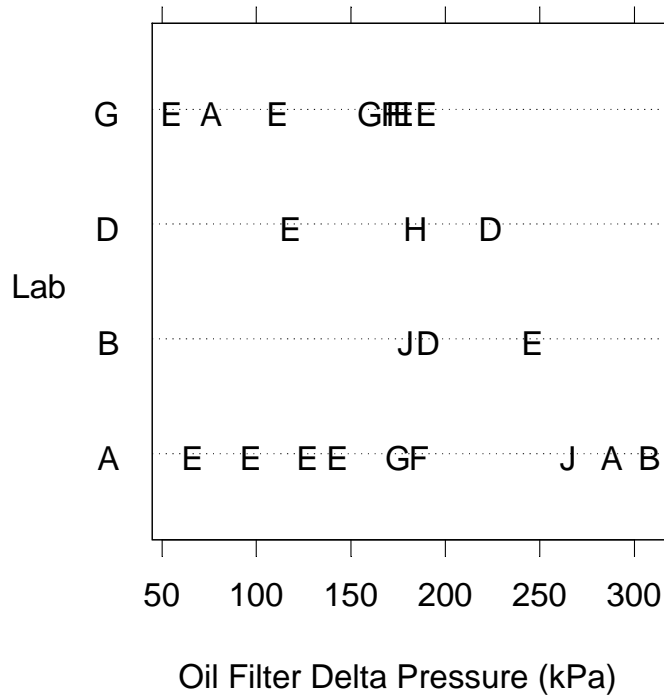
OFDP @ 250 hrs. (including extreme values)



- Technology X has higher OFDP than technologies Y and Z
- No lab or base stock effects
- Diagnostic plots show these points are highly influential

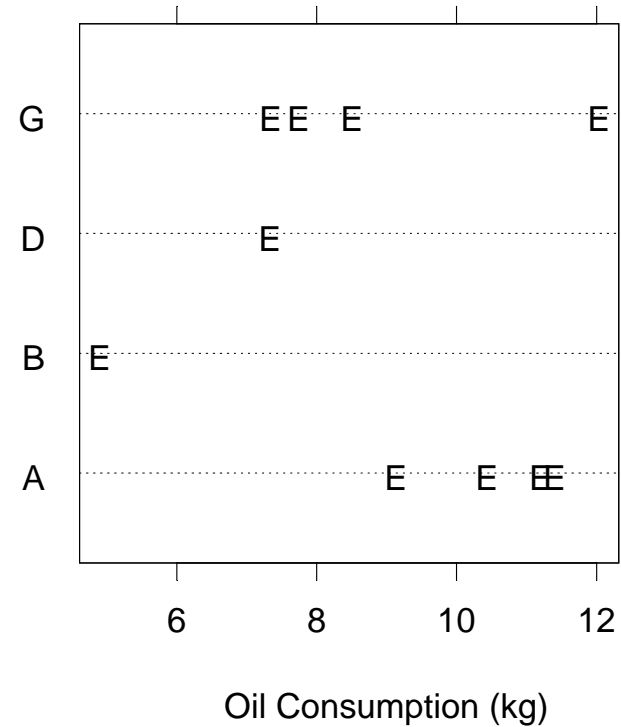
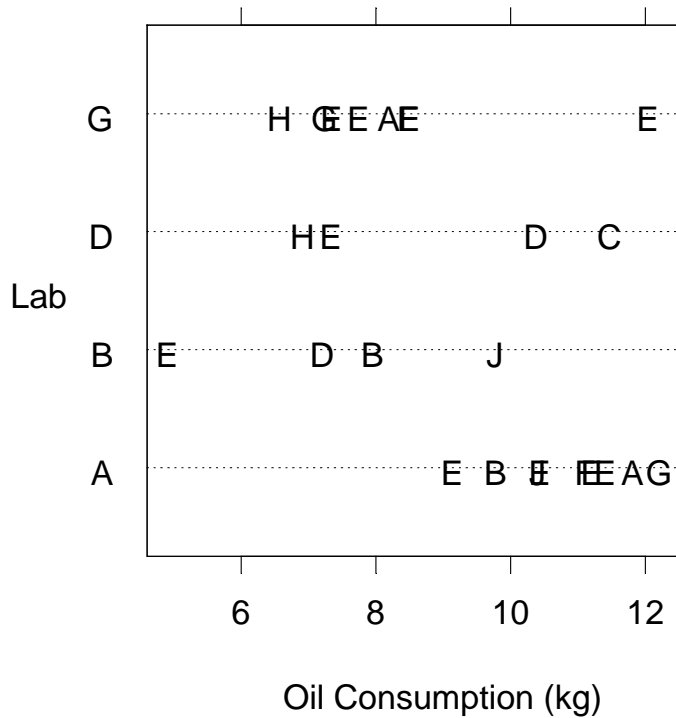
M11EGR

OFPD @ 250 hrs. (excluding extreme values)

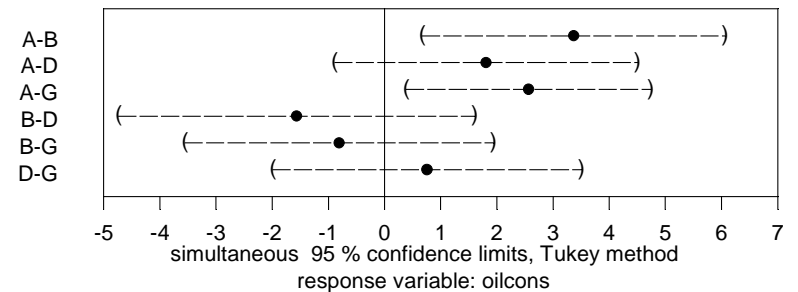


- No significant lab differences
- No significant base stock or technology effects
- However all information on oil C (Technology X in Base stock 3) is missing

Oil Consumption

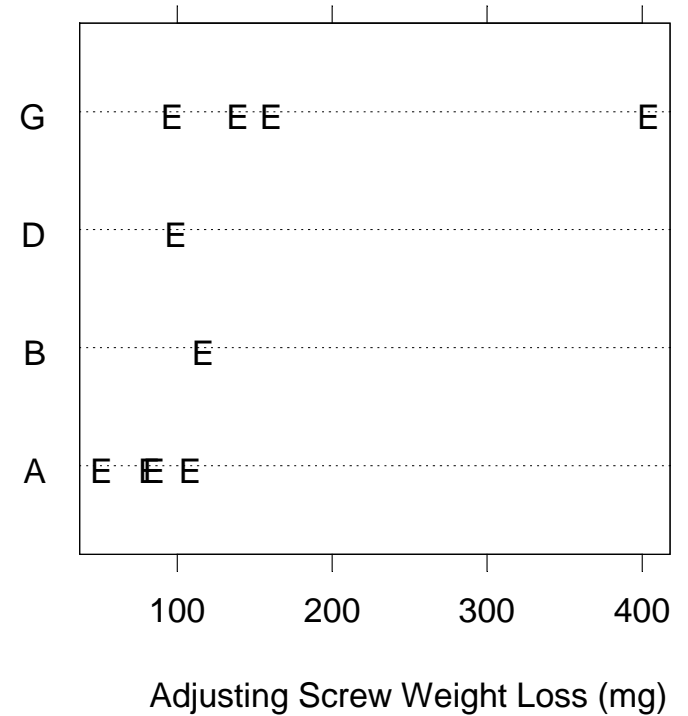
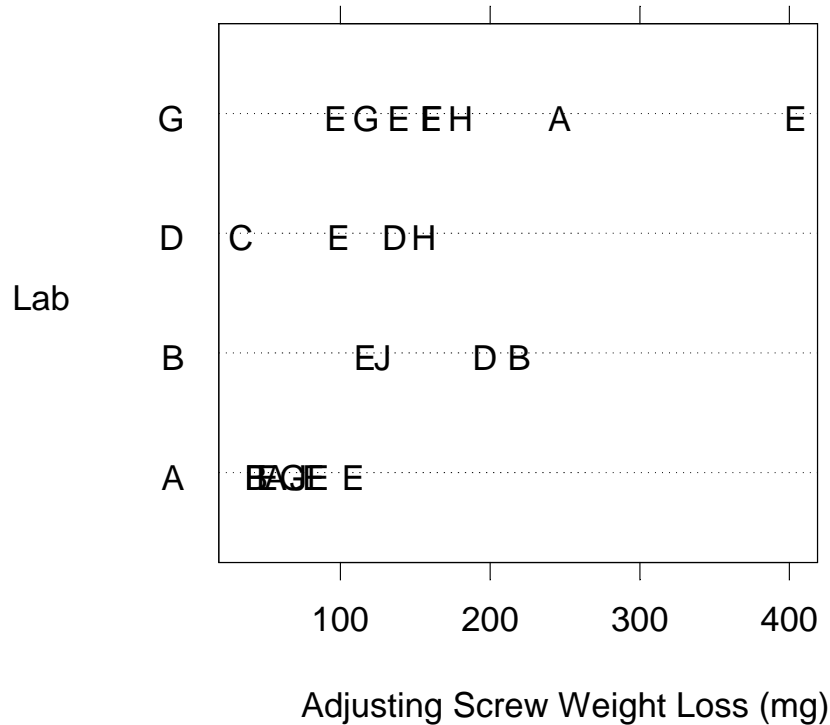


- Lab A has higher oil consumption than the other labs
- No technology or base stock effects

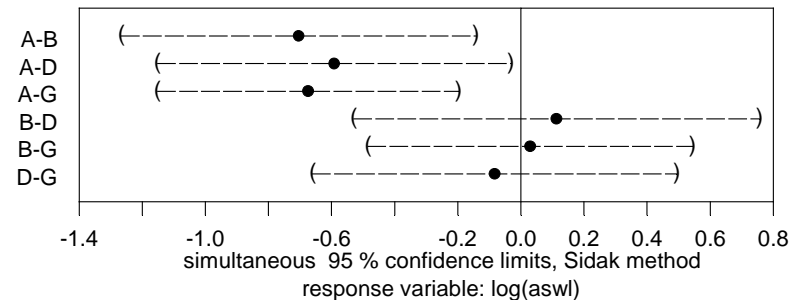


M11EGR

Adjusting Screw Weight Loss



- Lab A has lower ASWL than the other labs
- Technology and Base stock are not significant
- Log transform improves fit
- Excluding CMIR 38958 (ASWL = NA) & CMIR 38929 (ASWL = 404)



M11EGR

Precision estimates

Precision estimates

Parameter	AES	TRWL	XHDW	ASWL	OC	OFDP
Mean	8.40	134	26	134	9.0	133
Stand sd	0.36	24	12	97	1.6	36
Total sd	0.60	24	12	100	2.3	58
Repeatability	1.01	67	34	272	4.5	101
Reproducibility	1.68	67	34	280	6.4	162
Coef Var (%)	7.14	18	46	75	26	44

- All on oil E because only oil E had repeat runs in a lab

Parameter Correlations

TRWL	0.14	-0.10	-0.11	0.37	-0.31
-0.09	XHDW	-0.05	0.20	-0.16	-0.33
-0.10	-0.22	AES	-0.01	-0.01	-0.08
-0.22	0.34	-0.08	OFDP	-0.42	0.07
0.21	-0.13	0.31	-0.21	ASWL	-0.24
-0.35	-0.13	-0.30	0.03	-0.21	OC

- The upper triangle shows the partial (adjusted) correlations
- The lower triangle is the raw (unadjusted) correlations
- No significant correlations among the parameters (using 0.85 as the significance criterion)

PC-9 Matrix Design Task Force

PC-9 Precision/BOI Matrix Update

Presented to
Heavy Duty Engine Oil Classification Panel
Chicago, IL
July 11, 2001

Caterpillar 1R Matrix Testing

- **PC-9 MDTF 1R Proposal to HDEOCP**
 - Five Labs – Nine Test Stands – 18 Tests (Two Tests/Stand)
 - Three Test Oils:
 - 9 Runs PC-9M, 7 Runs PC-9A, and 2 Runs PC-9D
 - Each Stand will see PC-9A or PC-9M (7 of 9 stands see both)
 - Oil PC-9D should be run as the Second Test in One of the Two Original 1Q Stands at each Independent Lab as a Substitute for Oil PC-9A in that Stand

Caterpillar 1R Matrix Test Oil Selection

Code	Technology	Base Oil	Featured Oil for the Test Listed
PC-9A	X	1	T-10
PC-9D	Y	1	(M11/EGR = PC-9E)
PC-9M	W	4	1R

Caterpillar 1R Matrix Testing

PC-9 Matrix Test Plan								
Caterpillar 1R								
Lab 1			Lab 2	Lab 3			Lab 4	Lab 5
Stand 1	Stand 2	Stand 8	Stand 3	Stand 4	Stand 5	Stand 9	Stand 6	Stand 7
PC-9A	PC-9M	PC-9A	PC-9M	PC-9A	PC-9M	PC-9A	PC-9M	PC-9A
PC-9M	PC-9D	PC-9M	PC-9A	PC-9M	PC-9D	PC-9M	PC-9A	PC-9M

Caterpillar 1R Matrix Testing

- **1R Matrix Status**

- **Nine 1R Tests in Progress**
First Round Started Mid June
- **Estimated Completion of First Round:**
July 20, 2001
- **Estimated Completion Of Test Plan:**
August 21, 2001
- **Estimated Completion of Data Analysis:**
August 30, 2001



CATERPILLAR 1R TEST RESULTS

JULY 12, 2001

CATERPILLAR 1R TESTS



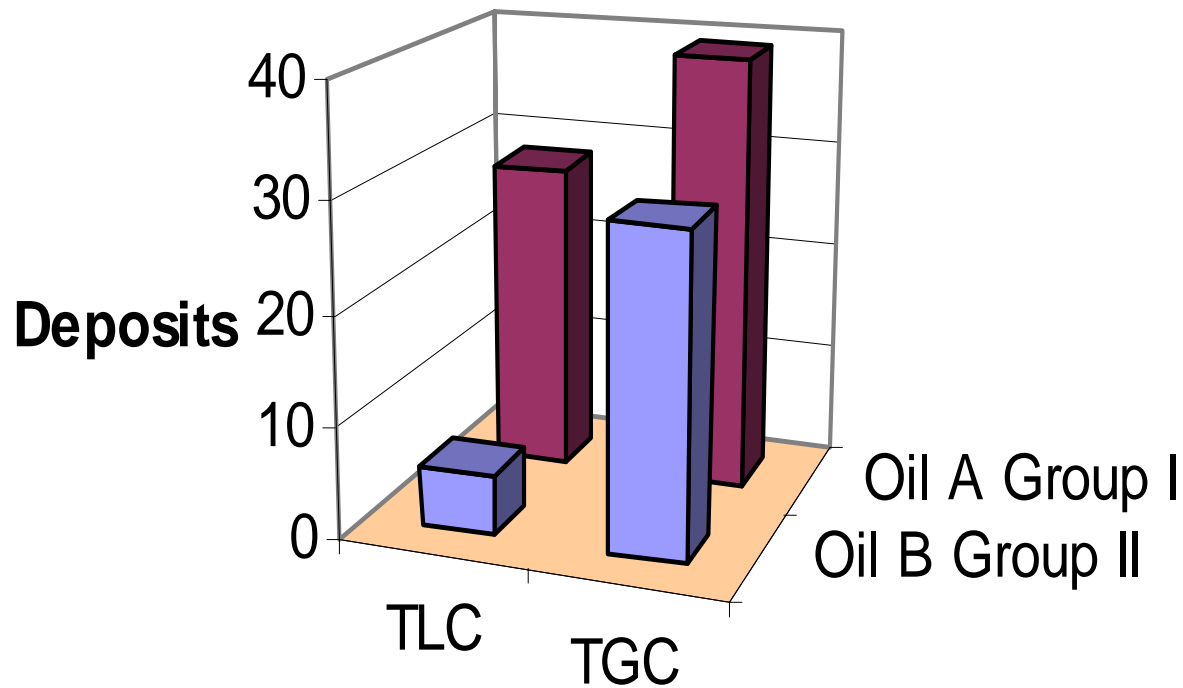
- **Base Oil Comparison**
- **Oxidation Effects**
- **Summary**

CAT 1R TEST RESULTS

Group I vs Group II base oils 15W-40 same Additive System



CAT 1R Deposits vs Base Oil Type

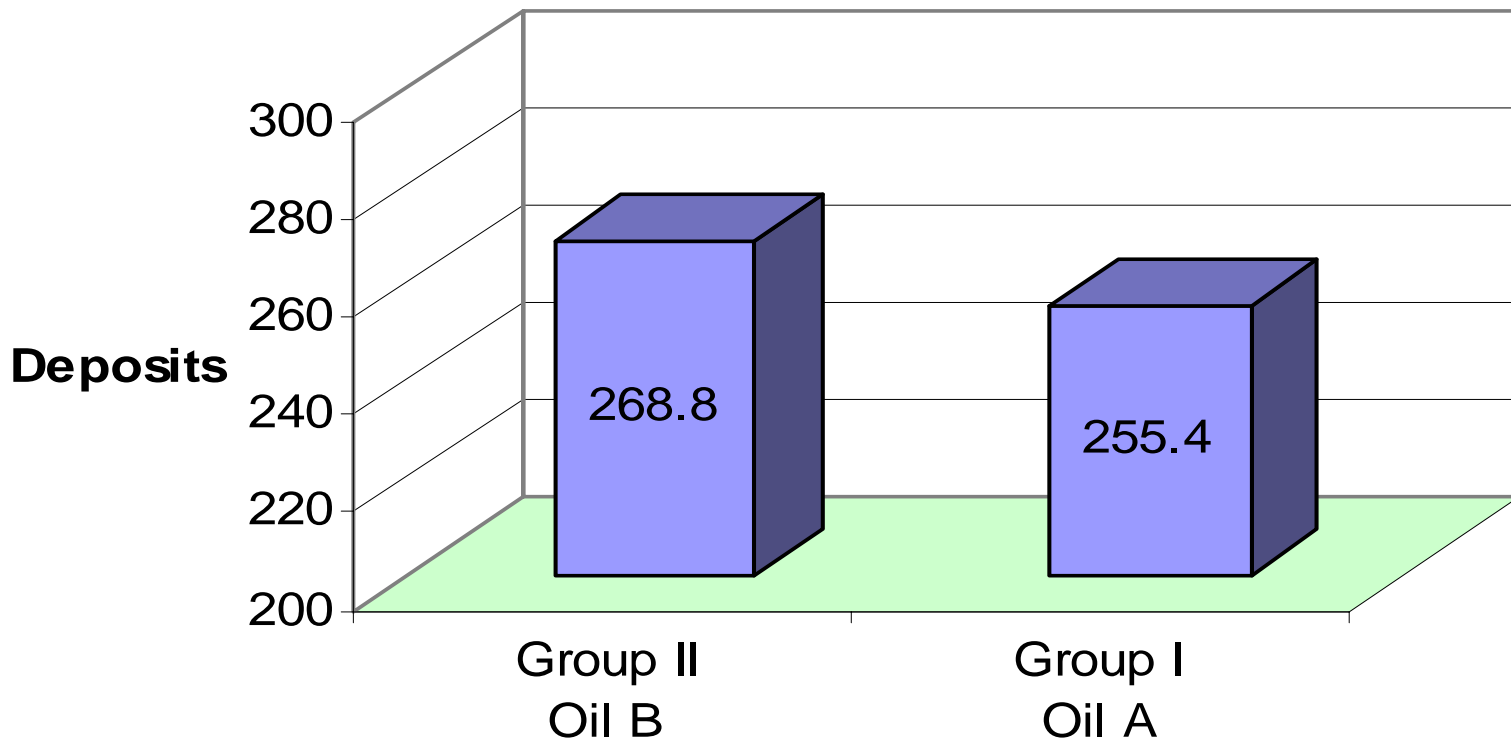


CAT 1R TEST RESULTS

Group I vs Group II base oils 15W-40 same Additive System

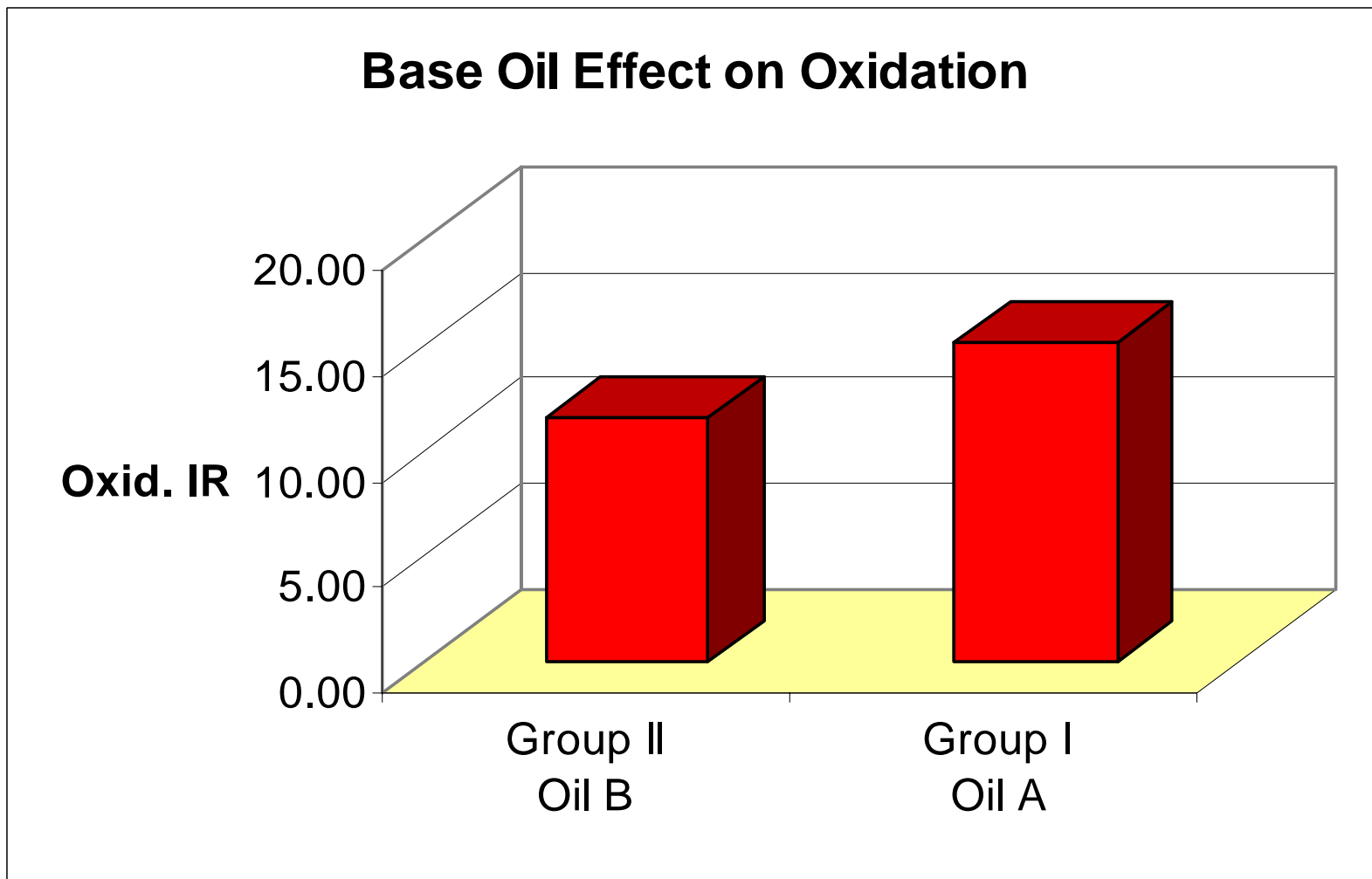


WDP vs Base Oil Type



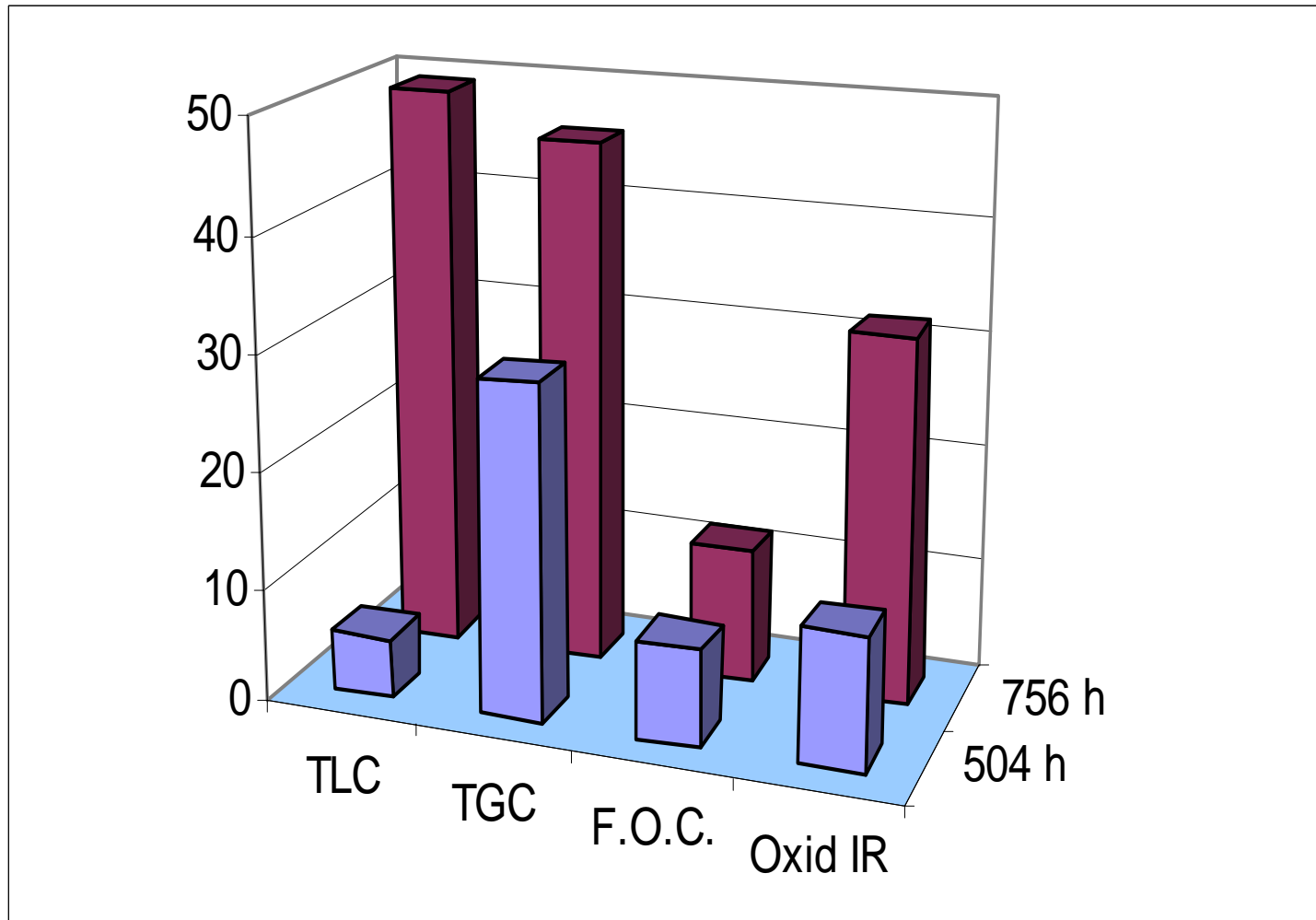
CAT 1R TEST RESULTS

Group I vs Group II base oils 15W-40 same Additive System



CAT 1R TEST RESULTS

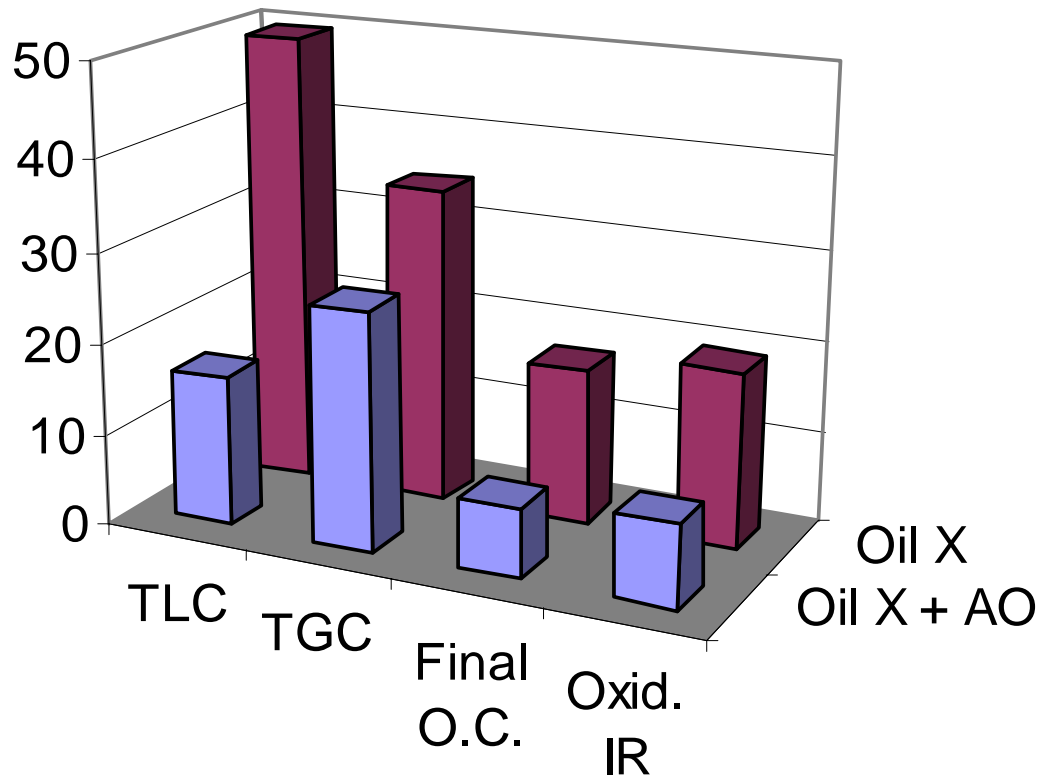
Oil B at Extended Hours



CAT 1R Test Results



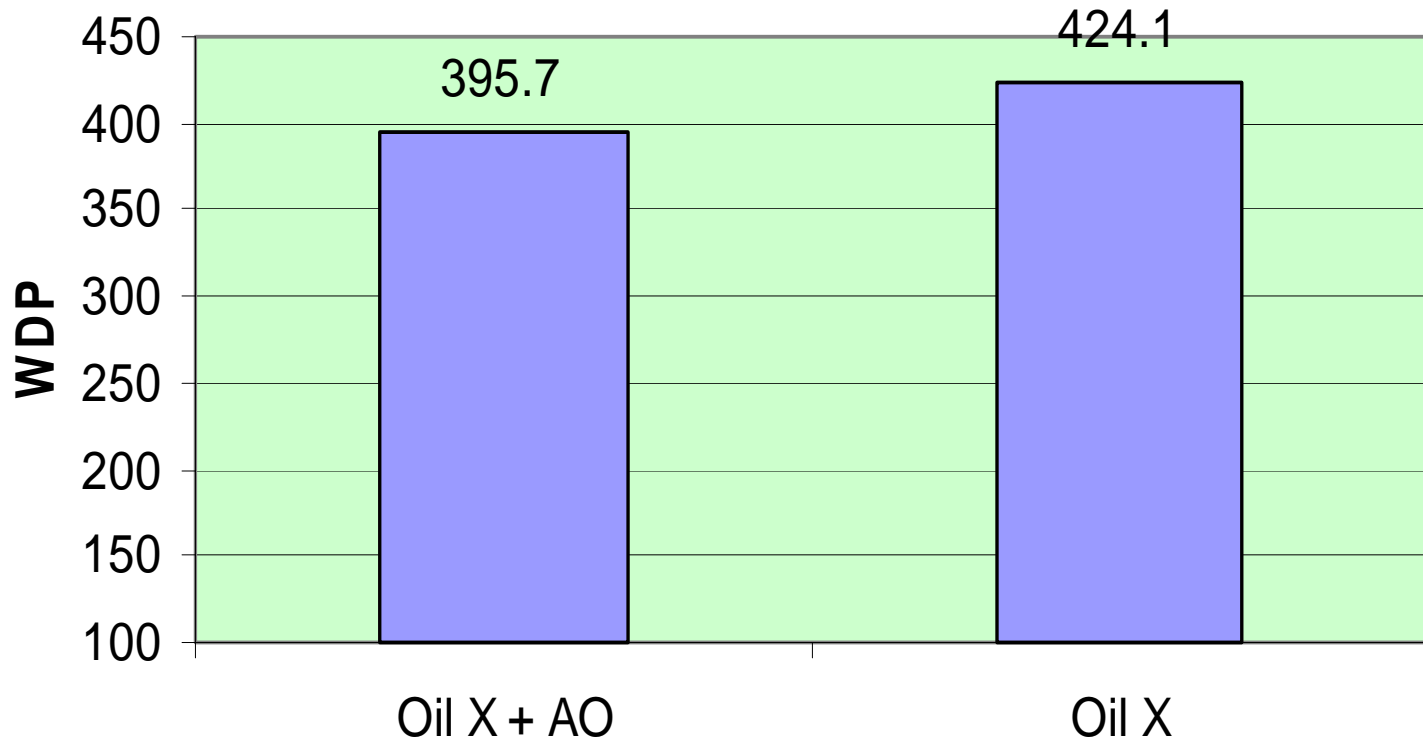
CAT 1R Effects of Oil Oxidation



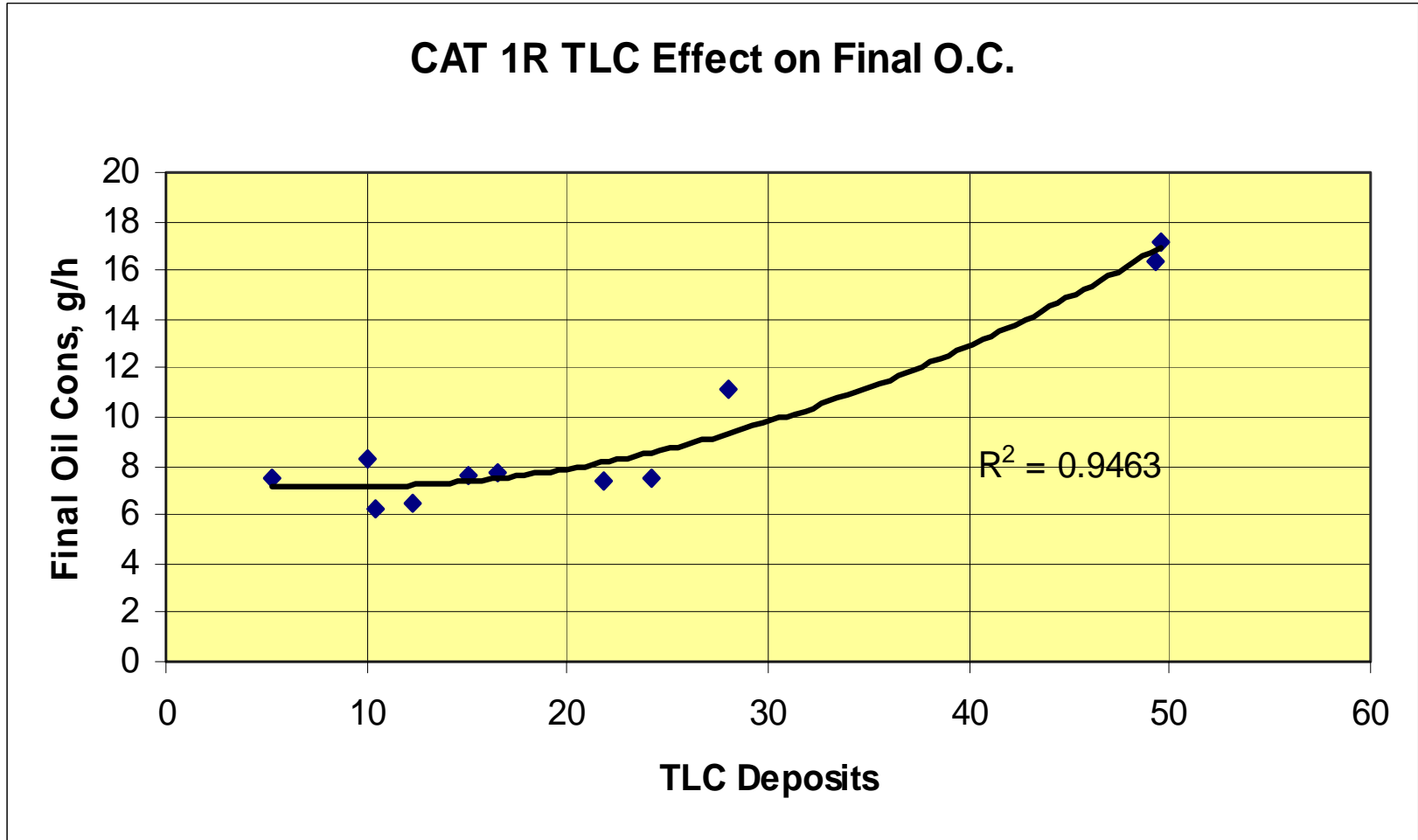
CAT 1R Test Results



CAT 1R Effects of Oil Oxidation



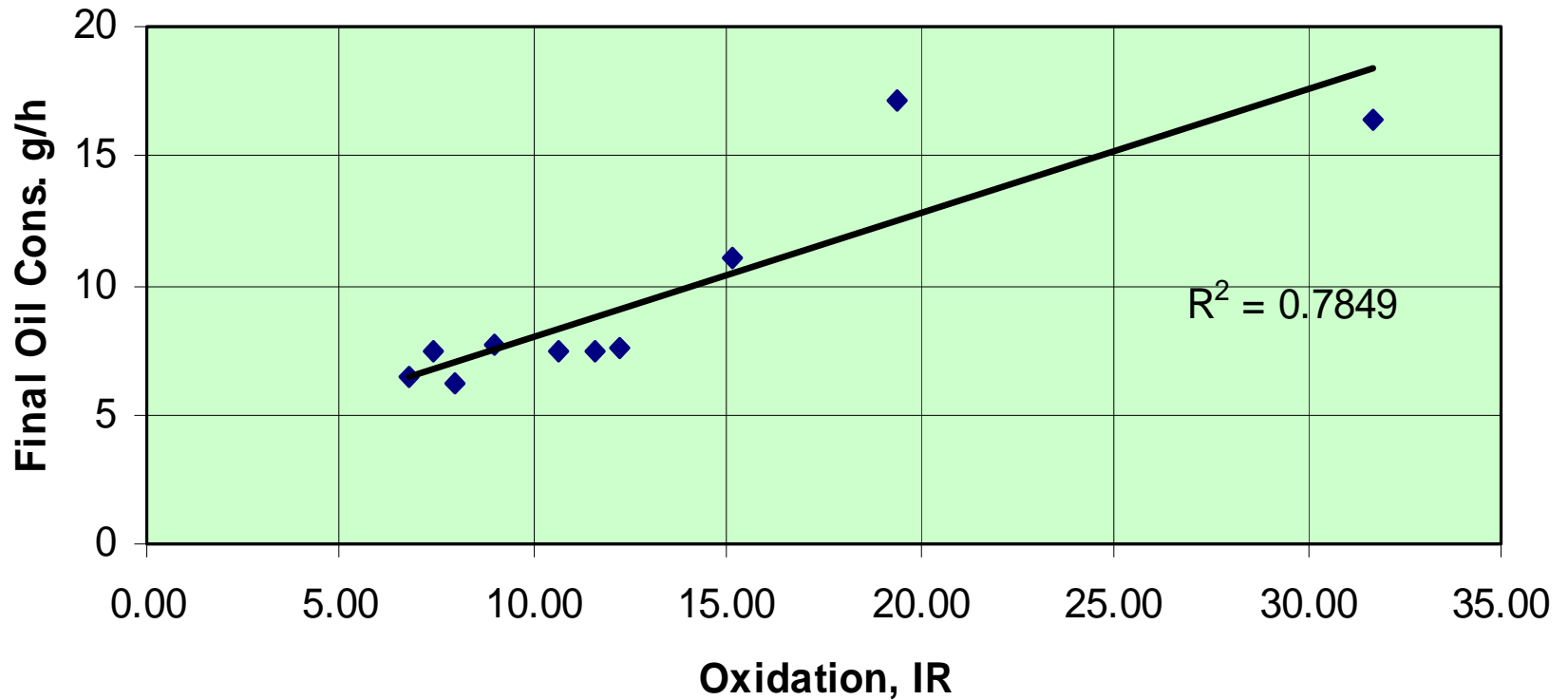
CAT 1R Test Results



CAT 1R Test Results



CAT 1R Effect of Oxidation on Final O.C.



Summary



- **Preliminary data does not support the CAT 1R read across from Group II to Group I base oils.**
- **CAT 1R TLC and TGF deposits are increased with increased oil oxidation. WDP does not separate oils on oxidation level.**
- **CAT 1R oil consumption increases with increasing TLC deposits.**
- **CAT 1R read across should be from the least oxidation stable case to the most. Perhaps it could be based on base oil saturates.**

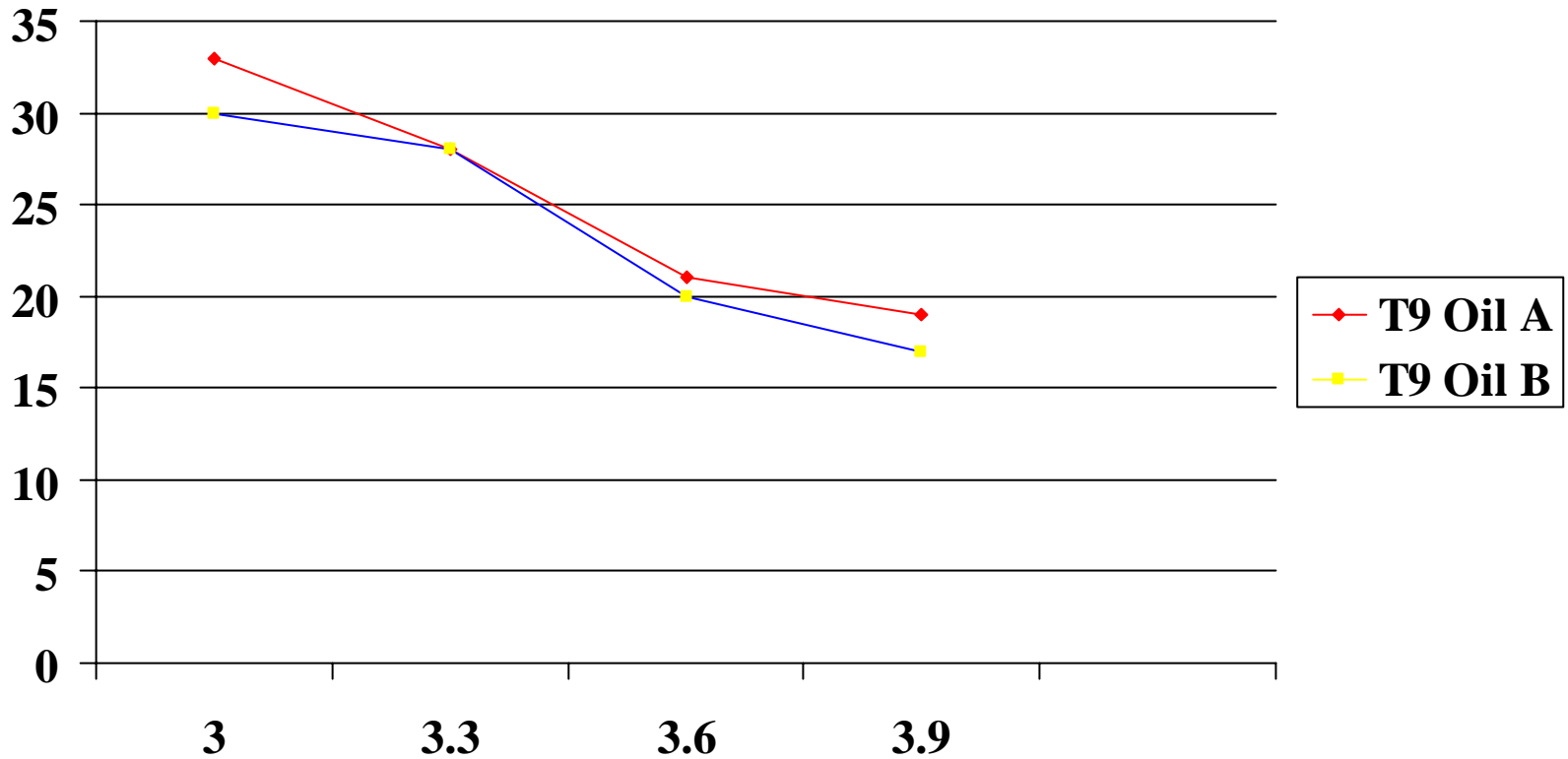
EMA'S PC9 HT/HS Recommendation

- 3.3 Minimum for XW30 after Shear
Engine sees sheared oil
No physical or chemical spec
- 3.5 Minimum for XW30 for new oil
Engine Test Wear Data
ACEA & Global DHD-1
- 3.3 Minimum in HEUI Test @ 5,10 hrs
Heavy Duty Diesel Engine Test

Mack T9

Liner Wear vs HTHS

LWS (um)

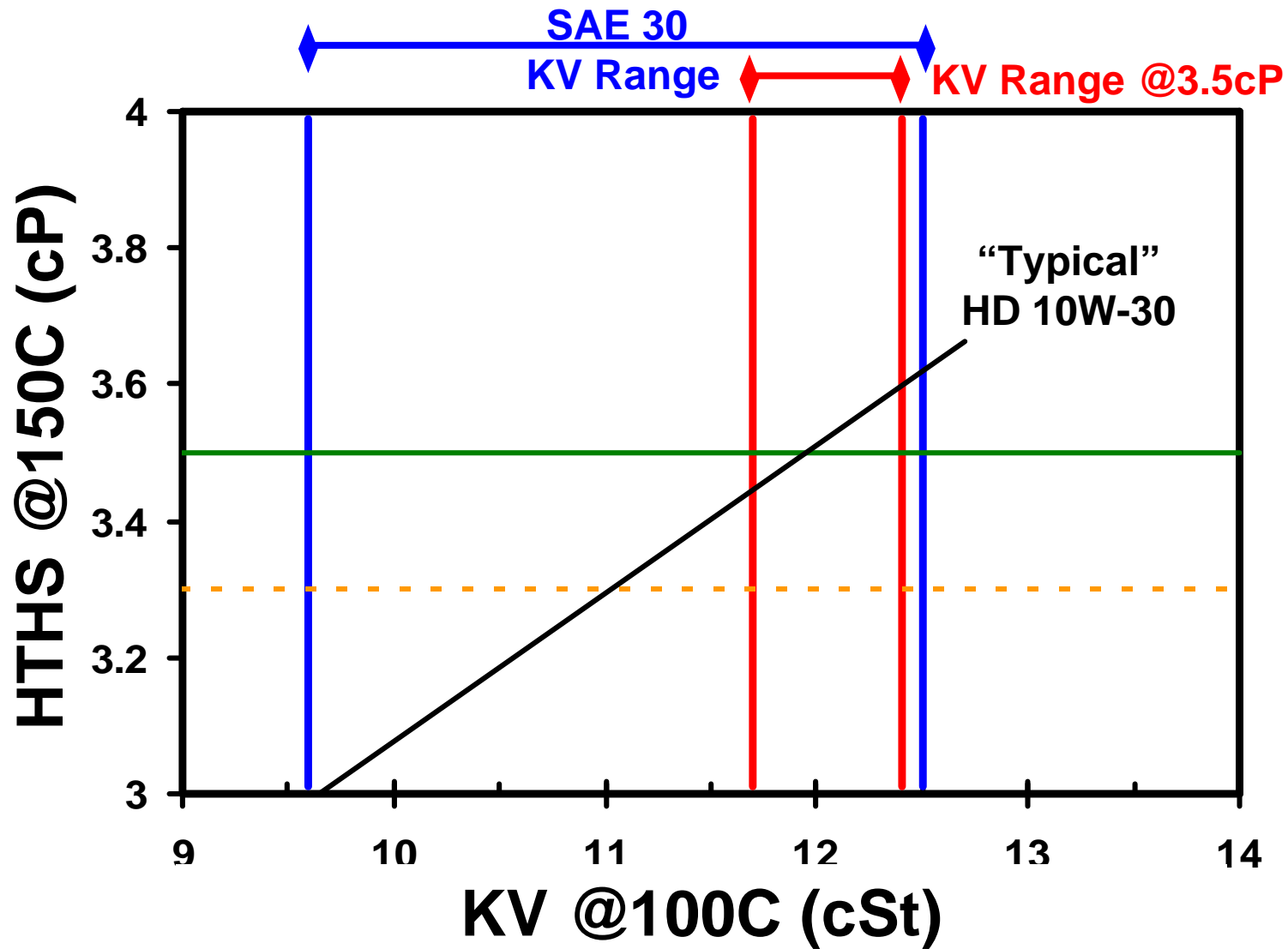


PC-9 HTHS Requirement for 10W-X Grades

ExxonMobil Perspective

- Prefer measurement on new oil
 - More reproducible, eliminates variability of oil source for sheared sample
- Minimum of 3.5 cP is probably achievable with PC-9 technology, however:
 - Allowable “blend space” is very limited, will be a challenge for commercial blending
 - Directionally poorer low temperature properties
 - Smaller fuel economy benefit
- If 3.5 cP is adopted as the PC-9 minimum HTHS for 10W-X oils; it should be identified as a “Non-Critical” specification

PC-9 HTHS Requirement for 10W-X Grades



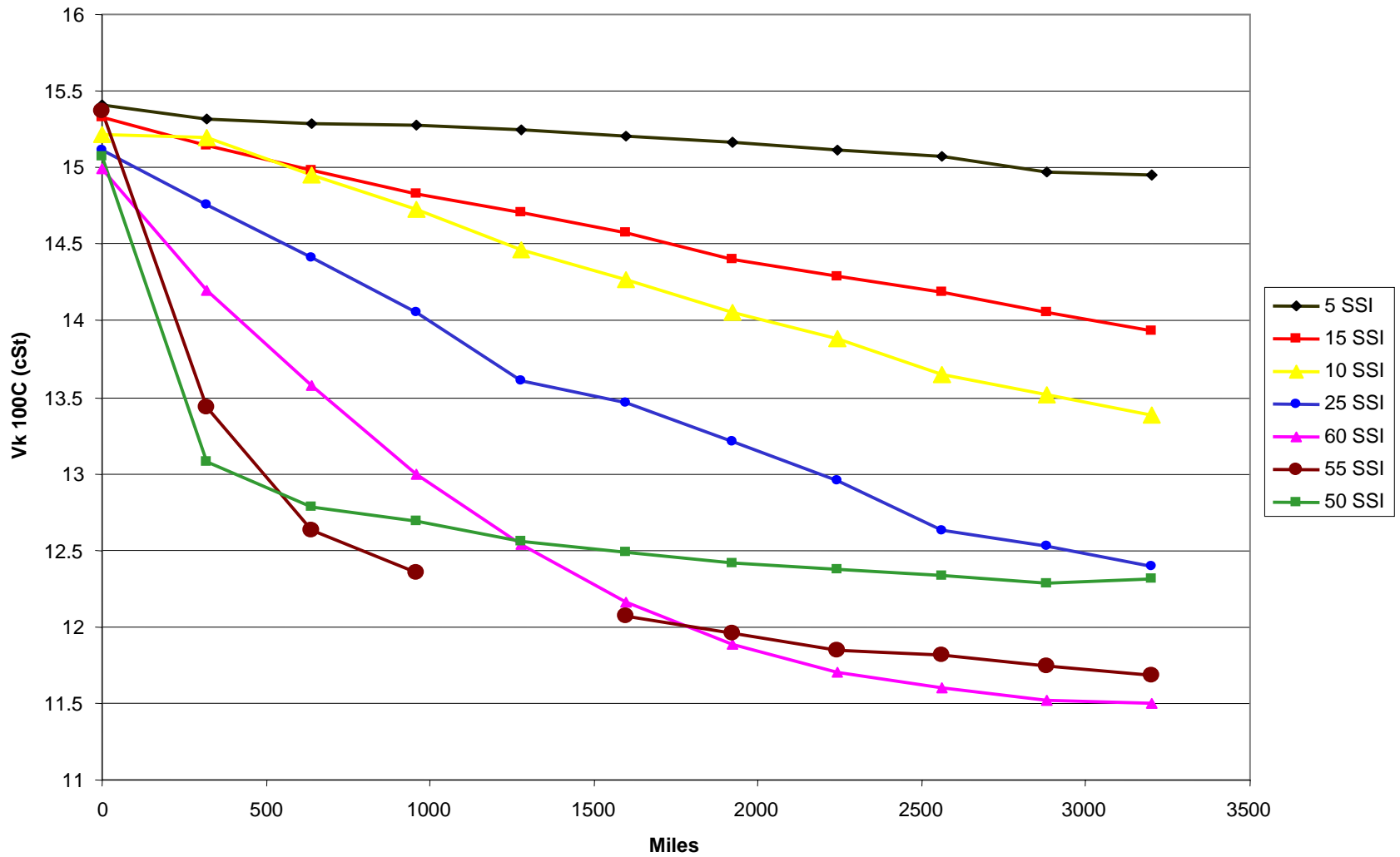
Infineum Concerns

- Inherent dislike for any arbitrary chemical or physical limits on oil formulations.
- Strong preference for performance specifications.
- Any viscometric limits on fresh oils can discriminate against the use of more shear-stable polymers.
 - As noted previously, KV100 loss and HTHS loss are related.
 - The current KV100 stay in grade limits in API CH-4 apply to oils after shear, not fresh.
- No data has been shown to support the need for increased HTHS viscosity.
 - Higher HTHS viscosity will adversely impact fuel economy.

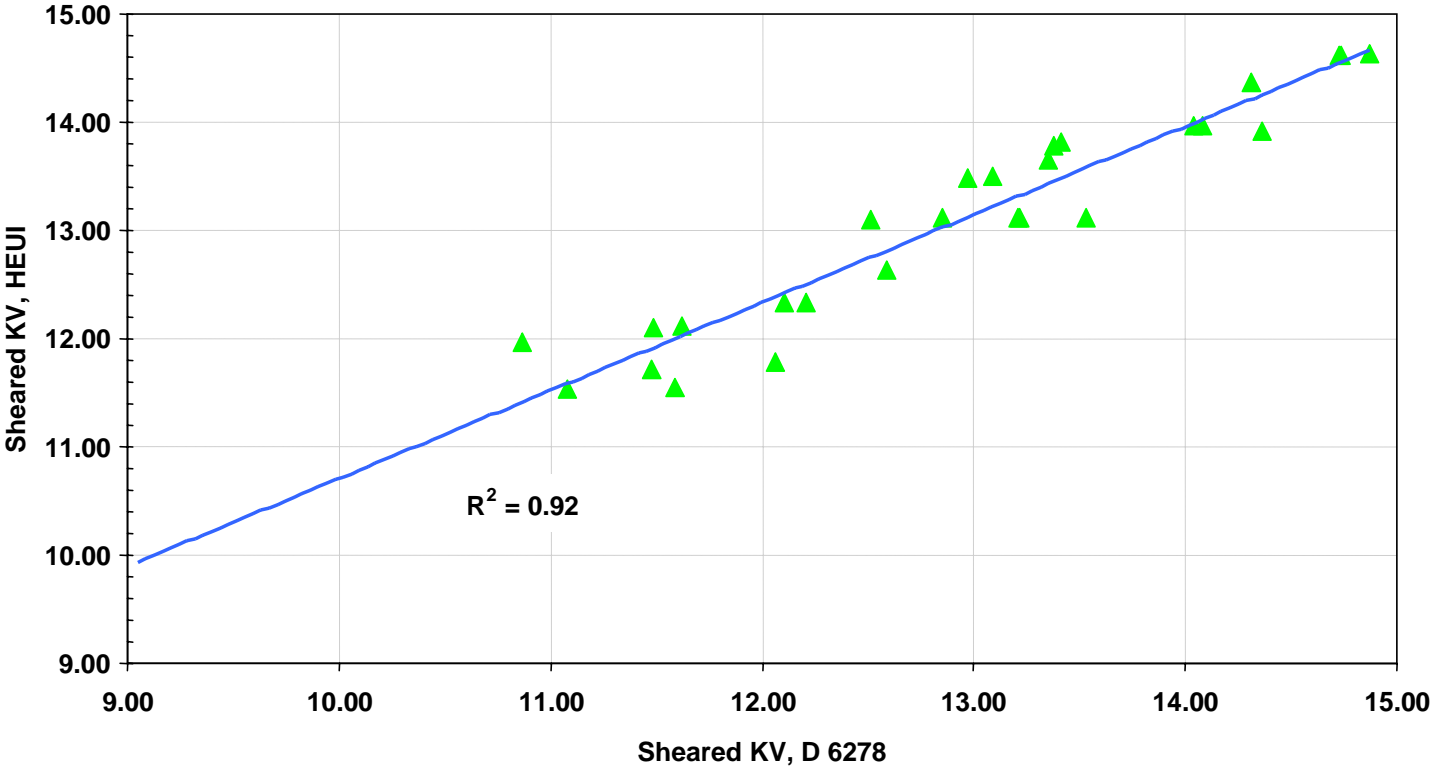
Potential HTHS Concerns

- Current SAE J300 limit of 2.9 cP for 10W-30 viscosity grade is biased toward passenger car fuel economy.
- Limits applied to fresh oil may not reflect actual performance in engine after even short periods of service.
 - A 2.9 cP oil with a 50 SSI viscosity modifier will shear to 2.5 cP.
 - Even the previously requested 10W-35 limit of 3.3 cP on a fresh oil with a 50 SSI viscosity modifier would shear to 2.9 cP.
- Idle oil pressure and minimum oil film thickness in bearings are related to lubricant viscometrics.
 - Both KV100 loss and HTHS loss after shear are related to one another as well as to the SSI of the viscosity modifier.

Viscosity Loss in the HEUI Equipped Trucks

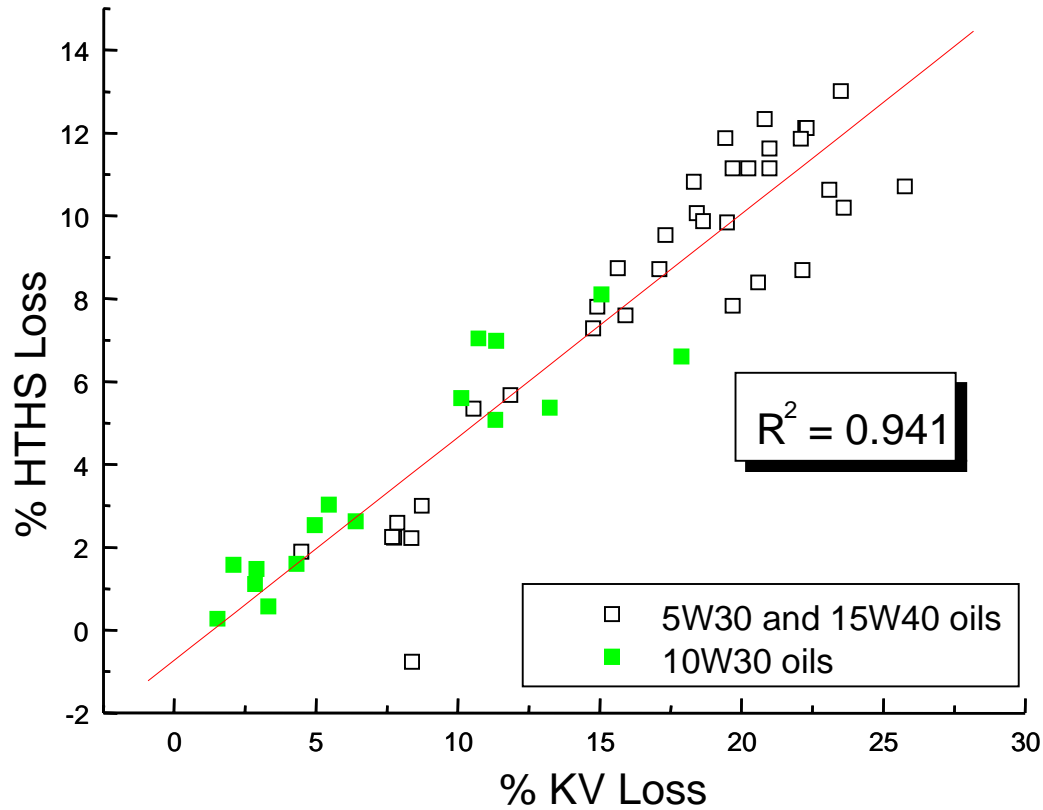


HEUI - D 6278 Correlation for 15W40 Formulations



Excellent correlation between field performance in HEUI equipped trucks and D-6278 bench shear stability test demonstrated for CH-4 development.

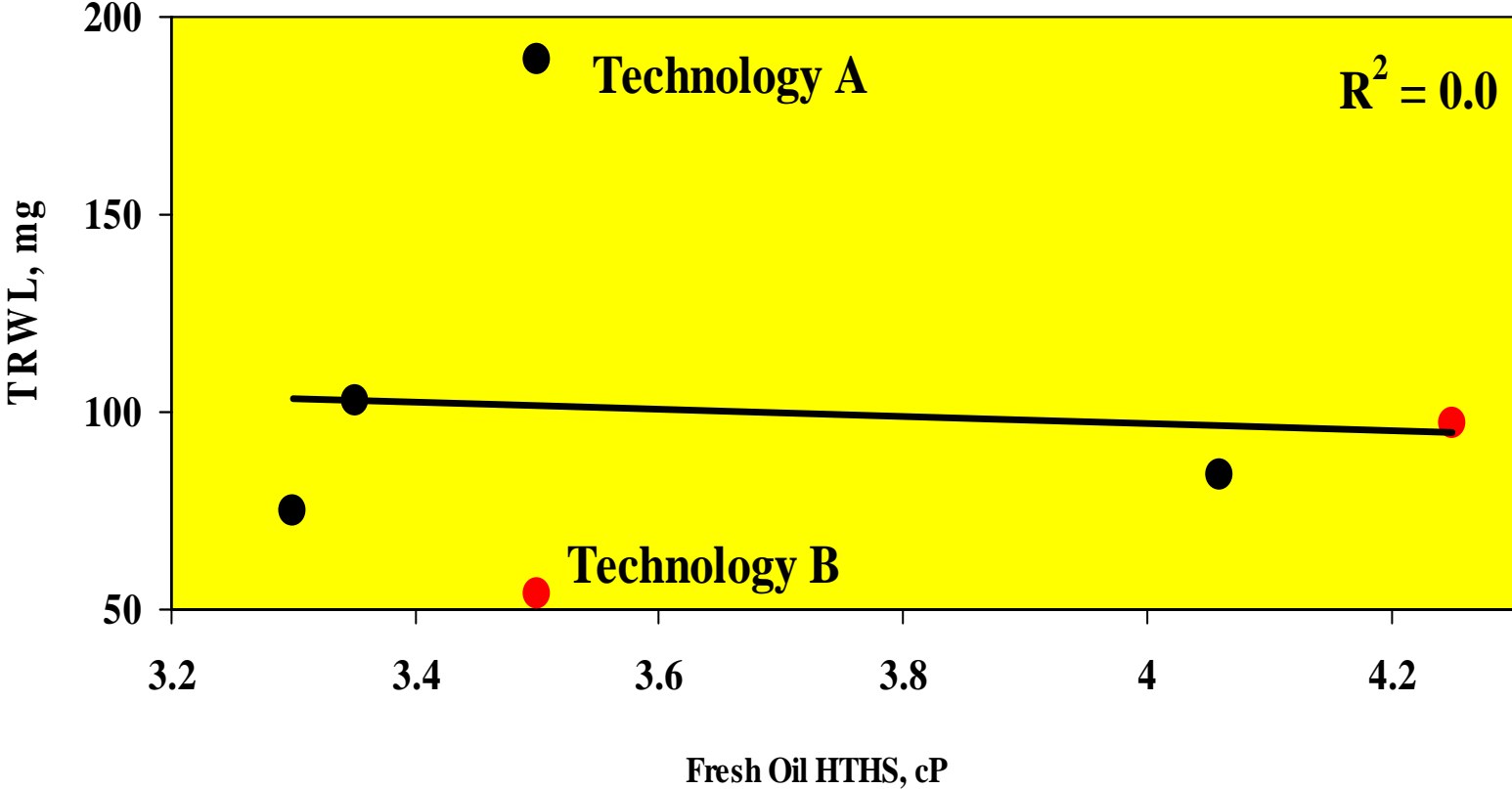
% HTHS Viscosity Loss vs. % KV Loss (5W30, 10W30 and 15W40 Grades)



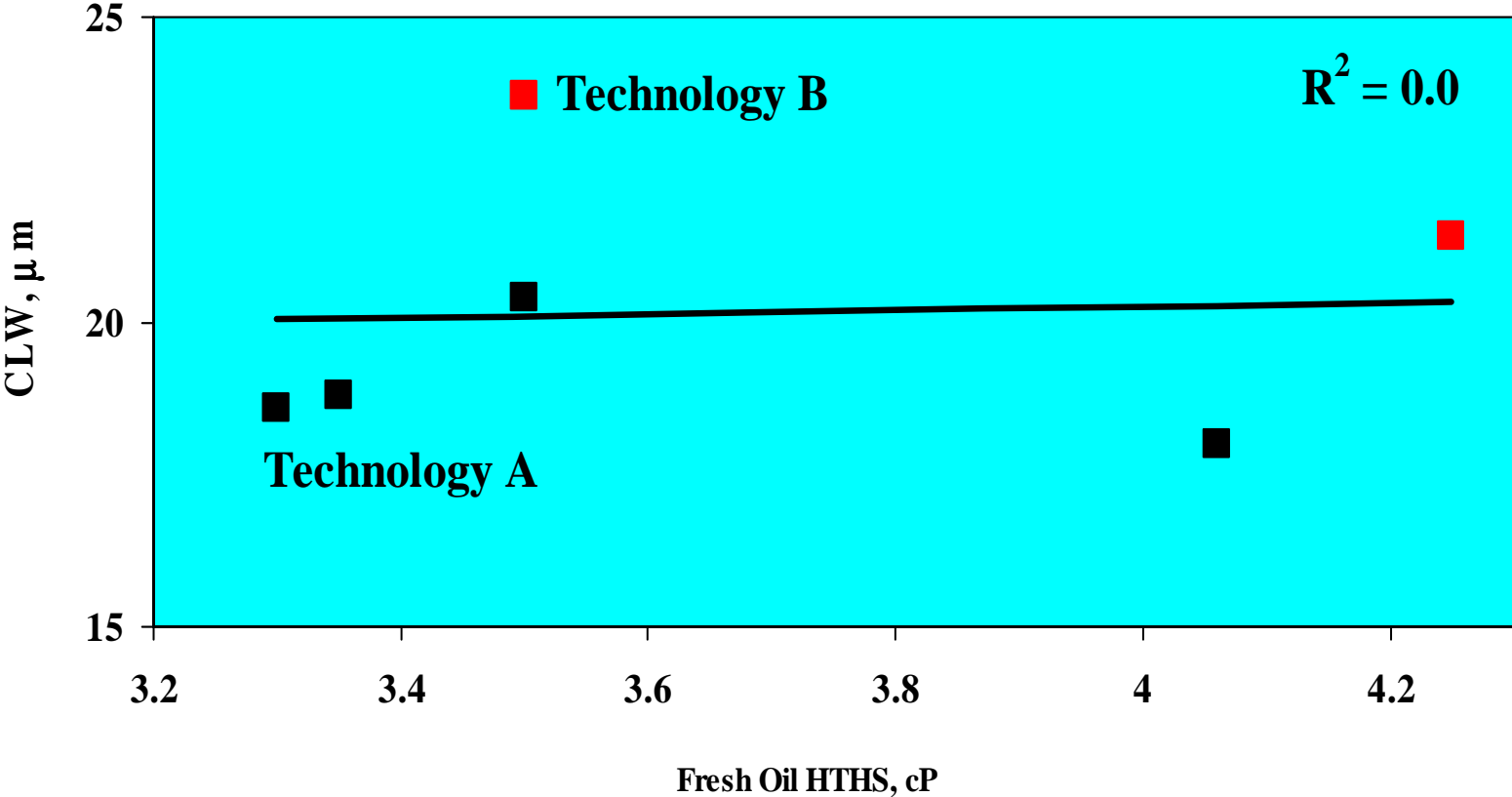
- Good correlation between % HTHS loss and % KV loss for wide range of viscosity grades.

Mack T-9 Performance versus HTHS

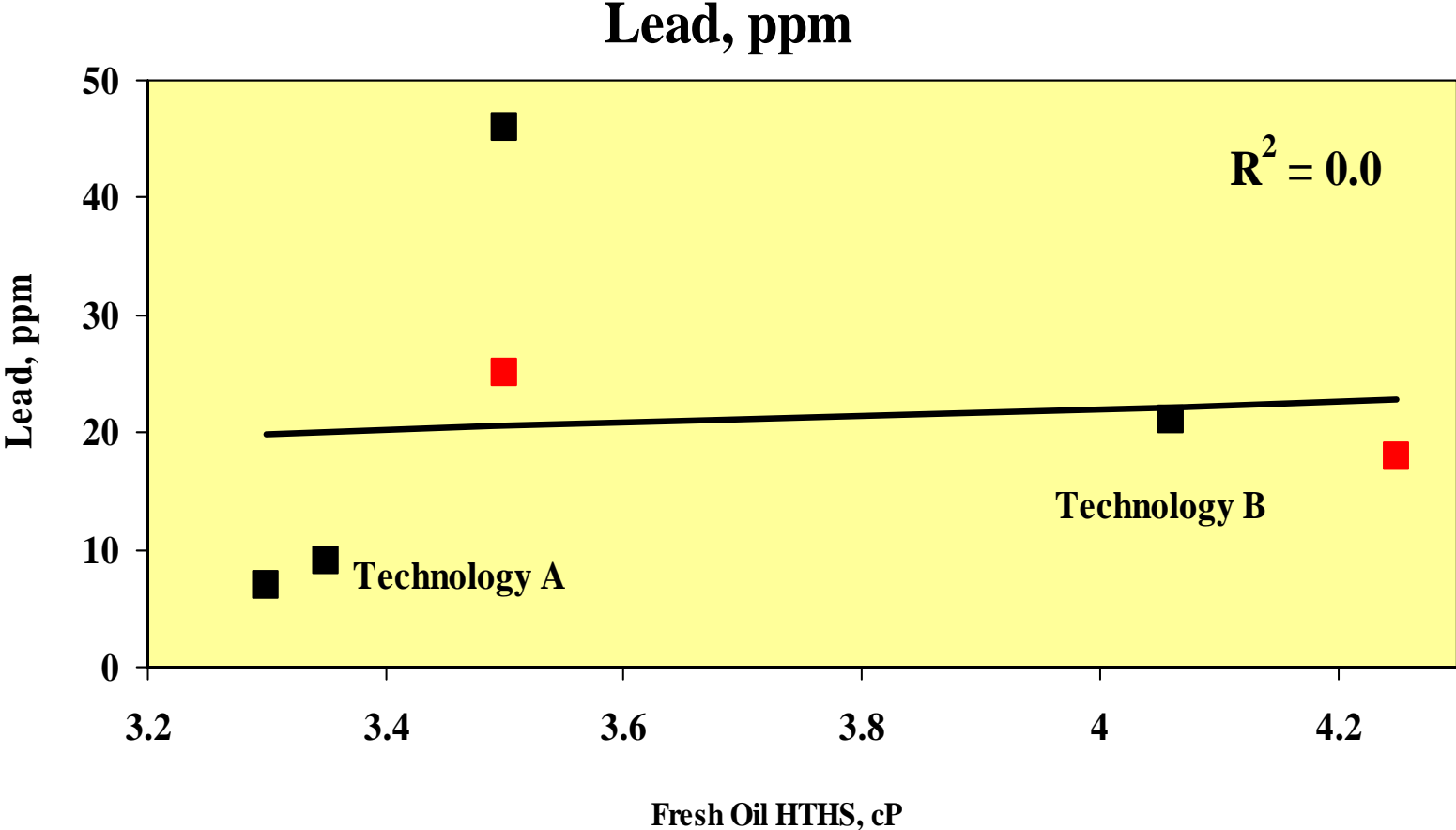
Total Ring Wt Loss



Mack T-9 Performance versus HTHS Cylinder Liner Wear



Mack T-9 Performance versus HTHS



Observations on 10W-30 HTHS.

- KV100 loss and HTHS loss are related to both one another and to the shear stability of the viscosity modifier.
 - The existing KV100 stay in grade requirement of 9.3 cSt after shear guarantees a minimum after shear HTHS of 3.0 cP, regardless of VM SSI.
 - This is well above the 2.5 cP after shear minimum which could happen with a 50 SSI polymer oil just meeting J300.
- The EMA accepted viscosity grade read for the new PC-9 tests looking at, among other parameters, engine wear is from 10W-30 to 15W-40.
 - 10W-30's must prove wear capability!

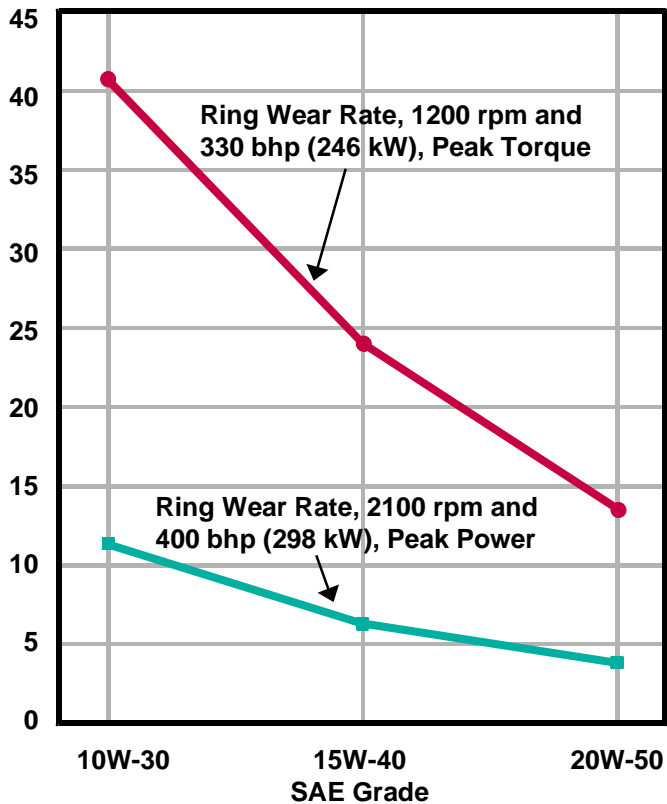
Summary

- The use of an after shear HTHS limit makes the parameter a performance test, not an arbitrary chemical/physical limit
- The existing KV100 limit of 9.3 cSt minimum guarantees an after shear HTHS minimum of 3.0 cP
- No data has been shown to support higher HTHS viscosity.
- Lower HTHS viscosity improves fuel economy.
- SAE 10W-30 oils will need to pass the engine wear tests to prove their performance capability.
- Infineum recommends an after shear HTHS to 3.0 cP minimum to conform to the current 9.3 cSt KV100 limit.
 - 15W-40 after shear HTHS of 3.7 cP minimum is not a problem.

SAE 932845, 1993

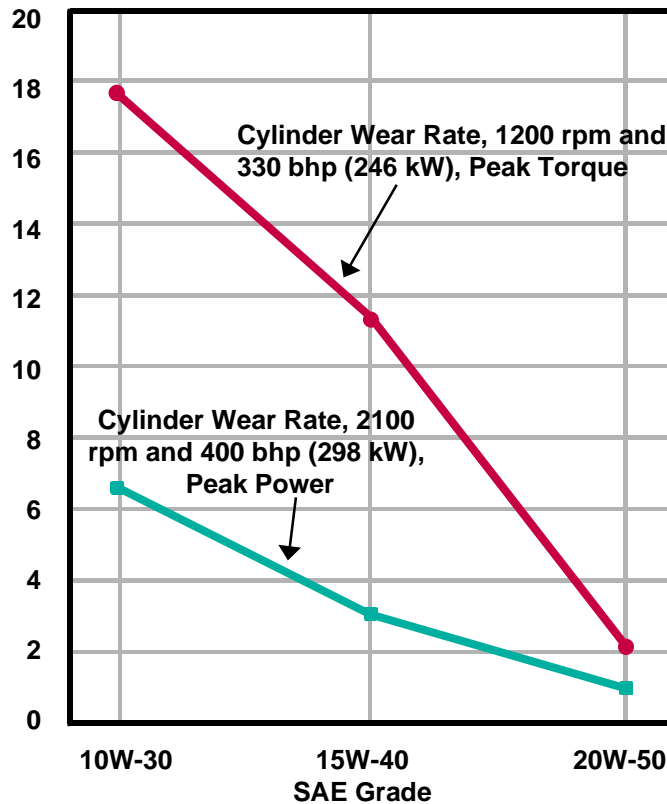
Increasing HT/HS Reduces Ring and Liner Wear

Chromium Piston Top Ring Wear Rate, nM/Hr, Hardened Cylinder Liner



(HT/HS) (3.2) (4.2) (5.3)

Cylinder Liner Wear Rate, nM/Hr, at Top Ring Reversal – Plasma Sprayed Top Ring and Hardened Liner



(HT/HS) (3.2) (4.2) (5.3)

Detroit Diesel Series 60

SLA Wear Technique for Ring and Liner Wear

Summary

Ref: SAE 932845

- Study Used the Same Base Oil, VI Improver and Additive Packages for All Three Viscosity Grades: SAE 10W-30/15W-40/20W-50
- SAE 10W-30 Produced Higher Wear Than SAE W-40 and 20W-50 – It Was Significantly Different From 20W-50 for All Four Measurements and it Was Significantly Different From the SAE 15W-40 for All, But Liner Wear at 1200 RPM.

ASTM D2 Meeting - Chicago, IL
July 11, 2001

**UPDATE ON ASTM LOTRUO
ACTIVITIES AND LOW TEMPERATURE
PROPERTIES OF PC-9 MATRIX OILS**

C.J. May

MRV Measurements of Sooted Oils

LOTRUO Activities

- **Following successful completion of round robin, method modifications and research report are in progress covering measurement of used, highly sooted oils by standard (D4684) and modified (external preheat) methods**
 - **viscosity precision statements obtained for both**
 - **expect to go to ballot within ~4-5 weeks**

IOL Data on PC-9 E-O-T Matrix oils

- **Have received 15 Mack T10 samples and 16 M11-EGR samples for analysis**
 - **virtually complete CCS/MRV/mod. MRV dataset on T10 samples**
 - **TMC website lists 11 T10 runs, 7 M11-EGR runs for which IOL has not received samples.**

LOW TEMP DATA ON T10 MATRIX OILS

SRC Data, Updated: July 9, 2001

CMIR Code	Matrix Code	Lab Code	TGA		-20°C D4684		-25°C D4684		-20°C Mod. MRV		-25°C Mod. MRV		
			Soot %	D5293	MRV	MRV Y. Str., Pa	MRV	MRV Y. Str., Pa	MRV	MRV Y. Str., Pa	MRV	MRV Y. Str., Pa	
38810	PC-9A	A	6.0	7,030		24,500	0<Y<=35	53,100	0<Y<=35	23,600	0<Y<=35	54,200	0<Y<=35
38811	PC-9A	D	5.5	5,900	11,910	19,900	0<Y<=35	43,900	0<Y<=35	19,800	0<Y<=35	43,100	0<Y<=35
38814	PC-9A	F	5.7	7,990		26,400	0<Y<=35	59,300	0<Y<=35	26,400	0<Y<=35	59,400	0<Y<=35
38942	PC-9A	A	4.8	5,900		19,100	0<Y<=35	42,100	0<Y<=35	19,000	0<Y<=35	41,800	0<Y<=35
38951	PC-9A	G	5.9	7,090		22,800	0<Y<=35	51,000	0<Y<=35	23,100	0<Y<=35	51,500	0<Y<=35
38939	PC-9C	A	5.4	7,650		23,200	0<Y<=35	61,300	0<Y<=35	22,700	0<Y<=35	58,700	0<Y<=35
38949	PC-9C	G	7.6	12,350		37,300	0<Y<=35	95,000	0<Y<=35	37,100	0<Y<=35	96,200	0<Y<=35
38937	PC-9E	A	4.8	5,190		19,500	0<Y<=35	102,400	140<Y<=175	20,100	0<Y<=35	203,500	175<Y<=210
38945	PC-9F	D	5.3	6,020		17,300	0<Y<=35	76,100	35<Y<=70	17,300	0<Y<=35	69,100	0<Y<=35
38947	PC-9H	G	7.1	7,270		19,900	0<Y<=35	57,100	0<Y<=35	19,700	0<Y<=35	58,100	0<Y<=35
38953	PC-9H	F	5.2	5,630		14,600	0<Y<=35	45,300	0<Y<=35	14,400	0<Y<=35	44,900	0<Y<=35
38941	PC-9G	A	5.5	6,460		18,900	0<Y<=35	42,300	0<Y<=35	18,900	0<Y<=35	46,900	0<Y<=35
38938	PC-9J	A	6.2	6,190		17,100	0<Y<=35	42,700	0<Y<=35	17,200	0<Y<=35	44,000	0<Y<=35
38948	PC-9J	G	5.7	6,370		17,600	0<Y<=35	41,000	0<Y<=35	17,300	0<Y<=35	40,900	0<Y<=35
38813				8,290		25,900	0<Y<=35	60,200	0<Y<=35	26,900	0<Y<=35	59,900	0<Y<=35
38940	PC-9E	A	5.9	5,190		23,600	35<Y<=70	262,100	175<Y<=210	24,200	35<Y<=70	260,700	175<Y<=210

LOW TEMP DATA - M11 EGR MATRIX OILS

SRC Data, Updated: July 9, 2001

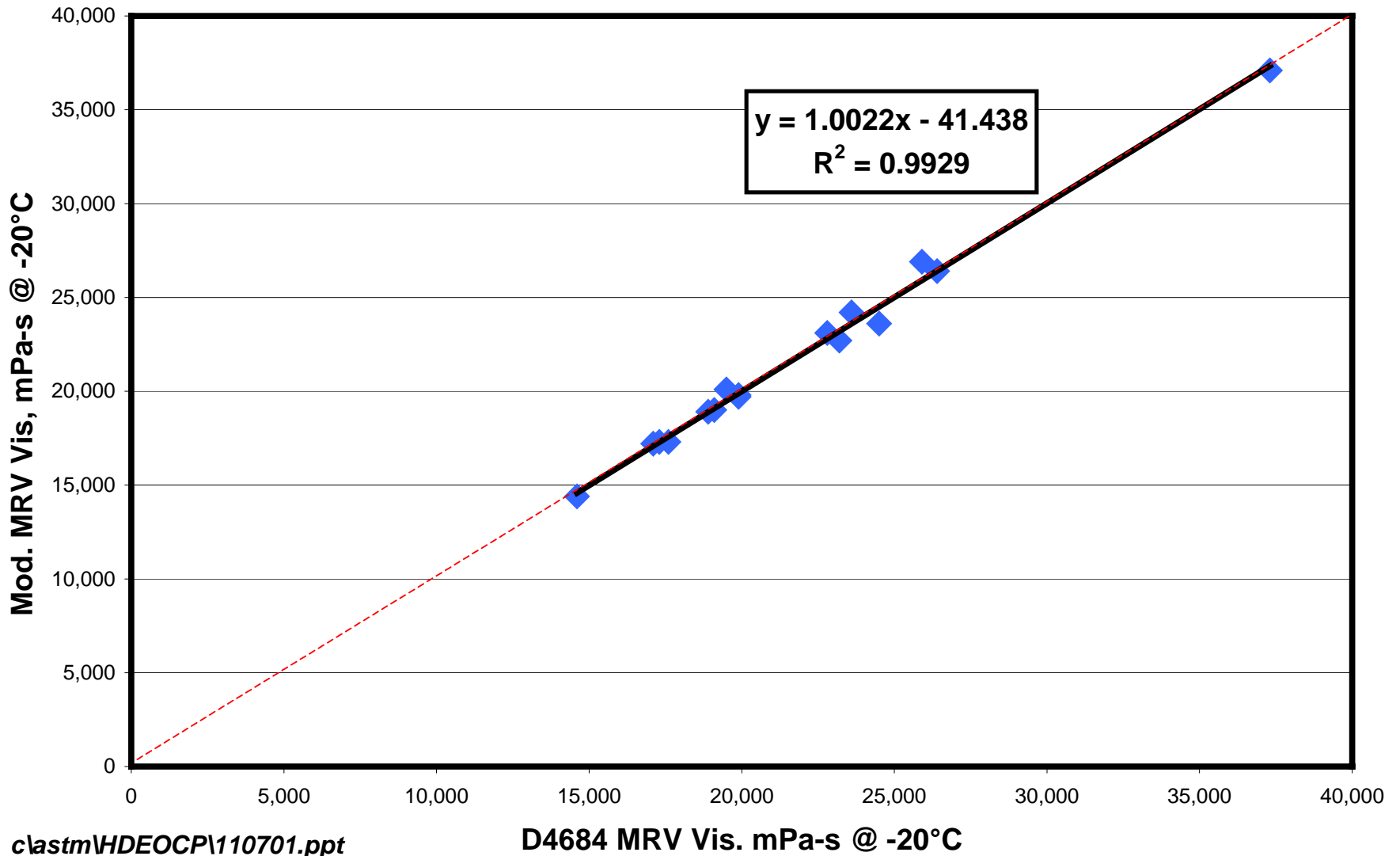
CMIR Code	Matrix Code	Lab Code	TGA										
			Soot %	D5293		-20°C D4684		-25°C D4684		-20°C Mod. MRV		-25°C Mod. MRV	
				-15C, cP	-20C, cP	MRV Vis., cP	Y. Str., Pa	MRV Vis., cP	Y. Str., Pa	-20C MRV Vis., cP	Y. Str., Pa	-25C MRV Vis., cP	Y. Str., Pa
38967	PC-9B	A	8.0	6,320		22,600	0<Y<=35	70,400	0<Y<=35	22,400	0<Y<=35	70,700	0<Y<=35
38927	PC-9E	G	9.1	5,590		26,300	0<Y<=35						
38928	PC-9E	G	7.8			22,300	0<Y<=35						
38929	PC-9E	G	8.8			29,400	0<Y<=35	208,500	140<Y<=175			211,700	140<Y<=175
38930	PC-9E	G	8.6	5,190		32,900	0<Y<=35	343,100	140<Y<=175			289,200	140<Y<=175
38931	PC-9E	D	8.1	5,320		28,200	0<Y<=35	214,100	105<Y<=140			187,700	105<Y<=140
38932	PC-9E	A	8.7	5,630		38,900	0<Y<=35	305,400	175<Y<=210				
38933	PC-9E	A	7.7	5,190		24,000	0<Y<=35	135,000	140<Y<=175			140,600	105<Y<=140
38934	PC-9E	A	7.8	5,380		31,300	0<Y<=35	262,300	140<Y<=175			311,200	140<Y<=175
38962	PC-9F	G	8.7	6,460		28,900	35<Y<=70	133,600	70<Y<=105			106,800	35<Y<=70
38968	PC-9A	A	7.9			23,000	0<Y<=35						
38935	PC-9E	A	8.0			23,800	0<Y<=35						
38969	PC-9G	A	7.8	5,520		64,800	210<Y<=245	683,700	315<Y<=350			529,700	210<Y<=245
38966	PC-9J	A	8.0	5,520		31,800	105<Y<=140	77,900	140<Y<=175			75,900	140<Y<=175
38958	PC-9C	G	7.4*			20,800	0<Y<=35	55,100	0<Y<=35			52,400	0<Y<=35
38970	PC-9F	A	7.9	5,940		24,600	0<Y<=35						

MATRIX TESTS POSTED BY TMC, BUT NO USED OIL SAMPLES RECEIVED

SRC Data, Updated: July 9, 2001

CMIR Code	Matrix Code	Lab Code	<u>TGA</u>
			<u>Soot</u> TMC
38809	PC-9A	A	
38815	PC-9A	B	
38954	PC-9A	F	
38943	PC-9B	D	
38957	PC-9D	B	
38952	PC-9F	F	
40919	PC-9B	B	
38946	PC-9D	G	
38950	PC-9E	G	
38944	PC-9G	D	
38956	PC-9J	B	
38936	PC-9E	B	
38963	PC-9D	D	
38968	PC-9A	A	
38935	PC-9E	A	
38959	PC-9A	G	
38971	PC-9D	B	
38961	PC-9G	G	

Comparison of Standard D4684 vs Modified MRV: T10 E-O-T Samples, -20°C



REPEAT T10 TESTS ON PC-9A, MRV USED/FRESH

CMIR Code	38811	38814	38810	38951	38942		
Ind. Oil Code	PC-9A	PC-9A	PC-9A	PC-9A	PC-9A	PC-9A	PC-9A
Lab Code	D	F	A	G	A		
Engine Source	T-10	T-10	T-10	T-10	T-10		(Fresh)
% Soot Reported	5.5	5.7	6.0	5.9	4.8		

@ -20°C

Averages

MRV Vis., cP	19,900	26,400	24,500	23,400	19,100	22,660	11,600
MRV Y. Str., Pa	0<Y<=35	0<Y<=35	0<Y<=35	0<Y<=35	0<Y<=35		0<Y<=35

@ -25°C

MRV Vis., cP	43,900	59,300	53,100	51,000	42,100	49,880	23,200/23,900**
MRV Y. Str., Pa	0<Y<=35	0<Y<=35	0<Y<=35	0<Y<=35	0<Y<=35		0<Y<=35

@ -20°C

Mod. MRV Vis., cP	19,800	26,400	23,600	23,100	19,000	22,380	
Mod. MRV Y. Str., Pa	0<Y<=35	0<Y<=35	0<Y<=35	0<Y<=35	0<Y<=35		

@ -25°C

Mod. MRV Vis., cP	43,100	59,400	54,200	51,500	41,800	50,000	
Mod. MRV Y. Str., Pa	0<Y<=35	0<Y<=35	0<Y<=35	0<Y<=35	0<Y<=35		

* TMC Website Data

REPEAT T10 TESTS, PC-9C,-9H: MRV USED/FRESH

CMIR Code	38939	38949		38953	38947	
Ind. Oil Code	PC-9C	PC-9C	PC-9C	PC-9H	PC-9H	PC-9H
Lab Code	A	G		F	G	
Engine Source	T-10	T-10	(Fresh)	T-10	T-10	(Fresh)
% Soot Reported	5.4	<u>7.6</u>		5.2	7.1	

@ -20°C

MRV Vis., cP	23,200	37,300	11,700	14,600	19,900	7,600
MRV Y. Str., Pa	0<Y<=35	0<Y<=35	0<Y<=35	0<Y<=35	0<Y<=35	0<Y<=35

@ -25°C

MRV Vis., cP	61,300	95,000	25,800/25,200*	45,300	57,100	18,200/19,100*
MRV Y. Str., Pa	0<Y<=35	0<Y<=35	0<Y<=35	0<Y<=35	0<Y<=35	0<Y<=35

@ -20°C

Mod. MRV Vis., cP	22,700	37,100		14,400	19,700	
Mod. MRV Y. Str., Pa	0<Y<=35	0<Y<=35		0<Y<=35	0<Y<=35	

@ -25°C

Mod. MRV Vis., cP	58,700	96,200		44,900	58,100	
Mod. MRV Y. Str., Pa	0<Y<=35	0<Y<=35		0<Y<=35	0<Y<=35	

REPEAT T10 TESTS, PC-9C,-9H: MRV USED/FRESH

CMIR Code	38937	38940		38938	38948	
Ind. Oil Code	PC-9E	PC-9E	PC-9E	PC-9J	PC-9J	PC-9J
Lab Code	A	A		A	G	
Engine Source	T-10	T-10	(Fresh)	T-10	T-10	(Fresh)
% Soot Reported	4.8	5.9		6.2	5.7	

@ -20°C

MRV Vis., cP	19,500	23,600	14,100	17,100	17,600	7,600
MRV Y. Str., Pa	0<Y<=35	35<Y<=70	0<Y<=35	0<Y<=35	0<Y<=35	0<Y<=35

@ -25°C

MRV Vis., cP	102,400	262,100	62,500/59,300*	42,700	41,000	18,200/19,100*
MRV Y. Str., Pa	140<Y<=175	175<Y<=210	<=140/<=140*	0<Y<=35	0<Y<=35	0<Y<=35

@ -20°C

Mod. MRV Vis., cP	20,100	24,200		17,200	17,300	
Mod. MRV Y. Str., Pa	0<Y<=35	35<Y<=70		0<Y<=35	0<Y<=35	

@ -25°C

Mod. MRV Vis., cP	203,500	260,700		44,000	40,900	
Mod. MRV Y. Str., Pa	175<Y<=210	175<Y<=210		0<Y<=35	0<Y<=35	

PROPERTIES OF FRESH PC-9 MATRIX OILS

Oil	Base Oil	DI Chem	TMC Data				IOL Data	
			KV@ 100°C	CCS@- 15°C	HTHS, cP	MRV @-25C	MRV@-20C	MRV@-25C
PC-9A	1	X	15.20	3304	4.22	23,900/NYS	11,600/NYS	23,200/NYS
PC-9B	2	X	15.18	3466	4.27	27,950/NYS	11,400/NYS	26,100/NYS
PC-9C	3	X	15.14	3500	4.26	25,168/NYS	11,700/NYS	25,800/NYS
PC-9D	1	Y	15.76	3128	4.17	51,600/30g	19,300/35 Pa	73,400/105 Pa
PC-9E	2	Y	15.47	3249	4.29	59,300/40g	14,100/NYS	62,500/105 Pa
PC-9F	3	Y	16.03	3430	4.32	51,100/NYS	11,500/NYS	50,800/NYS
PC-9G	1	Z	15.13	3450	4.07	29,500/NYS	11,200/NYS	37,700/35 Pa
PC-9H	2	Z	15.13	3350	4.14	19,100/NYS	7,600/NYS	18,200/NYS
PC-9J	3	Z	15.07	3155	4.16	17,300/NYS	7,700/NYS	16,500/NYS

Recommendations

- **If HDEOCP requires Mack T10 used oil MRV measurements at 5°C above fresh oil pumpability temperature (e.g. -20°C for test oil originally an SAE 15W-40), then the standard D4684 method should work well (based on PC-9 matrix oil data)**
 - **used oil viscosities very similar for both methods at this temperature**
 - **low evidence of yield stress at this temperature for either method**
 - **more straightforward for labs to run**
 - **modified method will be available should future requirement encompass higher soot level of used oil**

Low Temperature Pumpability of Used Heavy Duty Diesel Engine Oils from Field Trials

Presentation to HDEOCP
Chicago, IL
July 11, 2001

Jai G. Bansal
G. P. Fetterman

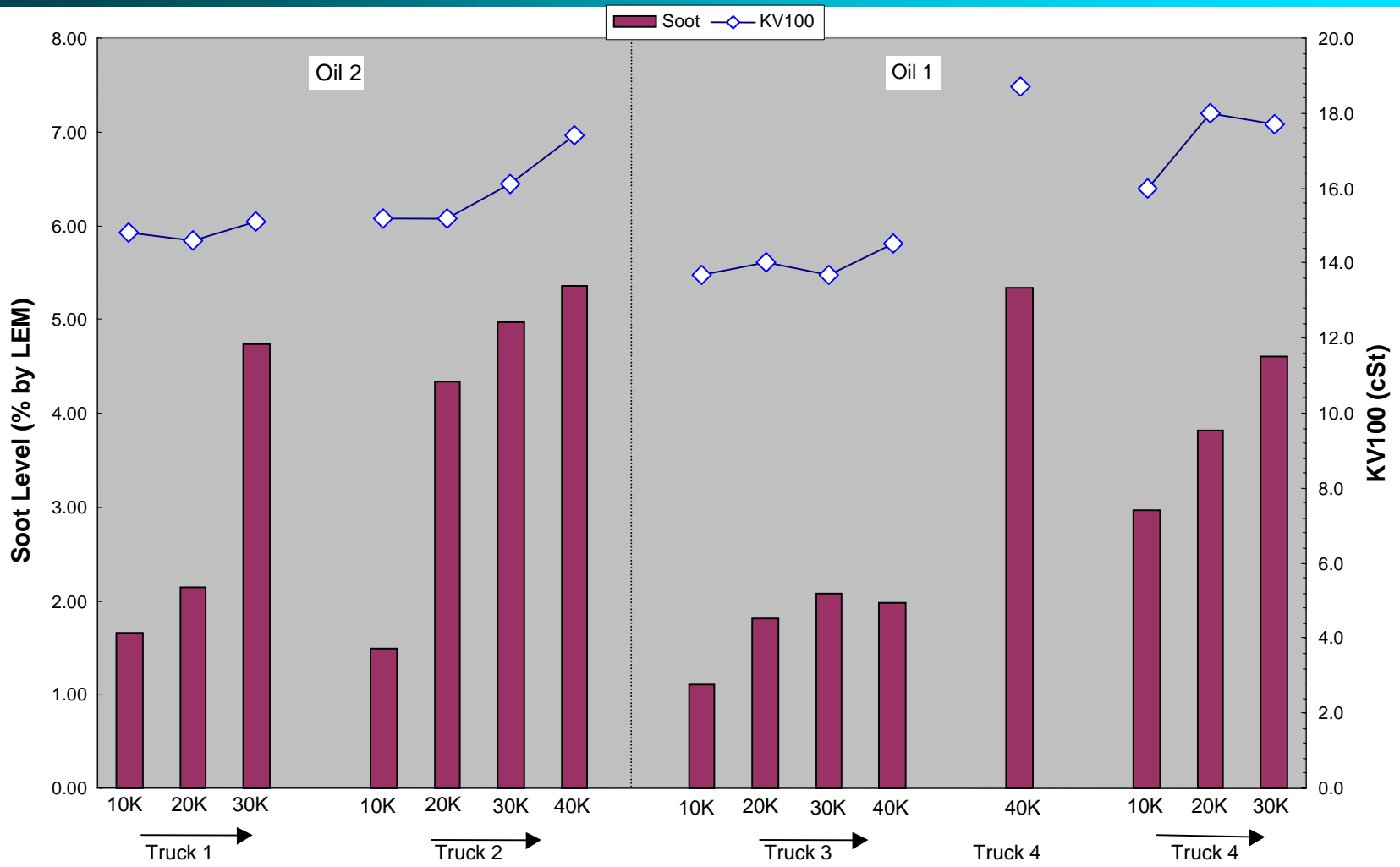
1. Field Test Overview



- ❖ Two SAE 15W-40 oils blended to similar KV100 and CCS
- ❖ Used identical DI additive and Group I basestock
- ❖ Blended to similar KV100 and CCS
- ❖ Both oils comfortably passed fresh oil MRV-TP1
- ❖ Mack E7-375 engines, short haul, full load, 40K miles ODI
- ❖ Oil samples collected at ~10K miles to ODI

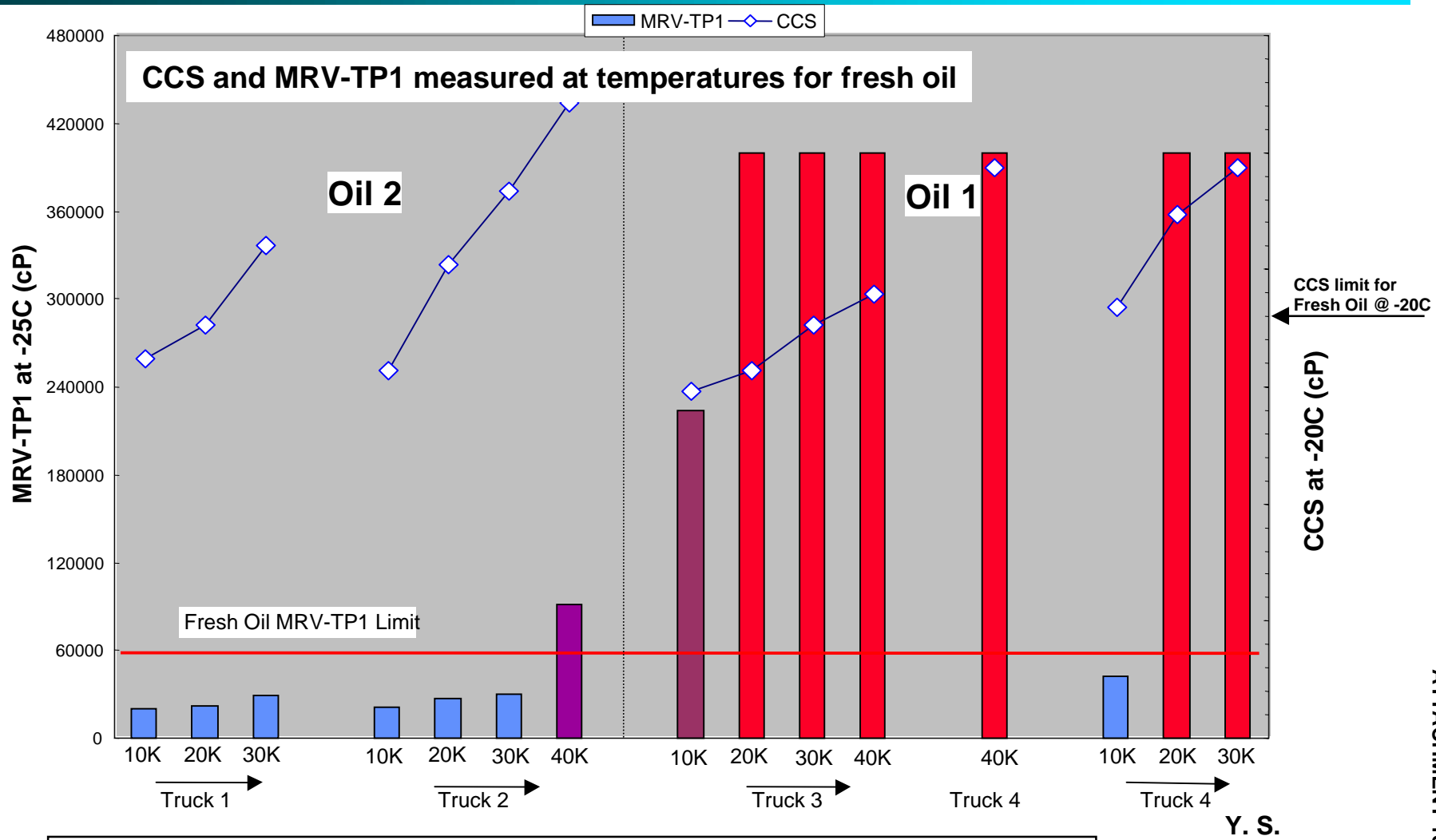
	Oil 1	Oil 2
KV @100C, cSt	15.2	15.47
CCS @-20C, cP	5660	5660
MRV-TP1 @ -25C, cP	15363	21995

Soot and KV100 versus Mileage



- Soot level increased with service
- Additive technology maintains excellent control of KV100

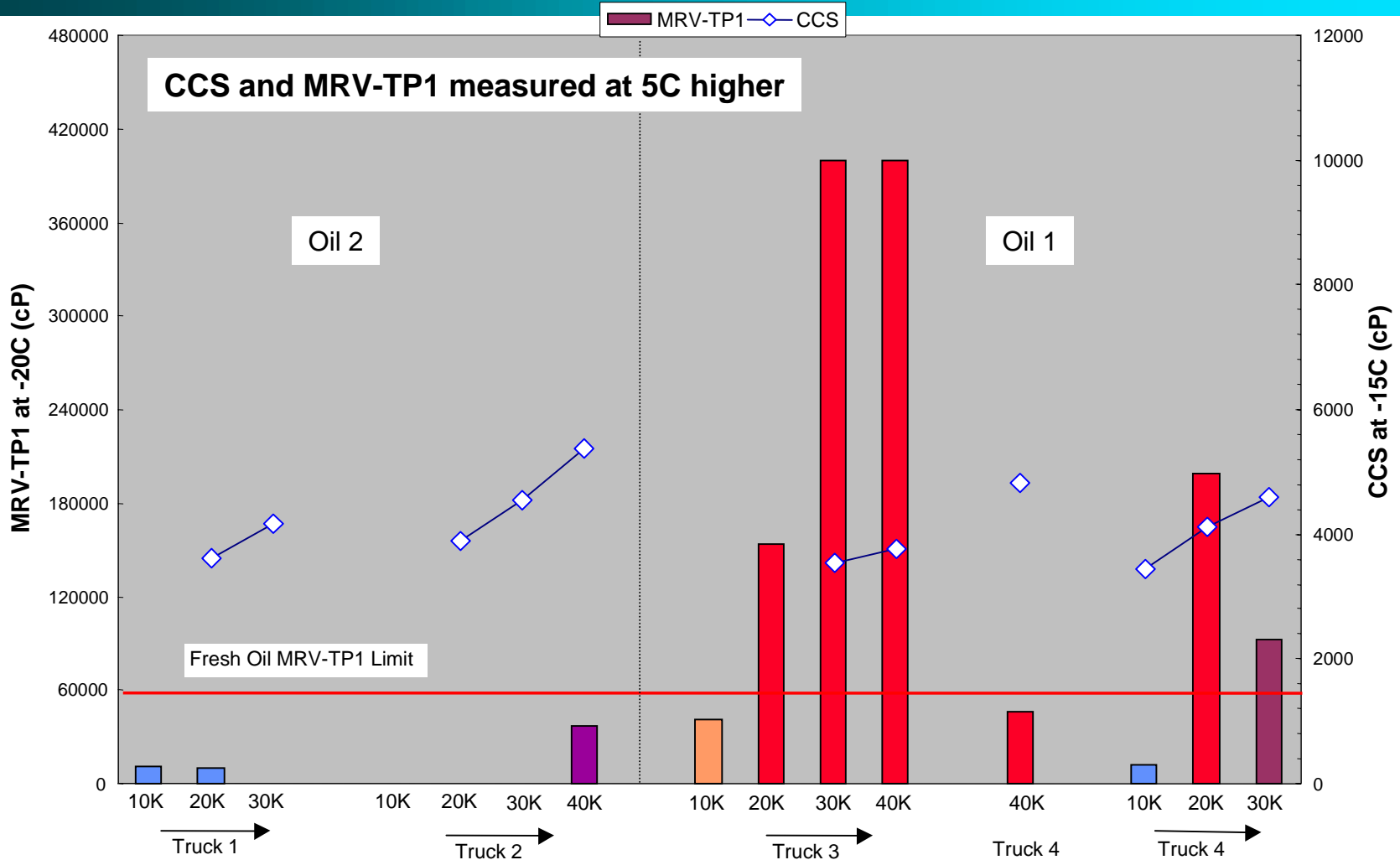
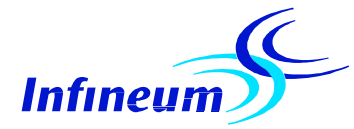
CCS and MRV-TP1 (@Standard Temperature)



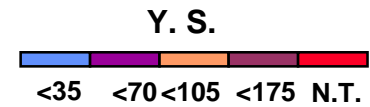
- Both oils show similar CCS increases in service
- Oil 1 significantly worse in MRV performance



CCS and MRV-TP1 (@5C Higher)



- Oil 1 continues to be poor in MRV performance even at 5C higher temp



2. Used Oils from Modified Mack T8E Tests

- ❖ Two commercial oils were sooted to ~10% soot in modified Mack T8E tests
- ❖ KV100 and MRV-TP1 were measured on end-of-test oils

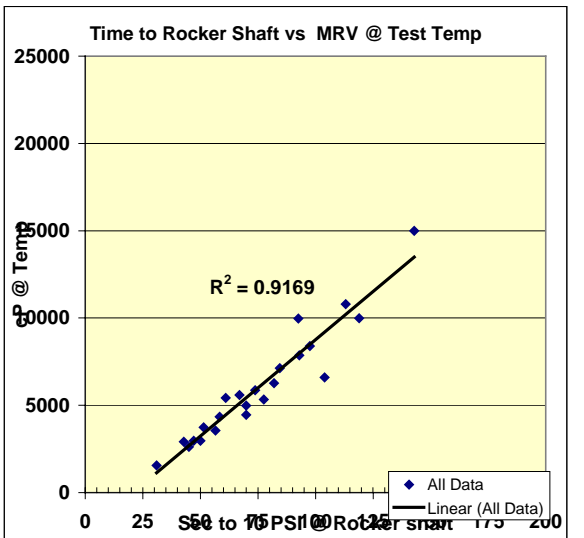
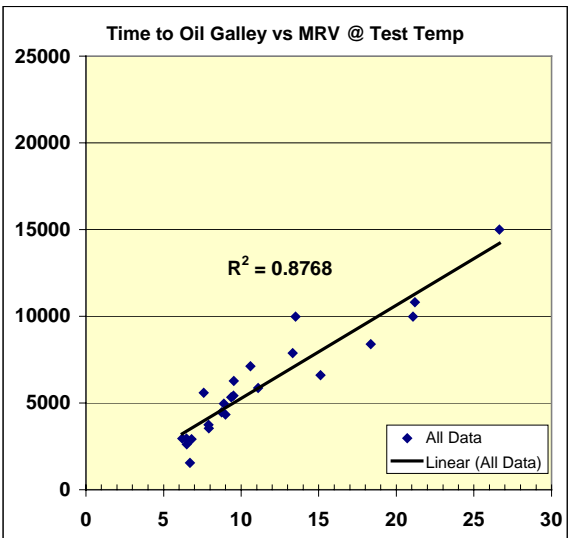
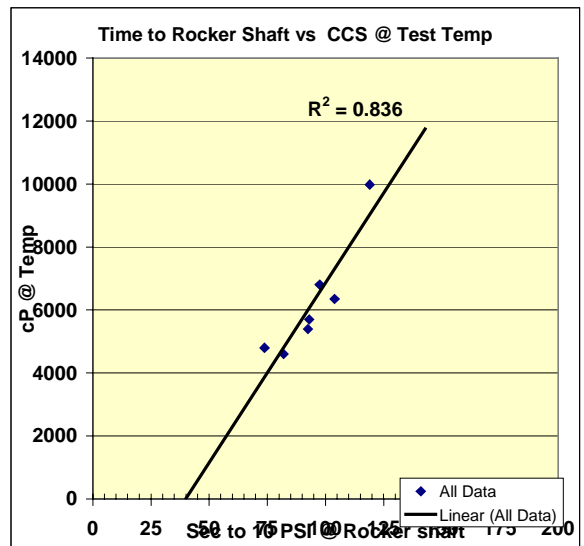
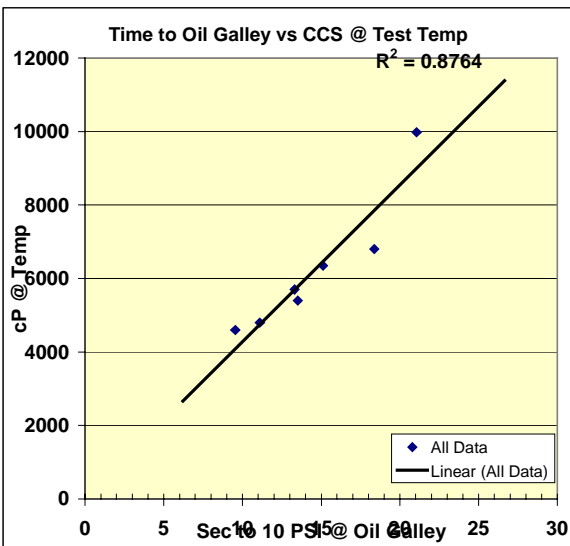
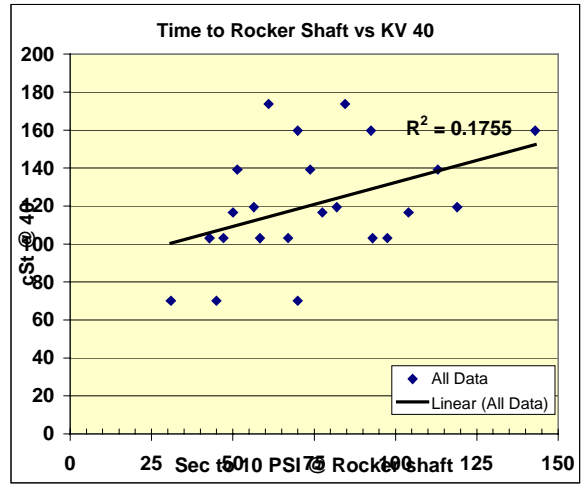
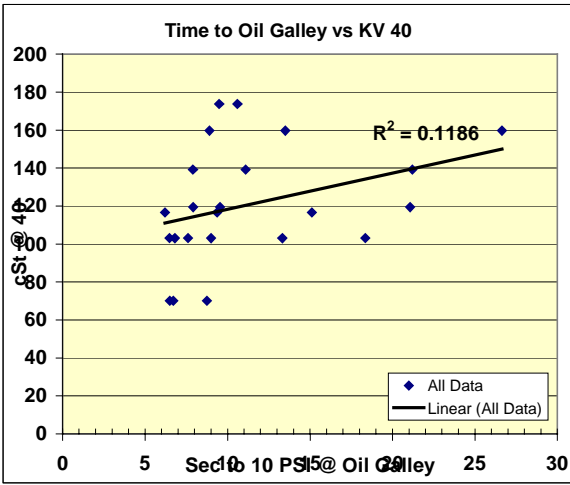
	Oil A	Oil B
End-of-Test Soot, %	10.5	9.2
KV100 Increase, cSt	46.34	81.90
Relative Viscosity @4.8% Soot	1.34	1.39
Slope of Relative Vis @5.8% Soot	0.38	0.40
Used oil MRV-TP1 @ -25C, cP	102225/<140 YS	64050 / <70 YS

- ❖ MRV-TP1 performance of used oil is not predicted by KV100 increase, relative viscosity or viscosity slope
 - If anything, an inverse correlation is evident

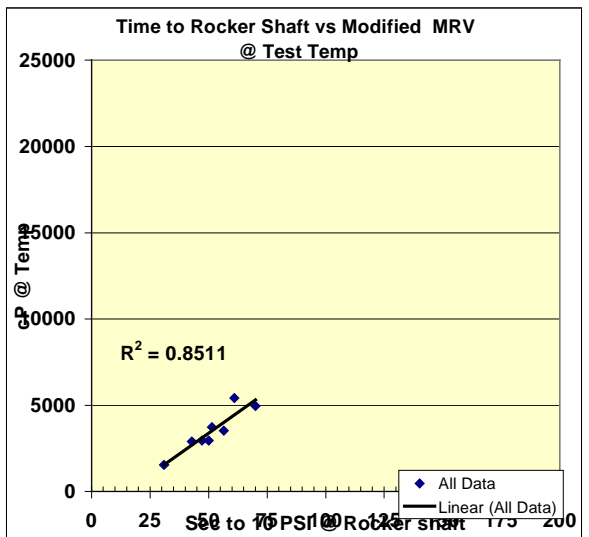
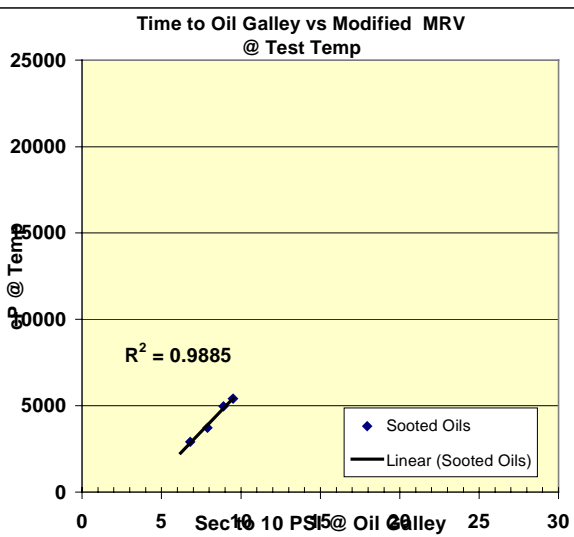
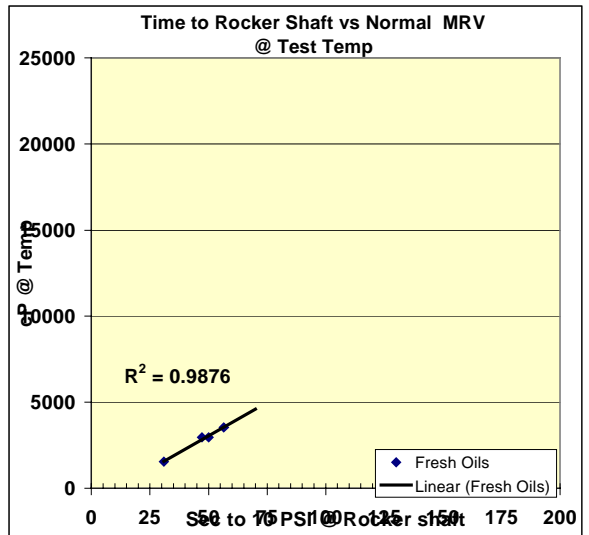
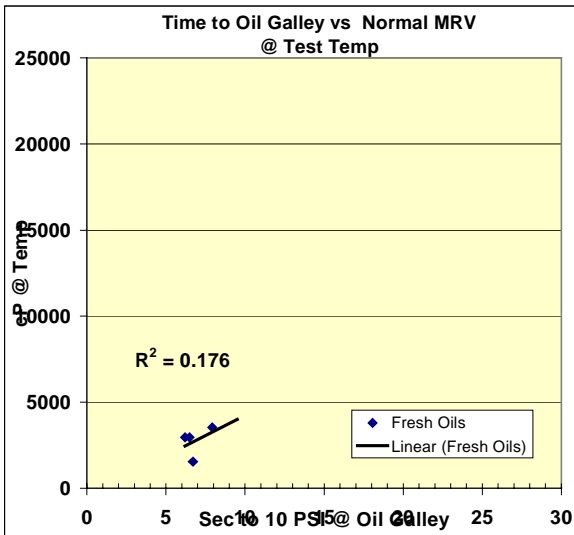
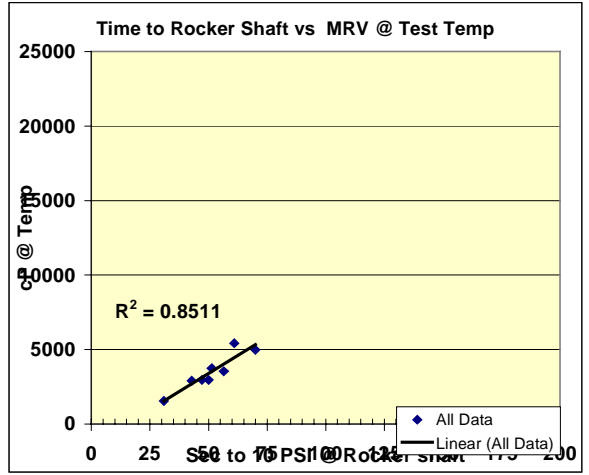
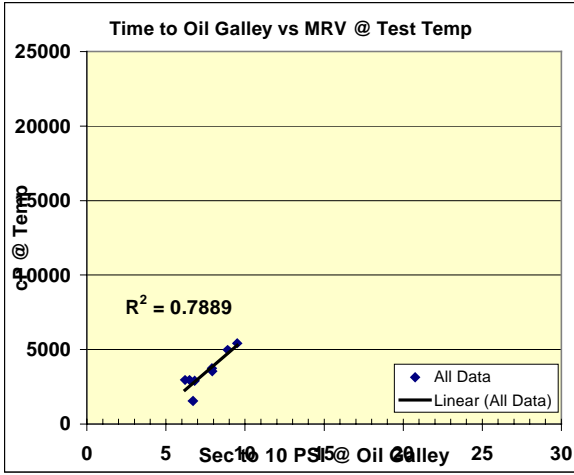
- ❖ **Certain oil exhibit potential for low temperature pumpability problems in the field**
 - **Some technologies may experience problems at soot levels as low as 2%**

- ❖ **Used oil MRV performance is NOT predicted by**
 - **Fresh oil MRV**
 - **KV100 increases or other common rheological measurements**

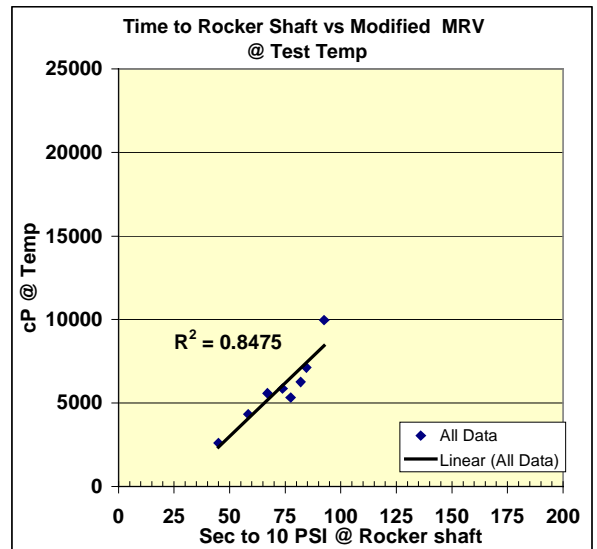
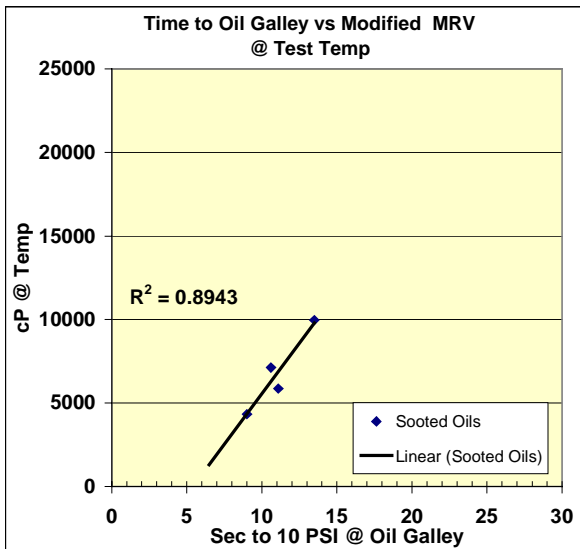
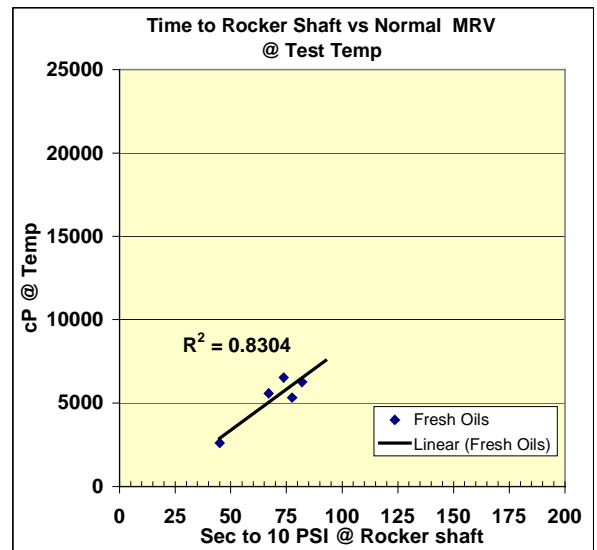
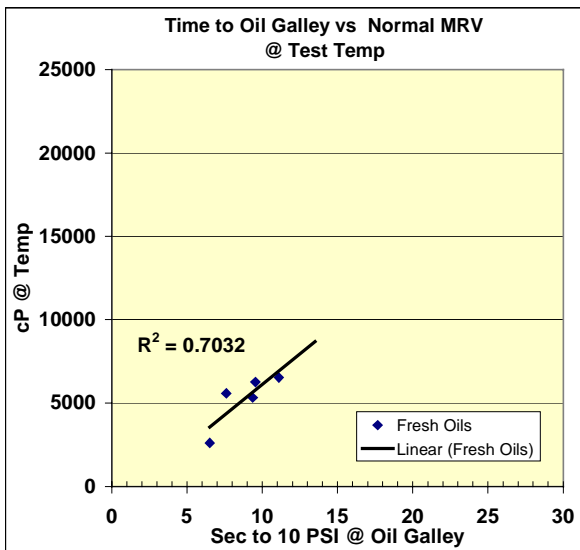
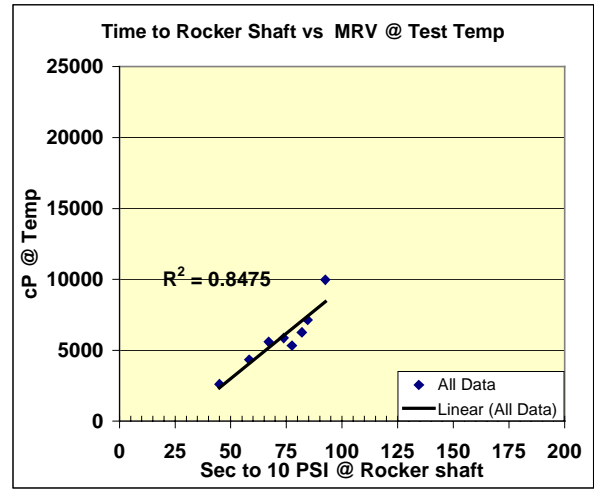
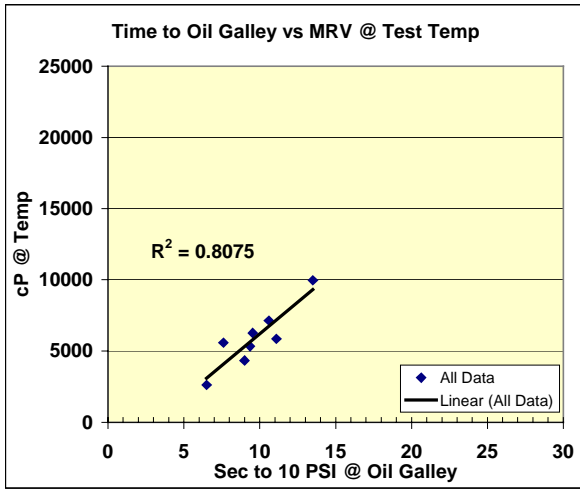
Data for All Oils, All Temps.



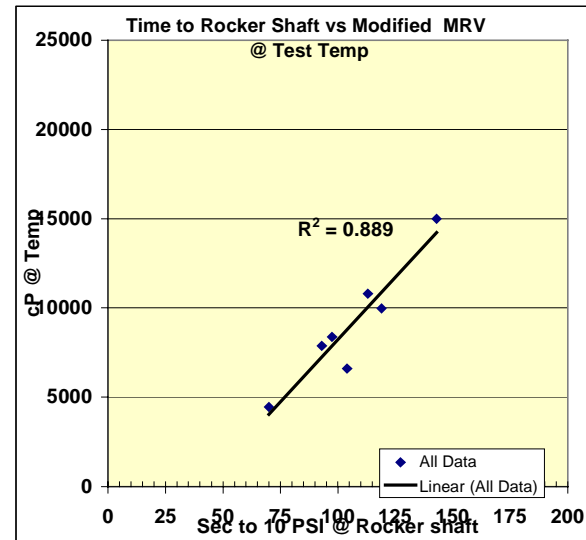
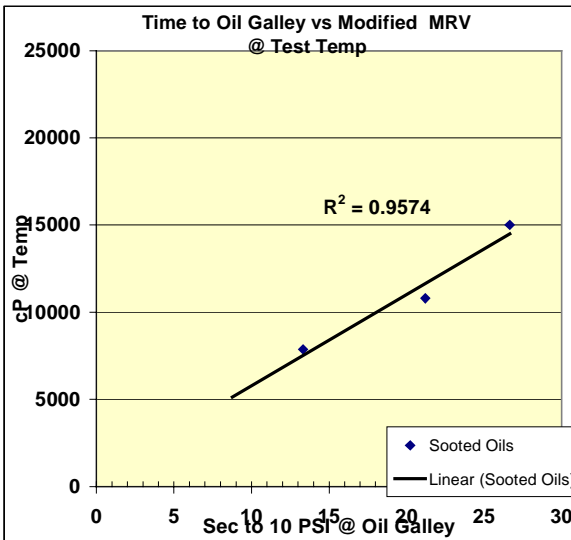
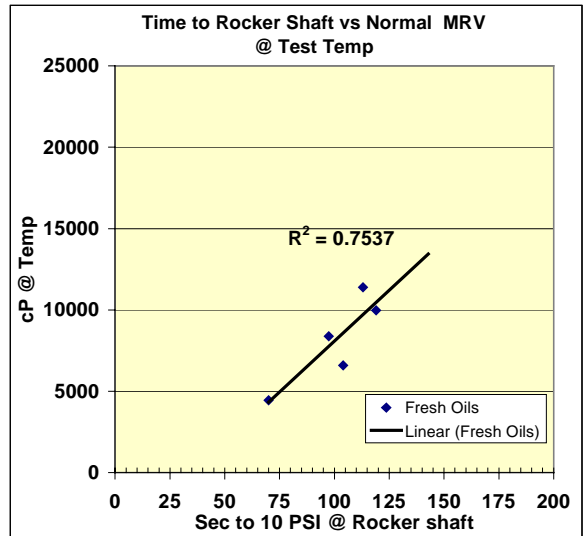
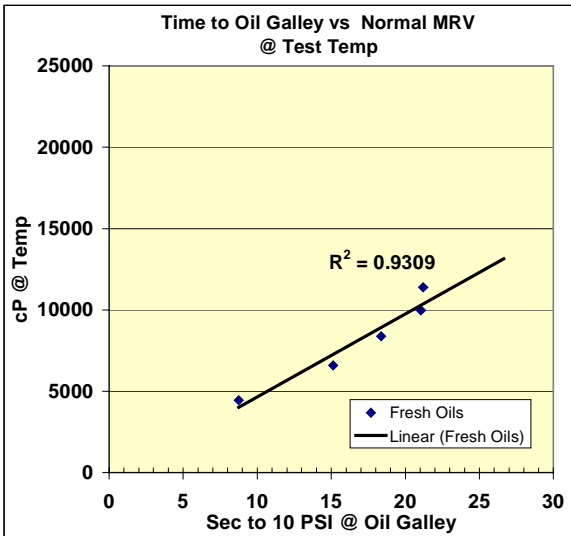
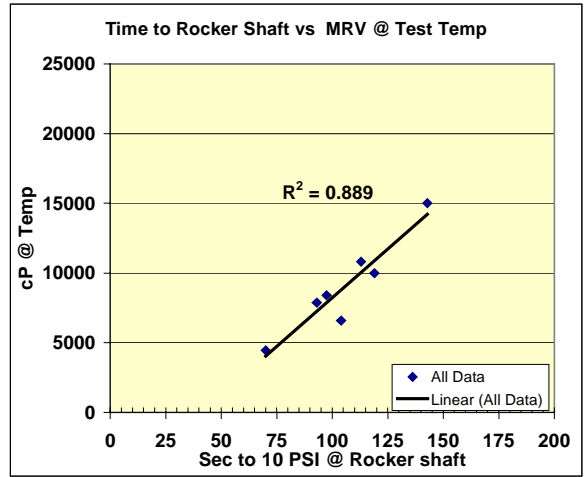
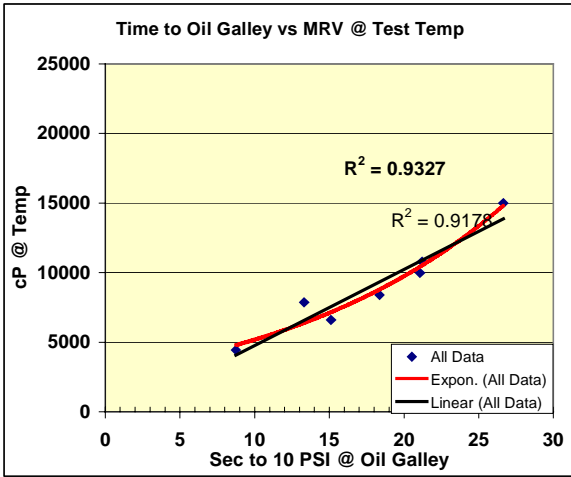
Data at -10 C



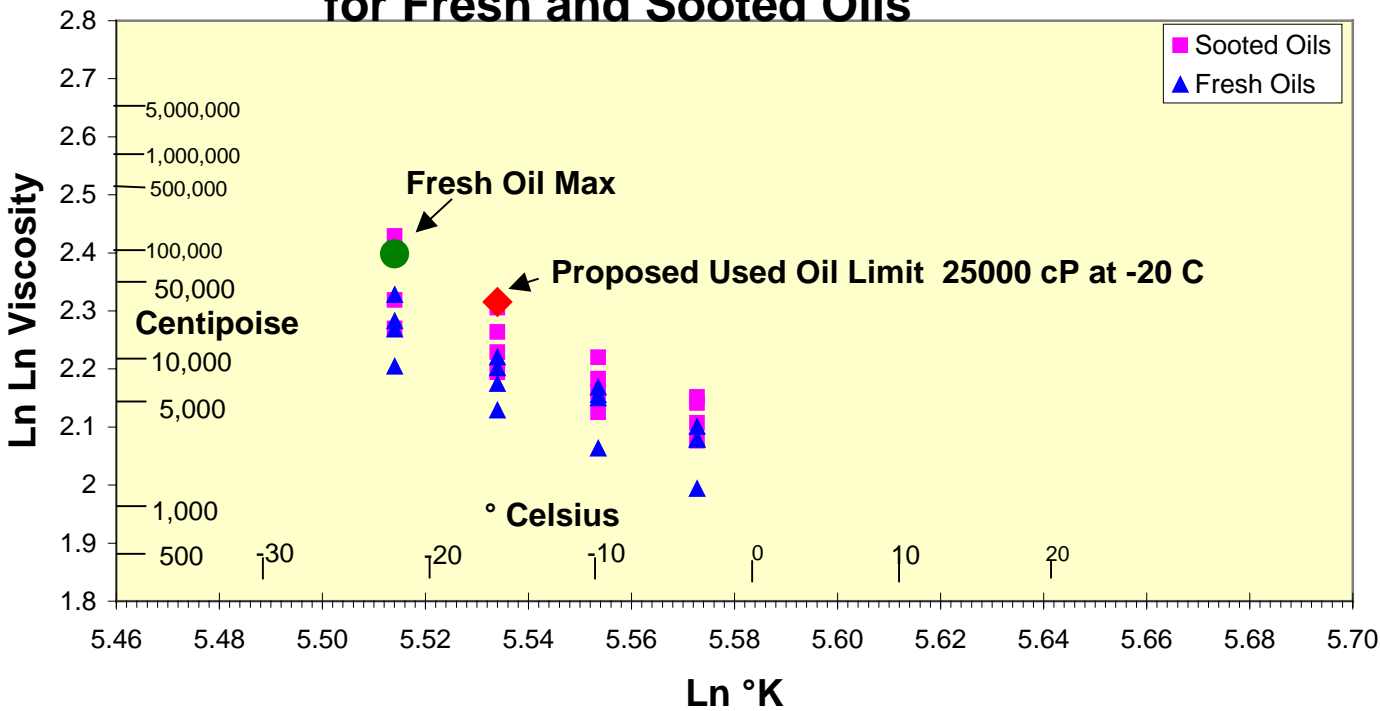
Data at -15 C



Data at -20 C



Viscosity Temperature Data for Fresh and Sooted Oils



PC-9 Elastomer TF Report to ASTM D02.B0 HDEOCP
July 11, 2001

- TF Meeting held in Columbus IN July 10 – Reached the following Conclusions and recommendations
 - A different statistical method was proposed to the TF for determining if a candidate oil is no worse than a reference oil. This method needs some further refining and will be w-mailed to the TF after refinement for further evaluation
 - The TF concluded that no one, two, or even 3 reference oils will adequately measure all critical parameters.
 - Some parameters have such small changes that comparison to a reference oil is inappropriate.
 - A reference oil will only protect on one side of a parameter
 - No one or two oils are the worst performers for all elastomer types all parameters
 - TMC 1006 (SF 105) comes closest or n ideal reference oil –it is the most aggressive oil in 3 elastomer types
 - Based on the notes above the Task Force Recommendations are given in the accompanying table
 - This method only works if we have a referee body for evaluating the significance of results outside the specified limit range which may be caused by changes in elastomer batches or other causes – And be accomplished rapidly – OEM representatives at the meeting believe the EMA can fulfill this role
- Test procedure will be balloted for standardization by D11.15
- A standing panel should be formed under D02.B0 Bench Test Surveillance Panel

Tom Boschert
PC-9 Elastomer TF leader

Summary of the PC-9 Elastomer Test Method

Four (4) elastomer types are immersed in the candidate oil and reference oil simultaneously using the same bath for 336 hours (14days).

There are 6 specimens for each elastomer type that are aged in each oil. Thus results from the candidate test for each elastomer are an average of 6 specimens and allows for the use of statistics. The reference oil that is being run simultaneously also is an average of 6 specimens.

The 4 elastomer types and the temperatures at which the specimens are aged at are:

Nitrile 100C

Fluoroelastomer 150C

Silicone 150C

Polyacrylate 150 C

The aged elastomers are measured for

% Volume change

Points hardness change

% Tensile Strength change

% Elongation change

The elastomers are from controlled batches distributed by a CPD

The reference oil is also controlled and distributed by the TMC

Summary

	Nitrile.....		SiliconePolyacrylate.....		FKM.....		
	OIL	Average Change	Standard Deviation	Original Change	Average Change	Standard Deviation	Original Change	Average Change	Standard Deviation	Original Change	Average Change	Standard Deviation	Original Change
Volume Change %	9G	0.2	0.1	-1.4			16.7	-2.0	0.2	-2.2			0.6
	J	-1.0	0.1	-0.7	26.3	0.1	8.3			-0.4	0.2	0.1	0.2
	P			0.8			18.8	-0.3	0.8	-1.0			0.0
	1006	-1.1	0.7	2.5	27.4	0.9	25.7	0.6	0.0	3.7	0.3	0.1	0.6
Points Hardness Change	9G	3.2	2.6	5.0			-13.0	2.9	1.3	4.0			2.0
	J	3.7	3.0	10.0	-19.1	0.7	-9.0			4.0	4.9	2.0	8.0
	P			0.0			-14.0	2.9	0.7	7.0			4.0
	1006	7.2	0.0	3.0	-18.1	2.3	-22.0	-1.7	1.4	-1.0	7.2	0.0	6.0
Tensile Strength % Change	9G	-0.9	3.4	-22.2			-15.9	4.5	4.5	3.4			-41.4
	J	-50.4	5.7	-61.1	4.5	3.8	-59.3			2.3	-55.1	18.6	-67.6
	P			-1.7			-11.1	-9.8	5.3	-13.4			-31.8
	1006	-33.8	2.3	-37.4	-2.2	2.9	-24.0	0.3	5.9	-2.5	-65.3	0.2	-68.8
Elongation Change %	9G	-28.9	6.5	-42.1			-14.4	-15.2	11.3	-15.6			-40.0
	J	-59.7	1.7	-63.6	-5.0	11.6	-22.7			-15.3	-53.7	16.5	-57.6
	P			-13.7			-11.1	-31.0	10.3	-42.7			-42.3
	1006	-56.9	2.3	-49.7	-14.0	9.9	-24.0	-10.5	4.3	-15.2	-54.2	1.1	-62.0

Elastomer Data

ASTM Elastomer Task Group

Summary of Industry Data Including PC-9 Matrix Oils

D M Stehouwer
Cummins Fuels & Lubes Group

Revised 07-16-01

Tom Boschert

Elastomer Test Limits

- ◆ **ASTM is close to selection of reference fluids**
- ◆ **Matrix oils A B C E G & H have been added to these graphs**
- ◆ **This data set has been sorted a new way, to group the oils in 3 categories**
 - **Commercial**
 - » **Includes CH4, CD / SH, ACEA**
 - » **Group I, II, and IV base oils**
 - **Candidate PC-9**
 - » **Includes candidates and 6 Matrix oils**
 - **Reference fluids**
 - » **Includes TMC reference oils and variations of TMC 1006**
 - » **Oils L, I, J, K are variations of TMC 1006 in Group II stocks**

Elastomer Test Limits

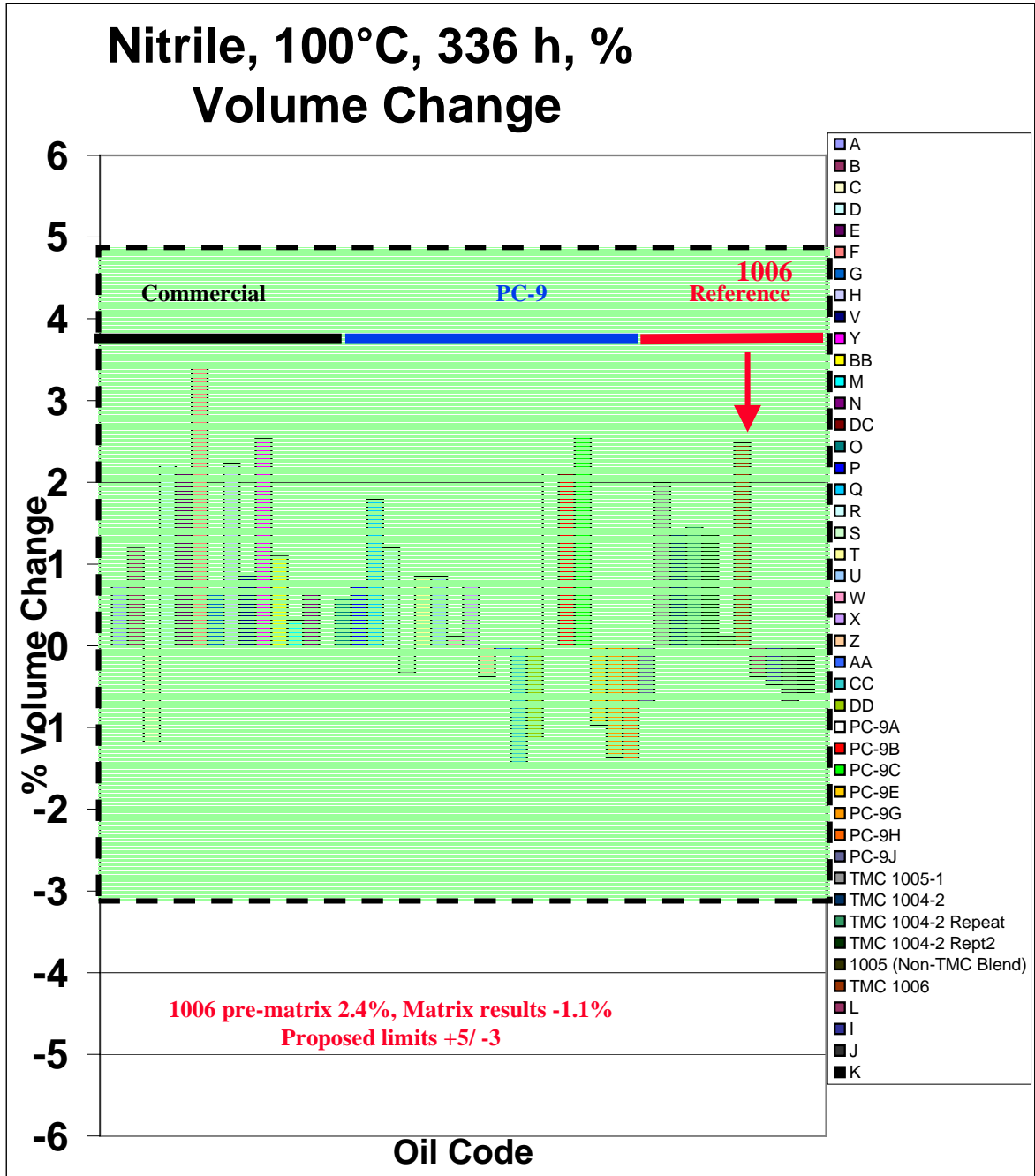
- ◆ **TMC 1006 (Service fluid 105) is proposed as reference for Nitrile, Silicone, & Fluorelastomer. Results of pre-matrix testing of TMC 1006 and the matrix (or round robin) tests of TMC 1006 are noted on each graph**
- ◆ **Reference oil has not been set for Polyacrylate.**
- ◆ **Proposed Elastomer test limits are written in red on each graph. Fixed limits are noted graphically by a dotted line.**

Nitrile

Nitrile, Elastomer Batch A21-35-2, 100°C, 336 Hours, All Data an Average of Three Samples

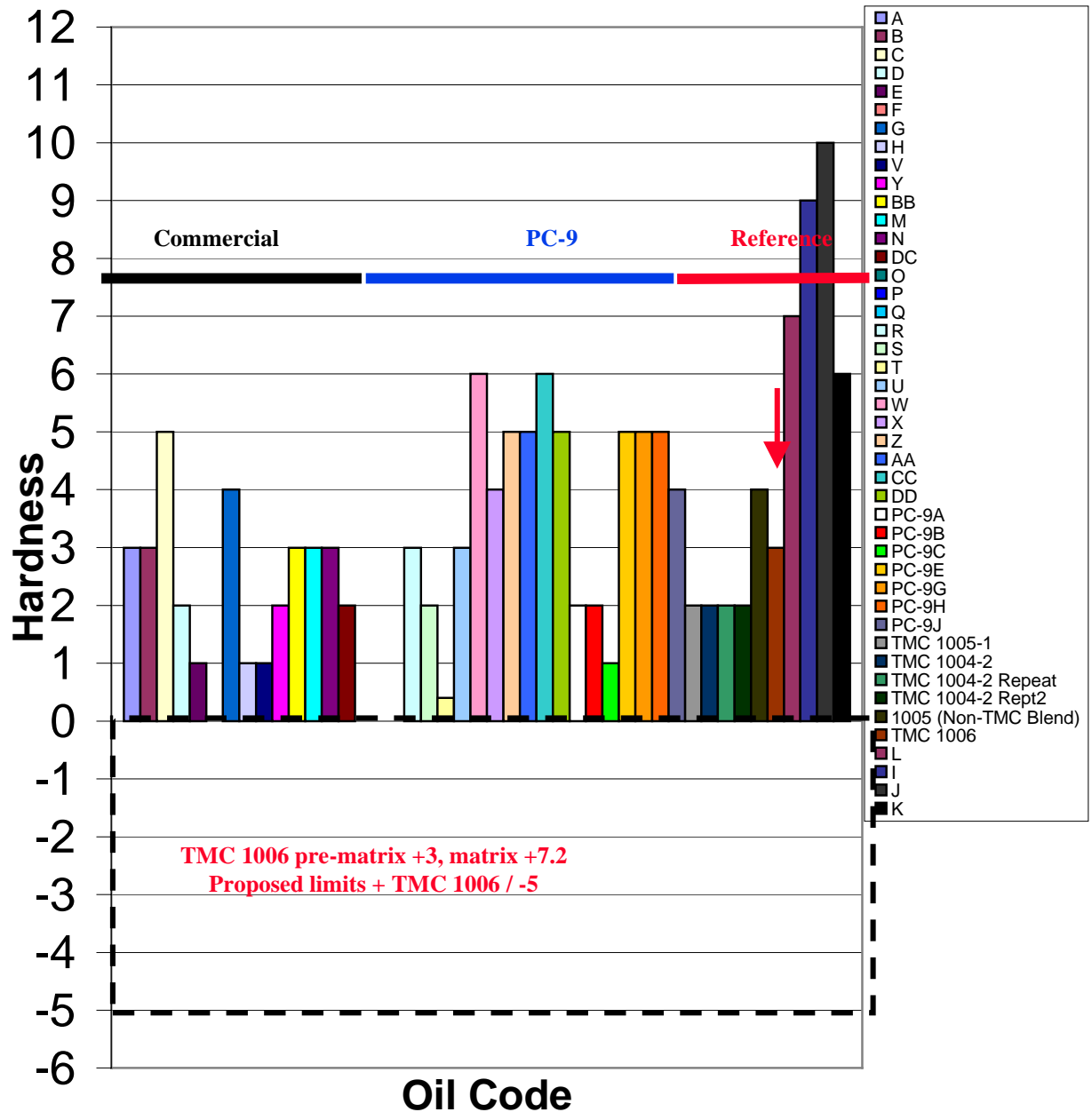
	Volume Change, %	Points Hardness Change	Tensile Strength Change, %	Elongation Change, %	Reversion	Oil Type
A	0.8	3	-7.6	-32.2	None	Commercial
B	1.2	3	-5.1	-34.0	None	Commercial
C	-1.2	5	-42.9	-53.2	None	Commercial
D	2.2	2	-4.7	-33.5	None	Commercial
E	2.1	1	-6.4	-39.2	None	Commercial
F	3.4	0	-0.2	-36.0	None	Commercial
G	0.7	4	-2.5	-33.2	None	Commercial
H	2.2	1	-16.6	-43.7	None	Commercial
V	0.9	1	-22.3	-29.6		Commercial
Y	2.5	2	-2.1	-22.7		Commercial
BB	1.1	3	2.2	-24.2		Commercial
M	0.3	3	1.5	-27.7	None	Commercial ACEA
N	0.7	3	4.6	-29.9	None	Commercial ACEA
DC	0.0	2	-4.0	-28.0		Commercial ACEB
O	0.6	0	4.4	-12.5	None	PC-9
P	0.8	0	-1.7	-13.7	None	PC-9
Q	1.8	0	-26.3	-31.5	None	PC-9
R	1.2	3	-5.8	-22.3	None	PC-9
S	-0.3	2	-5.8	-27.5	None	PC-9
T	0.9	0.4	-13.4	-35.9		PC-9
U	0.9	3	-18.2	-39.6		PC-9
W	0.1	6	-9.2	-34.7		PC-9
X	0.8	4	2.3	-29.8		PC-9
Z	-0.4	5	-5.1	-26.6		PC-9
AA	-0.1	5	-4.5	-31.9		PC-9
CC	-1.5	6	-4.0	-28.3		PC-9
DD	-1.1	5	0.6	-31.3		PC-9
PC-9A	2.16	2	-1.1	-25.8		PC-9
PC-9B	2.11	2	-12.3	-30.6		PC-9
PC-9C	2.56	1	1.8	-23.1		PC-9
PC-9E	-1.0	5	2.5	-27.4		PC-9
PC-9G	-1.4	5	-22.2	-42.1		PC-9
PC-9H	-1.4	5	-0.5	-28.6		PC-9
PC-9J	-0.7	4	0.7	-25.5		PC-9
TMC 1005-1	2.0	2	-1.6	-31.5	None	Reference
TMC 1004-2	1.4	2	-0.8	-31.5	None	Reference
TMC 1004-2 Repeat	1.5	2	-3.9	-38.5	None	Reference
TMC 1004-2 Rept2	1.4	2	8.8	-24.5	None	Reference
1005 (Non-TMC Blend)	0.1	4	-0.9	-29.4		Reference
TMC 1006	2.5	3	-37.4	-49.7	None	Reference
L	-0.4	7	-49.6	-67.2	None	Reference
I	-0.5	9	-59.4	-62.4	None	Reference
J	-0.7	10	-61.1	-63.6	None	Reference
K	-0.6	6	-47.1	-57.2	None	Reference

Nitriles



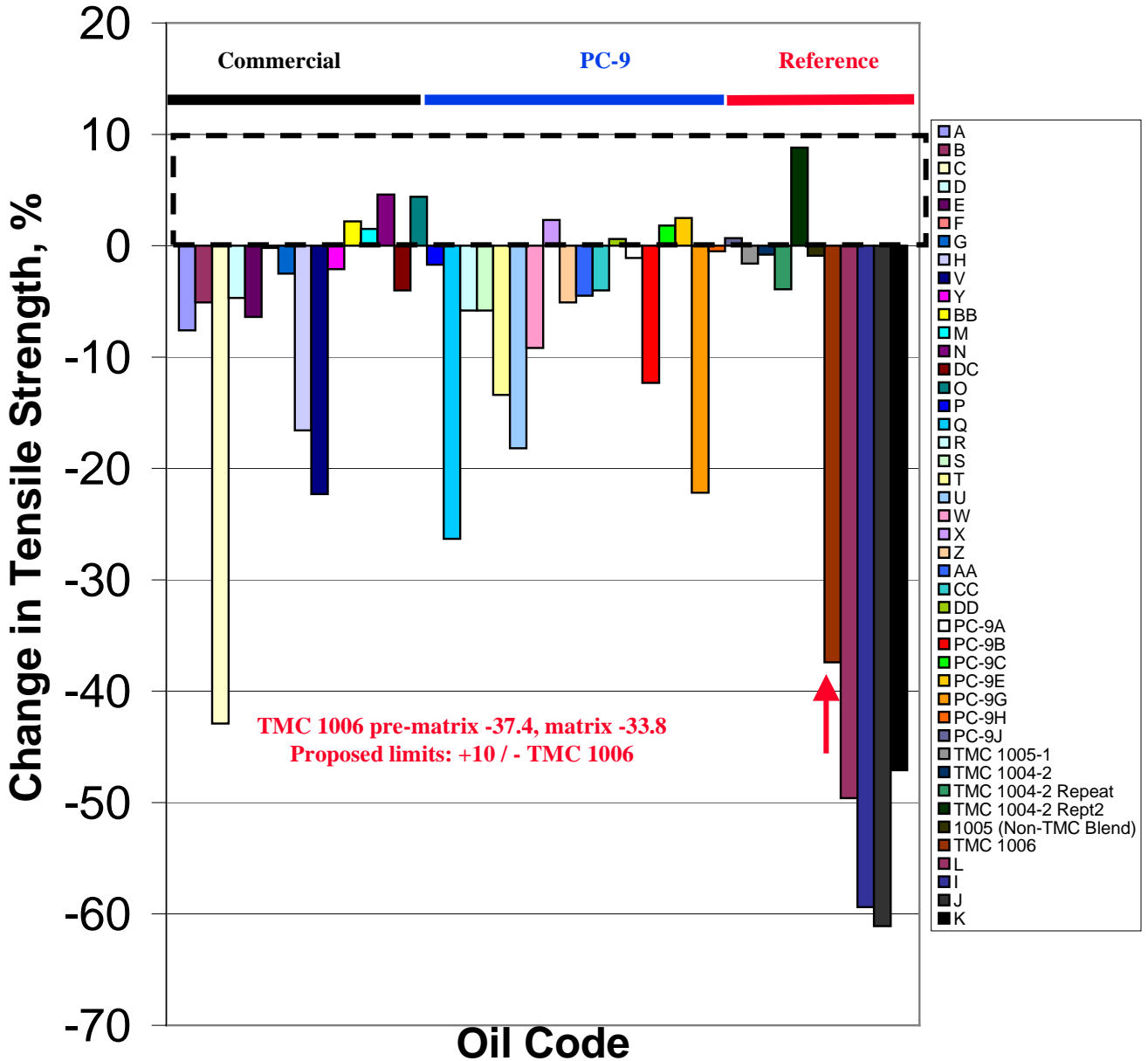
Nitriles

Nitrile, 100°C, 336 h, Hardness



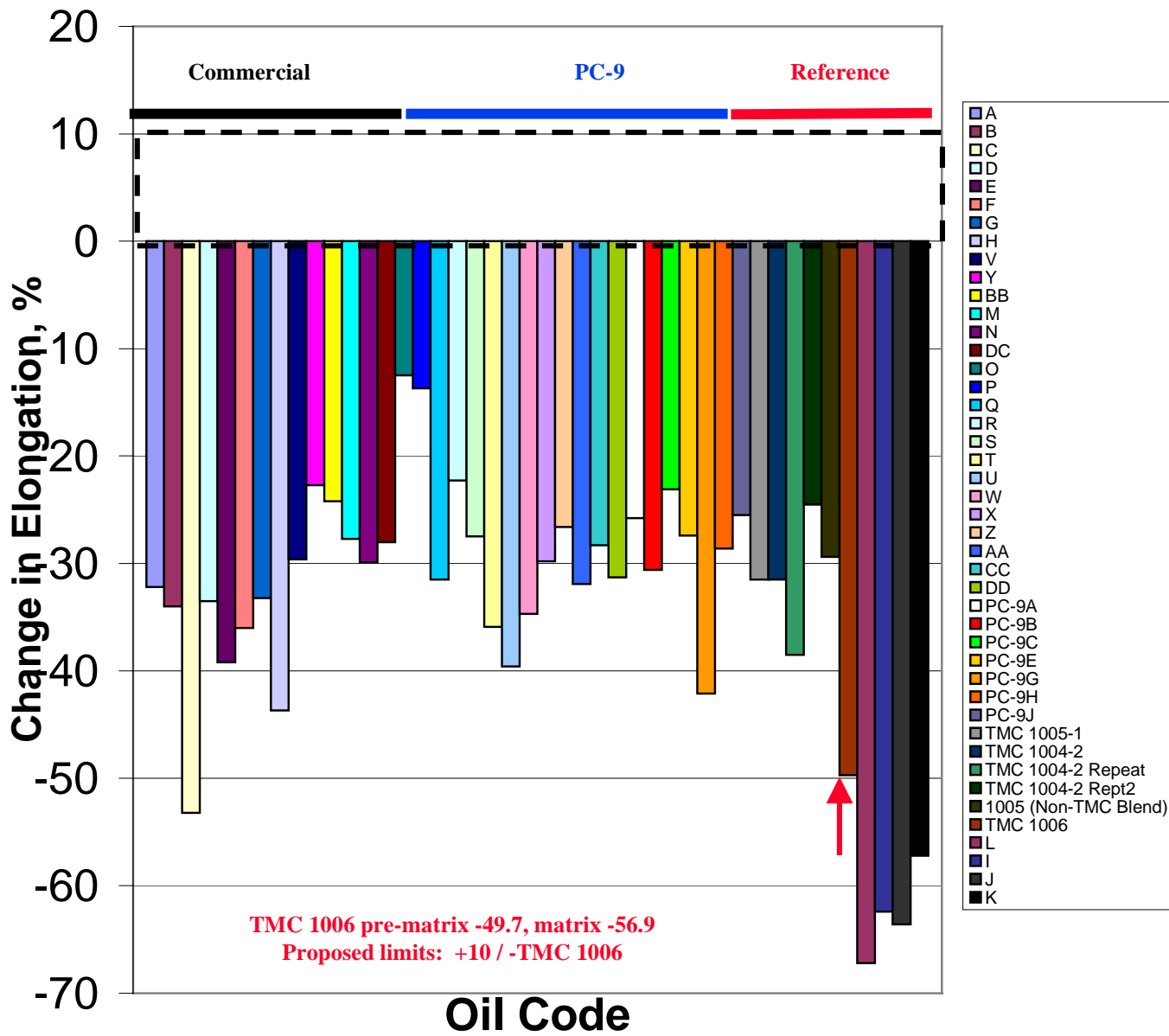
Nitriles

Nitrile, 100°C, 336 h, Change in Tensile Strength



Nitriles

**Nitrile, 100°C, 336 h,
Change in Elongation**

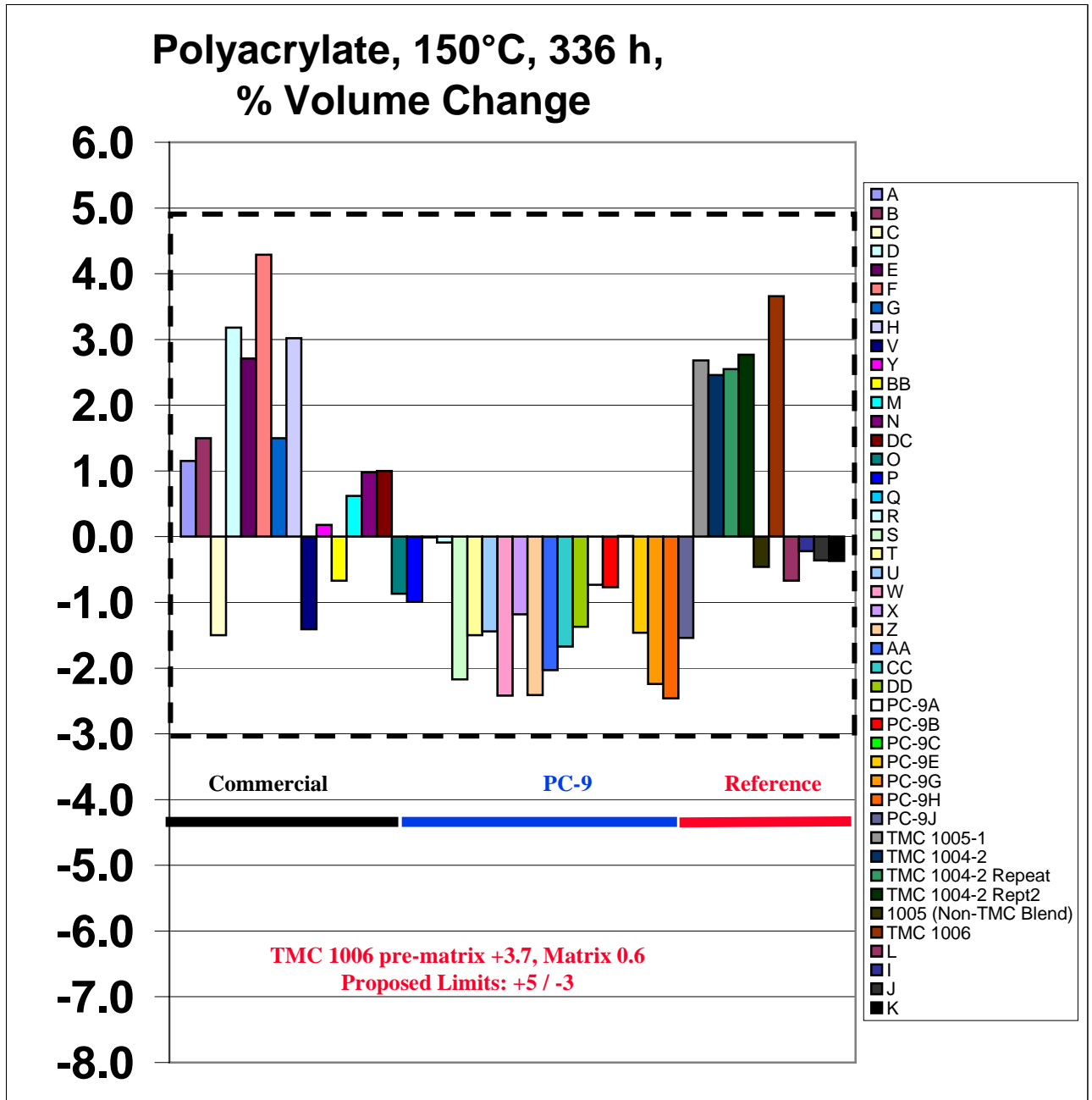


Polyacrylate

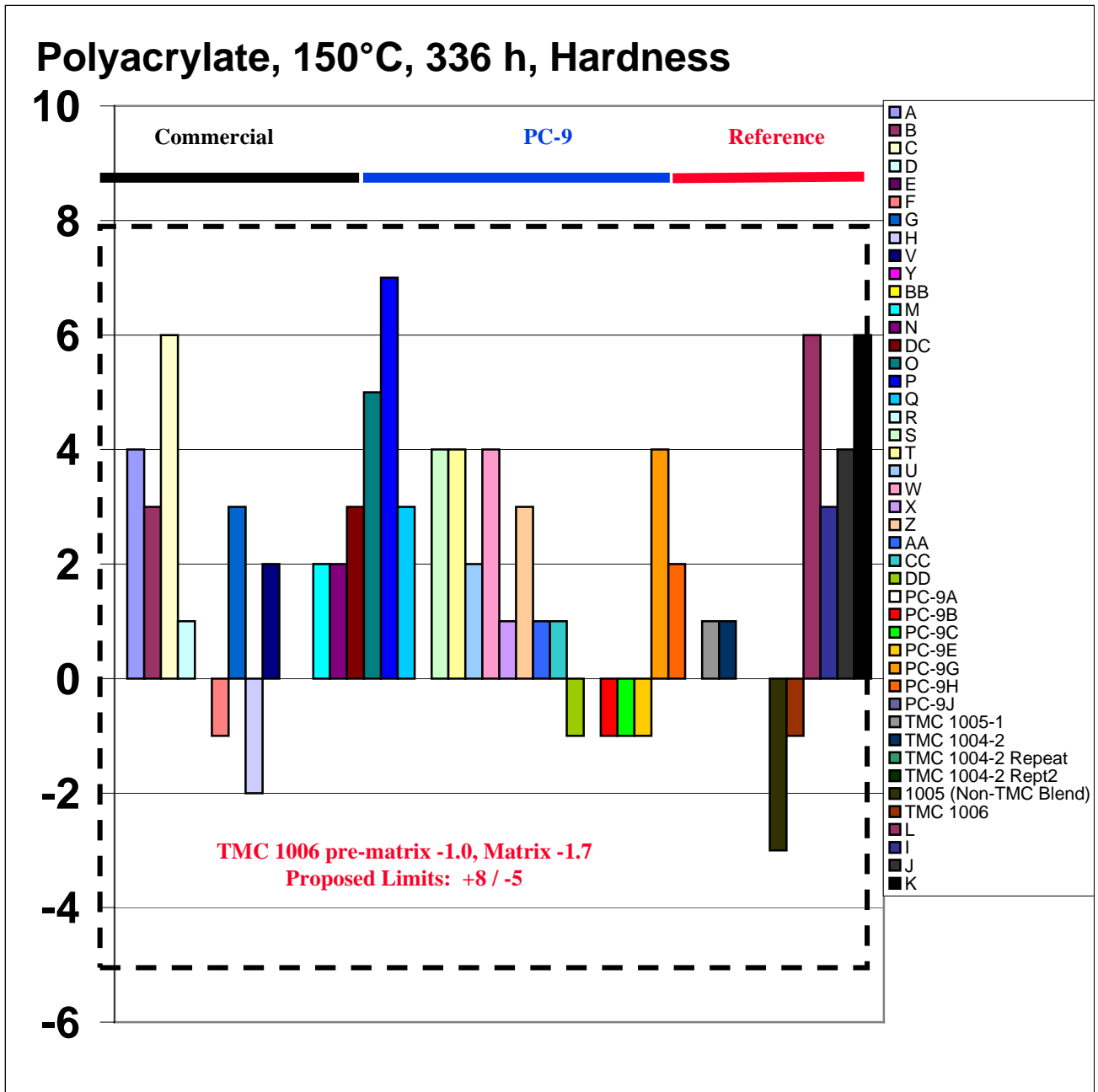
Polyacrylate, Elastomer Batch A21-35-1, 150°C, 336 Hours, All Data an Average of Three Samples

	Volume Change, %	Points Hardness Change	Tensile Strength Change, %	Elongation Change, %	Reversion	Oil Type
A	1.2	4	3.7	-19.6	None	Commercial
B	1.5	3	0.7	-17.3	None	Commercial
C	-1.5	6	-6.0	-25.3	None	Commercial
D	3.2	1	6.8	-14.2	None	Commercial
E	2.7	0	2.7	-17.8	None	Commercial
F	4.3	-1	-3.7	-26.0	None	Commercial
G	1.5	3	12.5	-20.3	None	Commercial
H	3.0	-2	8.1	-20.1	None	Commercial
V	-1.4	2	0.2	-22.0		Commercial
Y	0.2	0	-4.2	-14.8		Commercial
BB	-0.7	0	5.1	-8.1		Commercial
M	0.6	2	8.9	-12.0	Yes	Commercial ACEA
N	1.0	2	2.0	-0.7	Yes	Commercial ACEA
DC	1.0	3	1.0	-22.0		Commercial ACEB
O	-0.9	5	-14.6	-40.4		PC-9
P	-1.0	7	-13.4	-42.7		PC-9
Q	0.0	3	-14.7	-30.9		PC-9
R	-0.1	0	3.9	-4.1		PC-9
S	-2.2	4	1.4	-17.8		PC-9
T	-1.5	4	0.6	-29.2		PC-9
U	-1.4	2	3.7	-22.9		PC-9
W	-2.4	4	-4.0	-21.5		PC-9
X	-1.2	1	-3.6	-22.2		PC-9
Z	-2.4	3	3.9	-14.5		PC-9
AA	-2.0	1	7.7	-8.1		PC-9
CC	-1.7	1	5.7	-14.4		PC-9
DD	-1.4	-1	-2.3	-21.8		PC-9
PC-9A	-0.73	0	-2.8	-14.6		PC-9
PC-9B	-0.77	-1	5.8	-7.4		PC-9
PC-9C	0.01	-1	3.9	-10.3		PC-9
PC-9E	-1.5	-1	3.2	-7.7		PC-9
PC-9G	-2.2	4	3.4	-15.6		PC-9
PC-9H	-2.5	2	3.6	-13.3		PC-9
PC-9J	-1.5	0	1.9	-16.8		PC-9
TMC 1005-1	2.7	1	2.7	-18.3	None	Reference
TMC 1004-2	2.5	1	-3.8	-24.3	None	Reference
TMC 1004-2 Repeat	2.6	0	-1.5	-24.2	None	Reference
TMC 1004-2 Rept2	2.8	0	2.6	-1.8	None	Reference
1005 (Non-TMC Blend)	-0.5	-3	3.2	-13.8		Reference
TMC 1006	3.7	-1	-2.5	-15.2	None	Reference a
L	-0.7	6	9.1	-28.2	None	Reference a1
I	-0.2	3	11.1	-13.5	None	Reference b
J	-0.4	4	2.3	-15.3	None	Reference b
K	-0.4	6	3.5	-14.8	None	Reference b

Polyacrylate

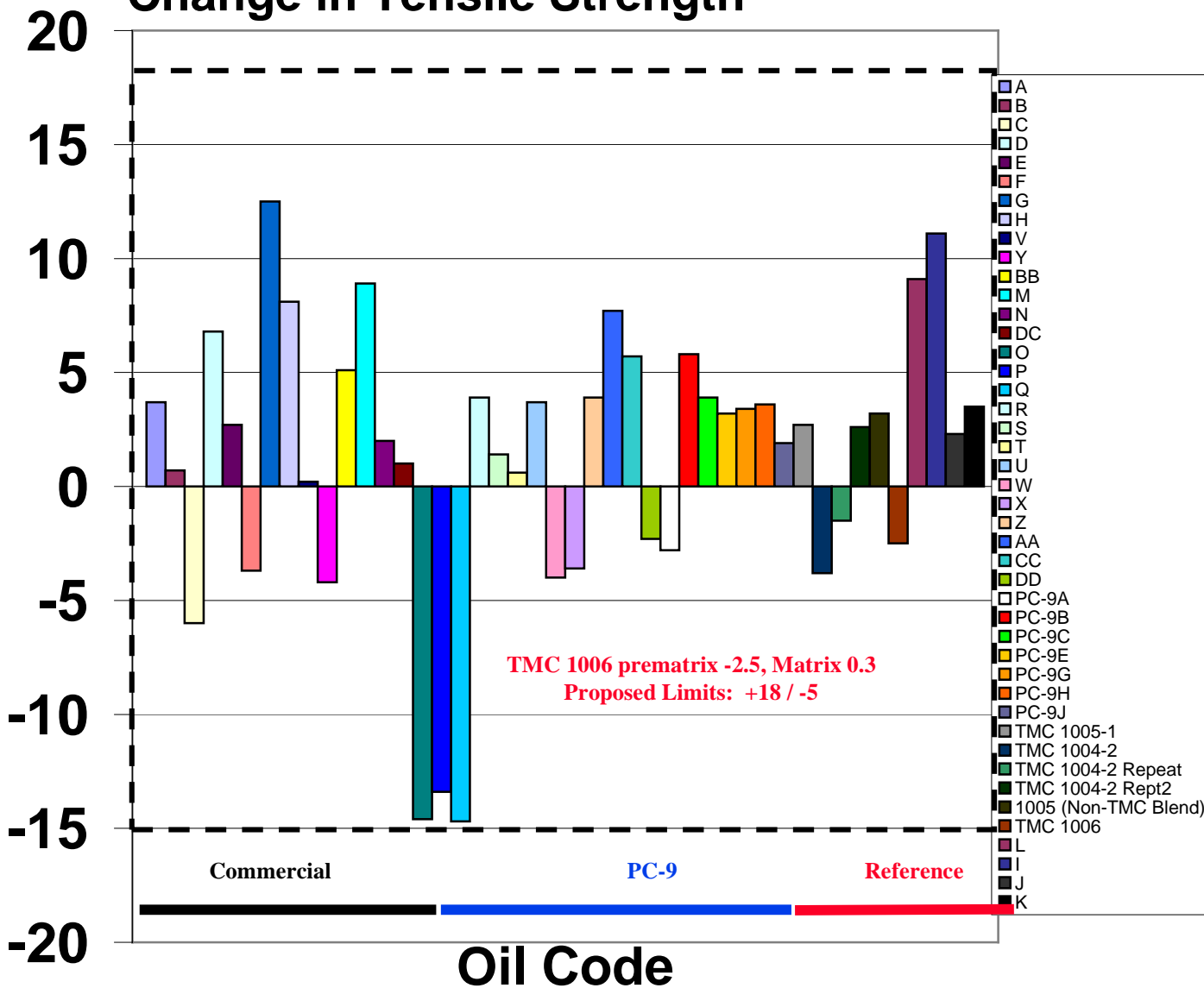


Polyacrylate



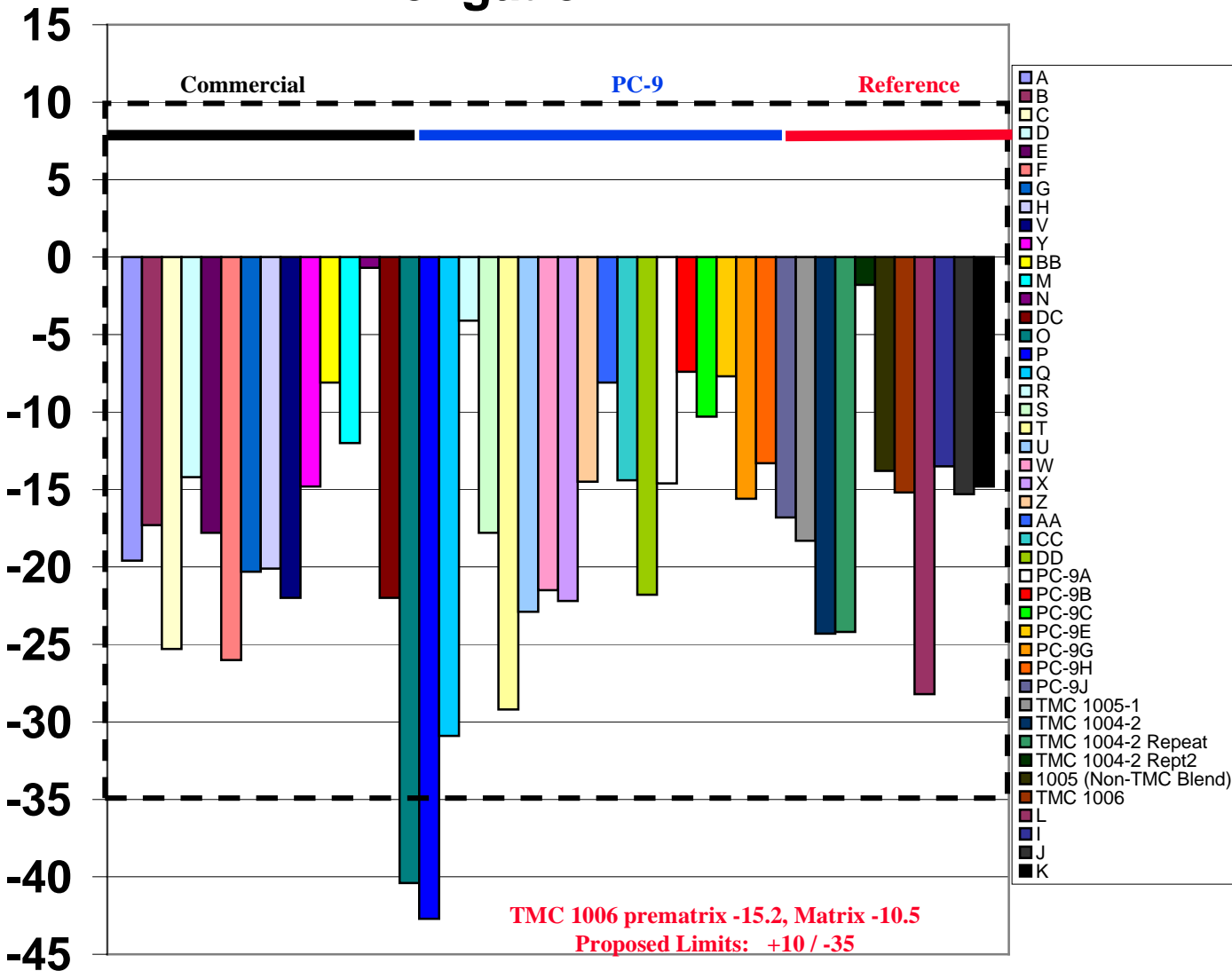
Polyacrylate

Polyacrylate, 150°C, 336 h,
Change in Tensile Strength



Polyacrylate

Polyacrylate, 150°C, 336 h, Change in Elongation

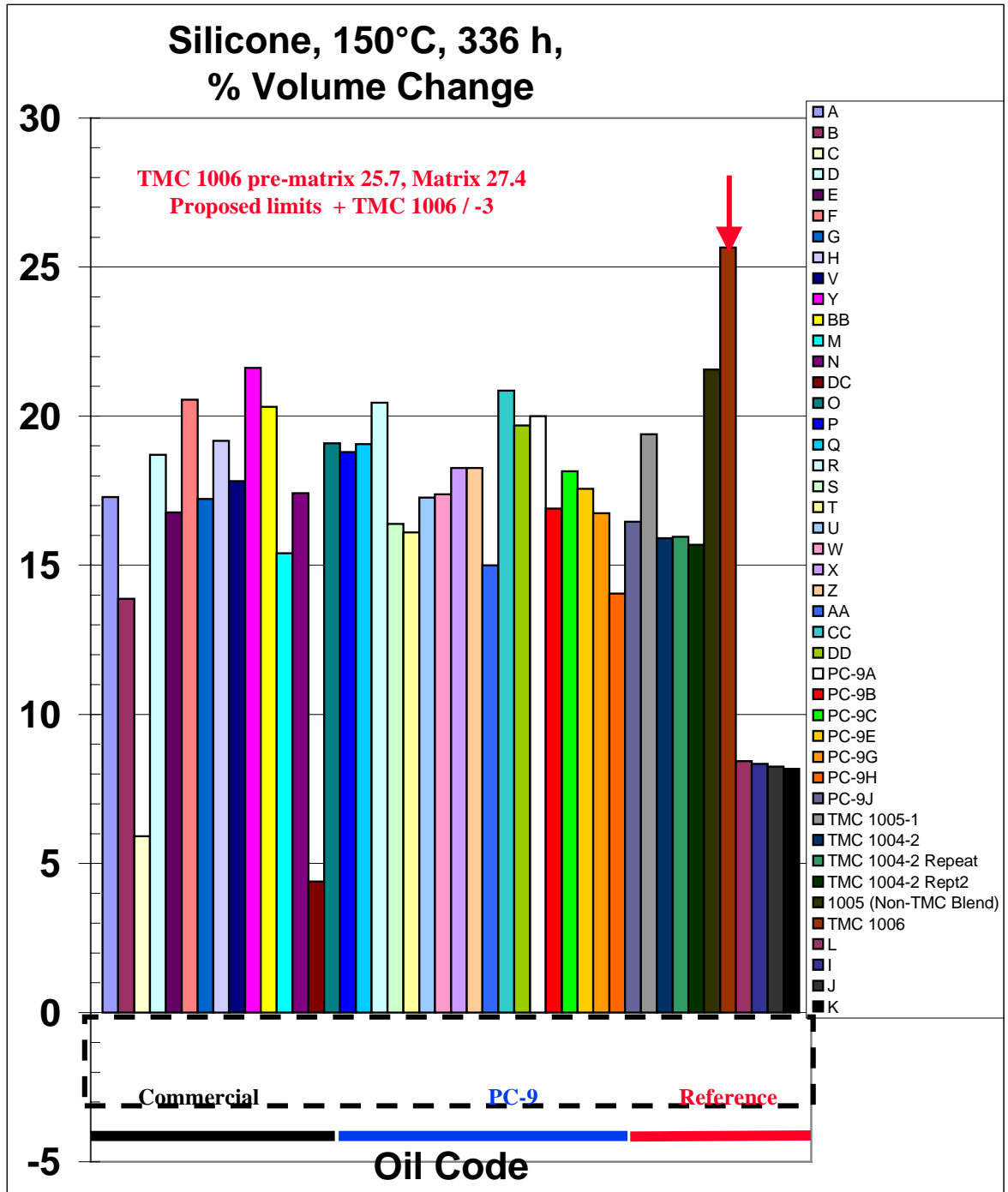


Silicone

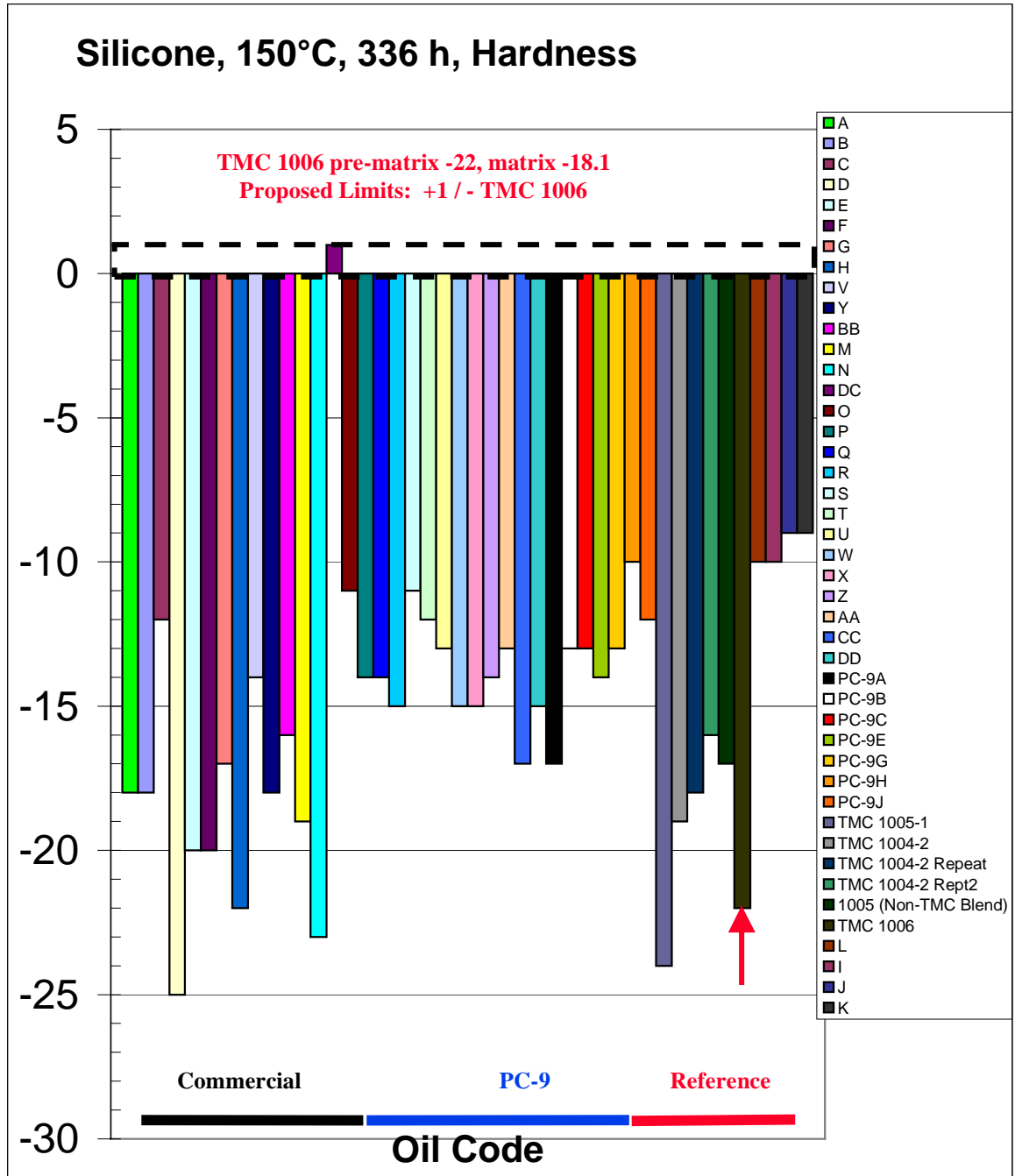
Silicone, Elastomer Batch 93-90-70, 150°C, 336 Hours, All Data an Average of Three Samples

	Volume Change, %	Points Hardness Change	Tensile Strength Change, %	Elongation Change, %	Reversion	Oil Type
A	17.3	-18	-31.4	0.5	None	Commercial
B	13.9	-18	-36.8	-8.4	None	Commercial
C	5.9	-12	-35.3	9.6	None	Commercial
D	18.7	-25	-43.7	25.6	None	Commercial
E	16.8	-20	-29.5	9.0	None	Commercial
F	20.6	-20	-18.8	2.3	None	Commercial
G	17.2	-17	-4.2	1.6	None	Commercial
H	19.2	-22	-36.7	11.0	None	Commercial
V	17.8	-14	-7.0	-30.7		Commercial
Y	21.6	-18	-24.4	-19.7		Commercial
BB	20.3	-16	-8.4	-9.3		Commercial
M	15.4	-19	-35.4	7.8	None	Commercial ACEA
N	17.4	-23	-44.6	9.2	None	Commercial ACEA
DC	4.4	1	-7.5	-31.8		Commercial ACEB
O	19.1	-11	-11.3	-8.6		PC-9
P	18.8	-14	-11.1	-14.0		PC-9
Q	19.1	-14	-5.8	-12.7		PC-9
R	20.5	-15	-11.2	-19.5		PC-9
S	16.4	-11	-4.7	-16.5		PC-9
T	16.1	-12	-8.0	-29.0		PC-9
U	17.3	-13	-2.3	-22.7		PC-9
W	17.4	-15	-2.3	-5.3		PC-9
X	18.3	-15	-6.9	-7.8		PC-9
Z	18.3	-14	-1.8	-6.1		PC-9
AA	15.0	-13	-4.6	-3.3		PC-9
CC	20.9	-17	-7.5	-7.5		PC-9
DD	19.7	-15	-3.9	-10.1		PC-9
PC-9A	20	-17	-17.5	-17		PC-9
PC-9B	16.9	-13	-10	-5.3		PC-9
PC-9C	18.15	-13	-10.5	-10.9		PC-9
PC-9E	17.6	-14	-1.8	-14.7		PC-9
PC-9G	16.7	-13	-15.9	-14.4		PC-9
PC-9H	14.1	-10	-3.4	-6.2		PC-9
PC-9J	16.5	-12	-6.3	-17.4		PC-9
TMC 1005-1	19.4	-24	-45.8	-13.6	None	Reference
TMC 1004-2	15.9	-19	-32.6	-4.9	None	Reference
TMC 1004-2 Repeat	16.0	-18	-30.0	-8.4	None	Reference
TMC 1004-2 Rept2	15.7	-16	-36.2	-13.4	None	Reference
1005 (Non-TMC Blend)	21.6	-17	-17.8	-21.6		Reference
TMC 1006	25.7	-22	-24.0	-6.7	None	Reference a
L	8.4	-10	-12.8	5.2	None	Reference a1
I	8.3	-10	-17.1	20.2	None	Reference b
J	8.3	-9	-59.3	-22.7	None	Reference b
K	8.2	-9	-31.2	-8.6	None	Reference b

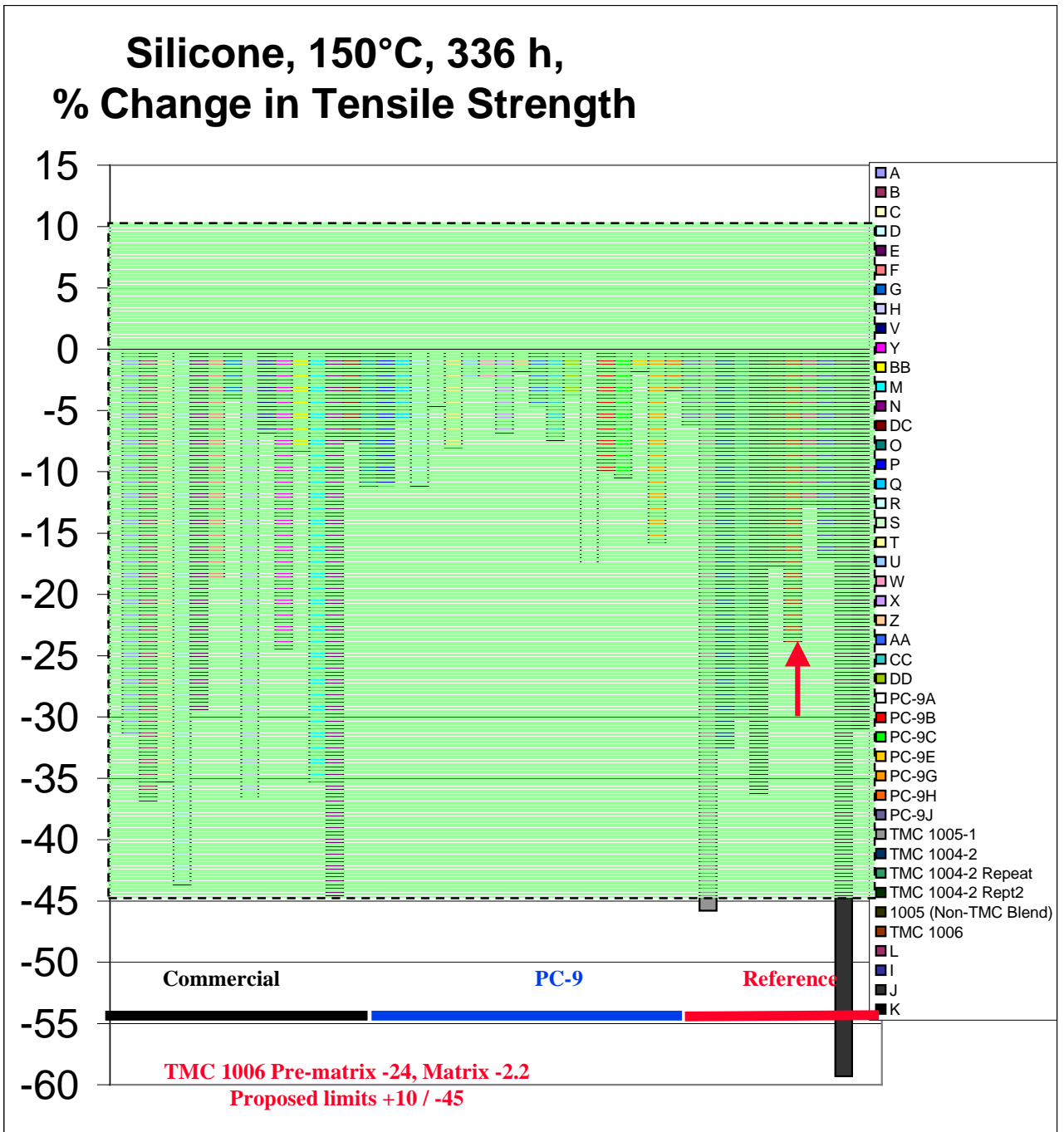
Silicone



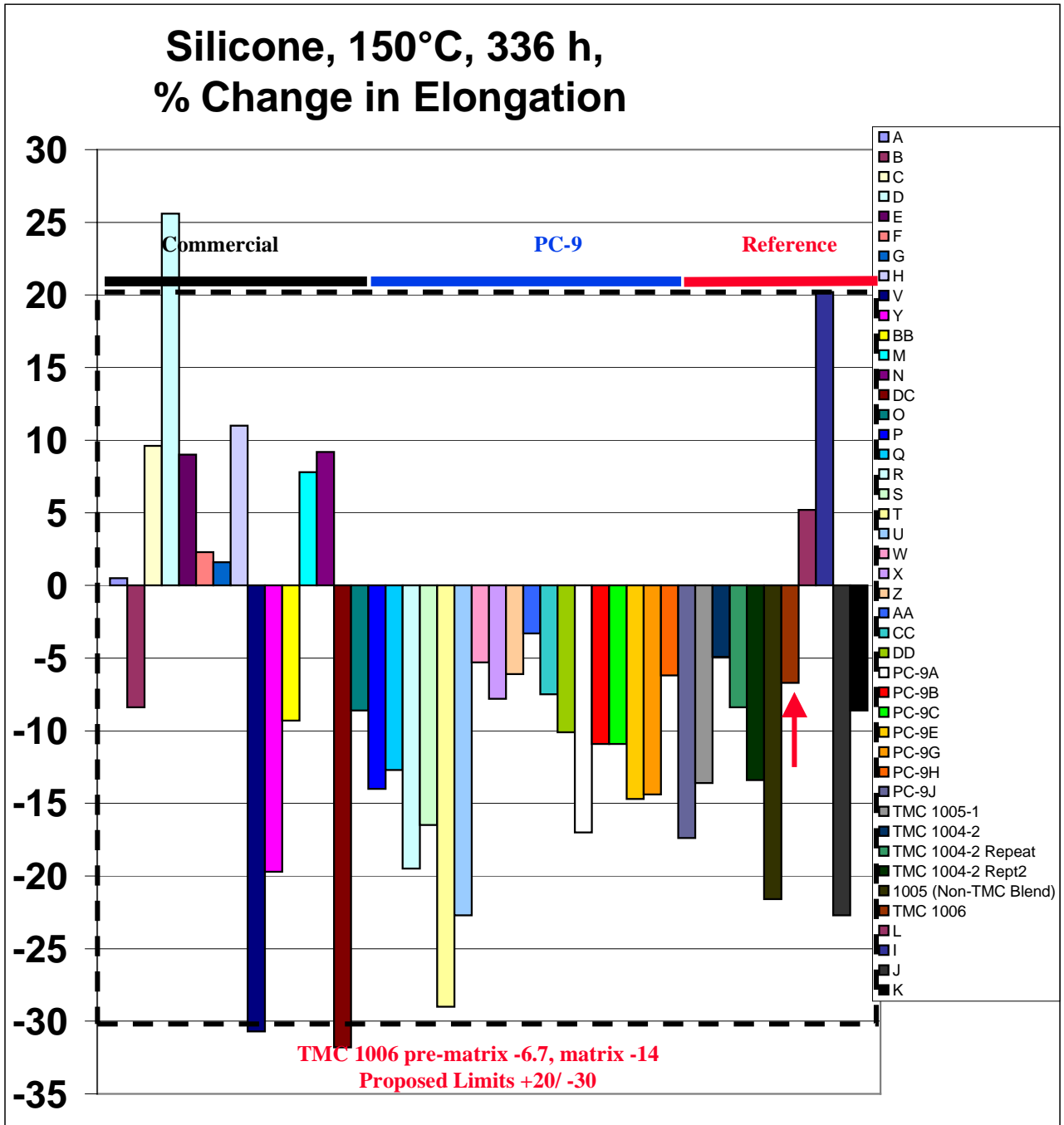
Silicone



Silicone



Silicone



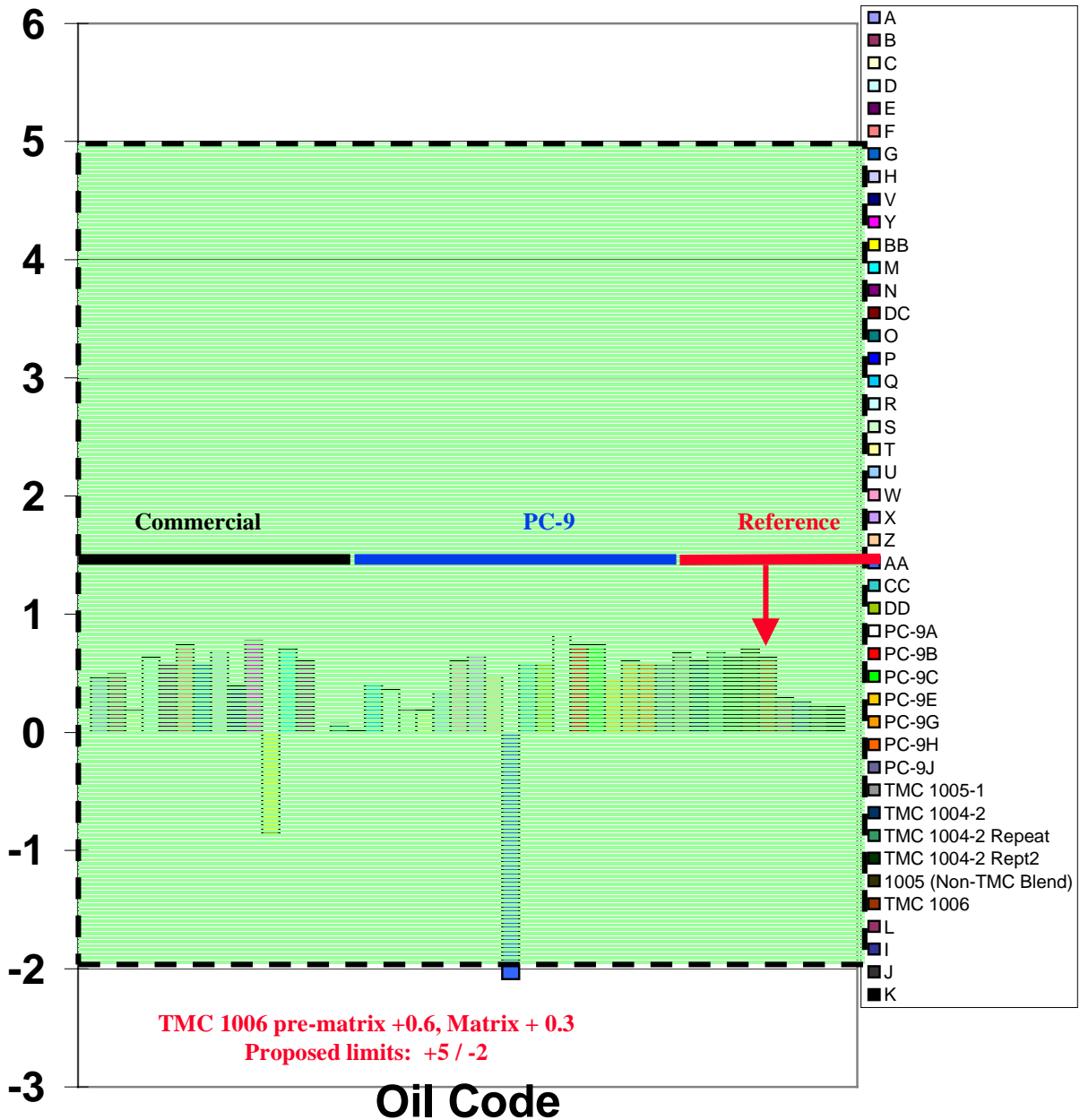
Fluoroelastomer

Fluoroelastomer, Elastomer Batch FC-2123, 150°C, 336 Hours, All Data an Average of Three Samples

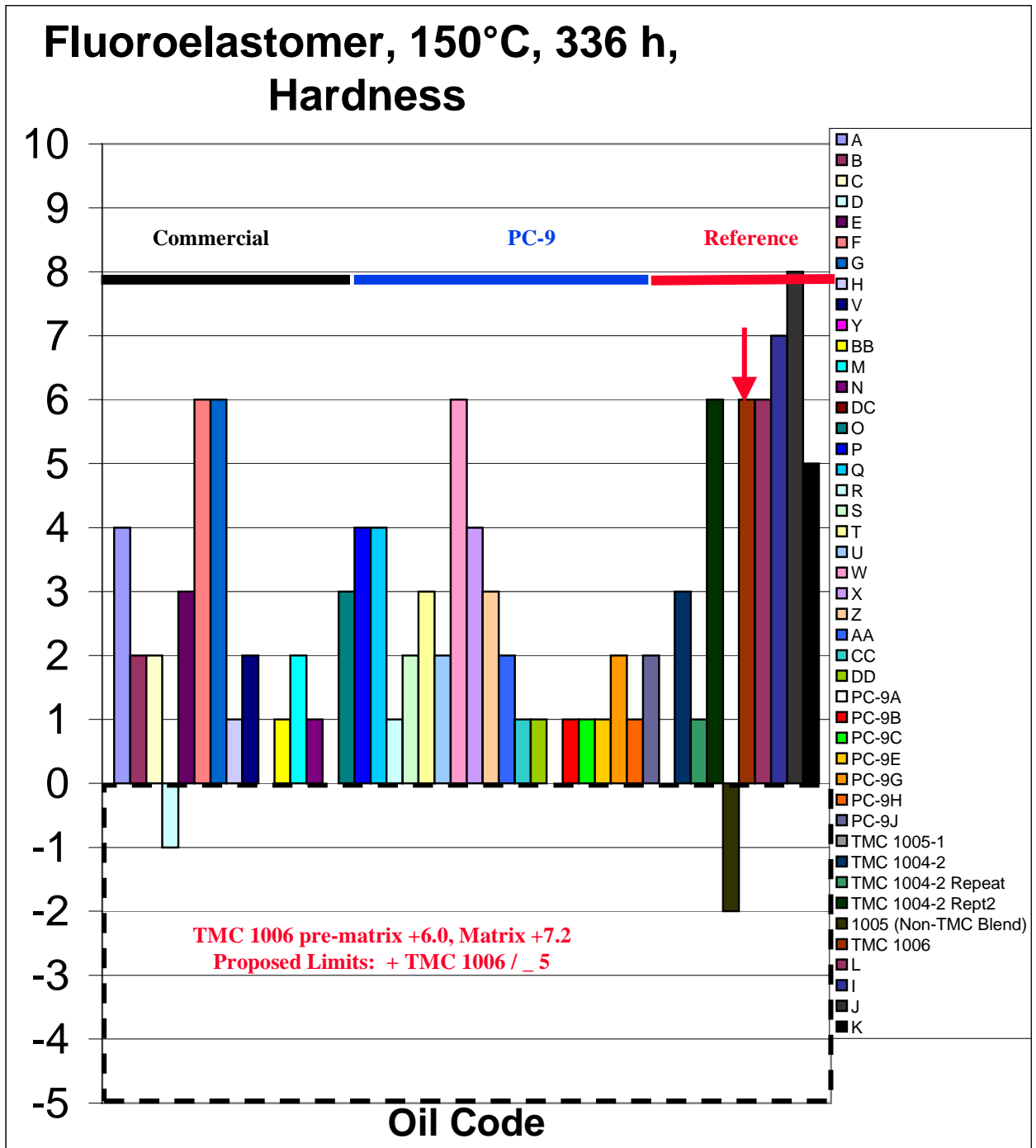
	Volume Change, %	Points Hardness Change	Tensile Strength Change, %	Elongation Change, %	Reversion	Oil Type
A	0.5	4	-47.0	-43.5	None	Commercial
B	0.5	2	-53.2	-53.5	None	Commercial
C	0.2	2	-55.4	-54.6	None	Commercial
D	0.6	-1	-30.9	-38.4	None	Commercial
E	0.6	3	-51.2	-55.1	None	Commercial
F	0.7	6	-53.4	-47.3	None	Commercial
G	0.6	6	-61.6	-54.8	None	Commercial
H	0.7	1	-35.1	-43.1	None	Commercial
V	0.4	2	-46.1	-48.7		Commercial
Y	0.8	0	-29.9	-30.9		Commercial
BB	-0.9	1	-58.0	-48.8		Commercial
M	0.7	2	-47.9	-42.6	None	Commercial ACEA
N	0.6	1	-43.8	-44.7	None	Commercial ACEA
DC	0.0	0	-34.0	-38.0		Commercial ACEB
O	0.1	3	-31.2	-39.4		PC-9
P	0.0	4	-31.8	-42.3		PC-9
Q	0.4	4	-37.8	-41.8		PC-9
R	0.4	1	-23.8	-45.6		PC-9
S	0.2	2	-23.6	-44.5		PC-9
T	0.2	3	-43.8	-56.3		PC-9
U	0.3	2	-43.8	-49.3		PC-9
W	0.6	6	-50.8	-40.2		PC-9
X	0.7	4	-46.2	-38.3		PC-9
Z	0.5	3	-42.5	-41.0		PC-9
AA	-2.1	2	-44.7	-45.2		PC-9
CC	0.6	1	-41.3	-39.3		PC-9
DD	0.6	1	-37.9	-39.7		PC-9
PC-9A	0.83	0	-41.5	-41.6		PC-9
PC-9B	0.74	1	-43.9	-47.1		PC-9
PC-9C	0.74	1	-45.3	-47		PC-9
PC-9E	0.5	1	-44.2	-40.6		PC-9
PC-9G	0.6	2	-41.4	-40.0		PC-9
PC-9H	0.6	1	-42.3	-39.6		PC-9
PC-9J	0.6	2	-44.1	-43.7		PC-9
TMC 1005-1	0.7	0	-39.7	-43.0	None	Reference
TMC 1004-2	0.6	3	-43.7	-48.7	None	Reference
TMC 1004-2 Repeat	0.7	1	-45.8	-55.9	None	Reference
TMC 1004-2 Rept2	0.6	6	-47.7	-45.1	None	Reference
1005 (Non-TMC Blend)	0.7	-2	-29.8	-36.7		Reference
TMC 1006	0.6	6	-68.8	-62.0	None	Reference a
L	0.3	6	-64.8	-61.4	None	Reference a1
I	0.3	7	-68.0	-70.0	None	Reference b
J	0.2	8	-67.6	-57.6	None	Reference b
K	0.2	5	-61.4	-54.6	None	Reference b

Fluoroelastomer

Fluoroelastomer, 150°C, 336 h,
% Volume Change

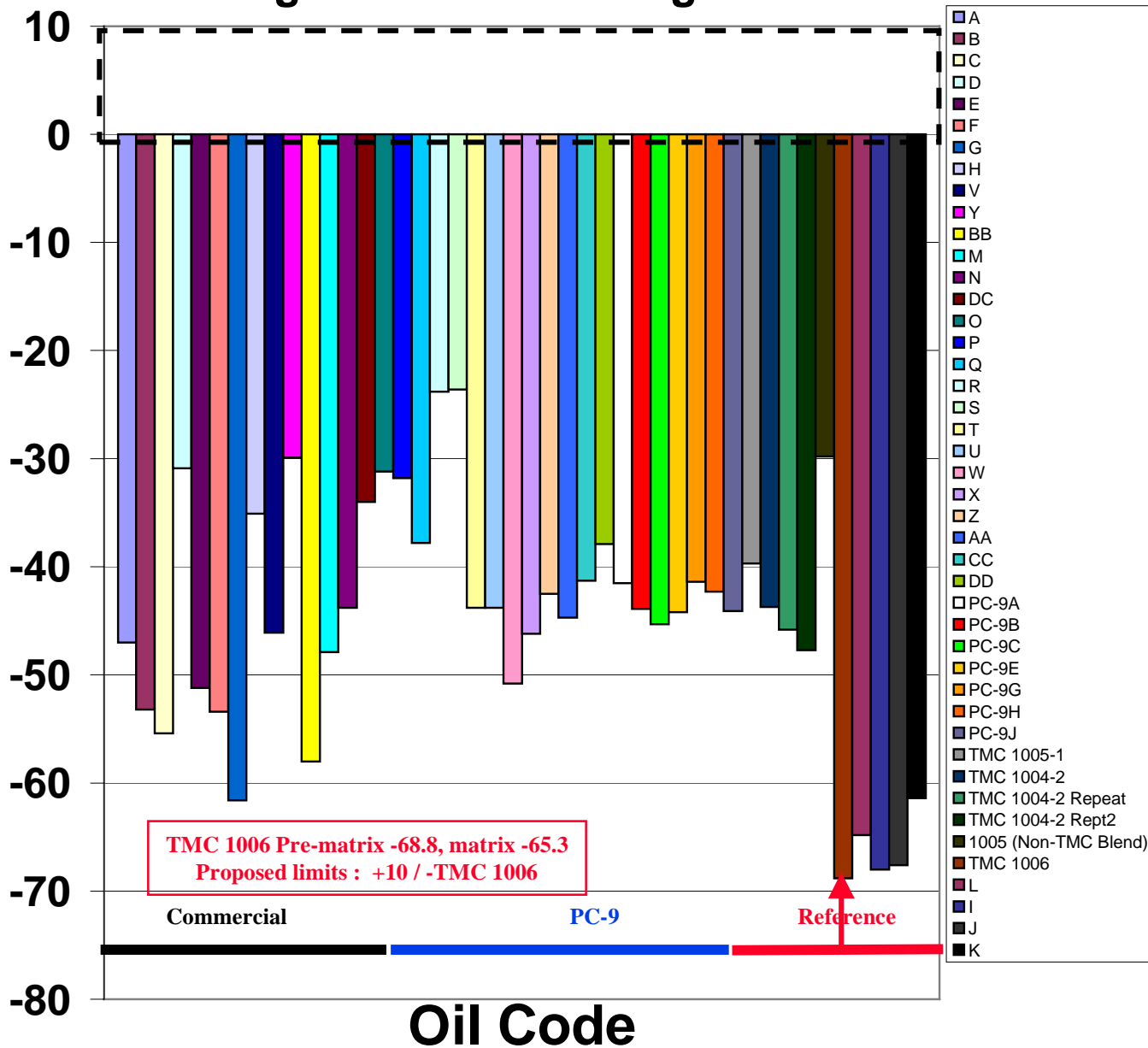


Fluoroelastomer



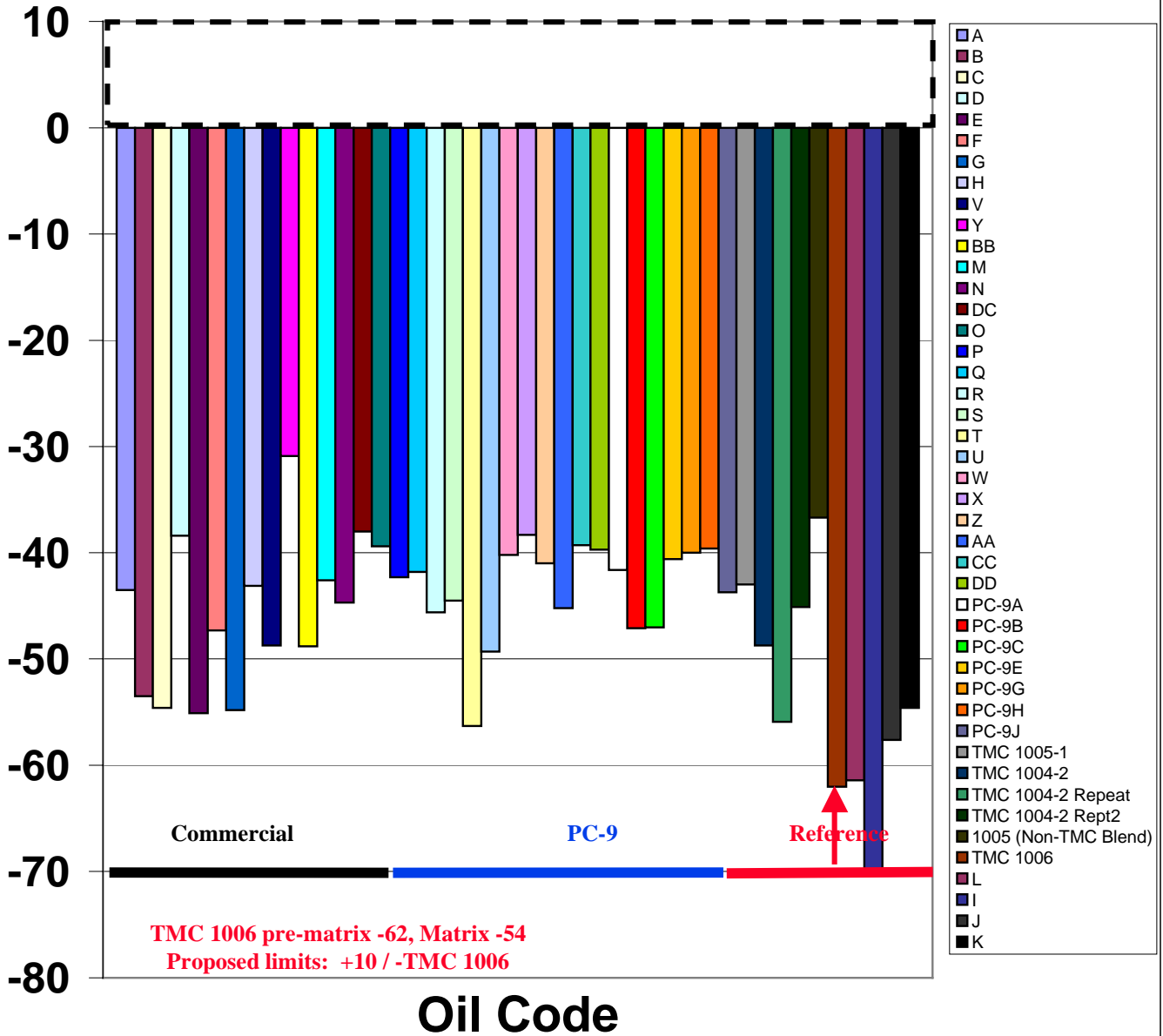
Fluoroelastomer

Fluoroelastomer, 150°C, 336 h,
Change in Tensile Strength



Fluoroelastomer

Fluoroelastomer, 150°C, 336 h,
Change in Elongation



Standard Test Method: Test Method for Determining Automotive Engine Oil Compatibility with Typical Seal Rubbers and Elastomers”

Introduction

This test method standard calls for the use of reference immersion oils and reference test materials (rubber or elastomer compounds) that are used in the evaluation of candidate oils or candidate compounds. Most of the reference materials of all types used in various in Committee D11 standards are materials that have been evaluated and accepted by the use of D4678, “Preparation, Testing, Acceptance, Documentation and Use of Reference Materials”. These D4678 materials are designated as Industry Reference Materials or IRMs.

For a number of reasons the reference materials of this engine oil compatibility standard were not able to be evaluated by the procedures of D4678 and therefore do not bear the name IRM. The reference materials of this engine oil standard are offered for use in the evaluation of candidate rubbers, elastomers and oils on an ad hoc basis.

1. Scope -

1.1 Effective sealing action requires that the rubber or elastomer compound used for any seal have a high level of resistance to the liquid or oil in which it is immersed. When a high level of resistance exists, the rubber is said to be compatible with the liquid or oil.

1.2 This standard provides test method procedures for a *preliminary* or first order evaluation of the compatibility of (1) oils classified as Automotive Engine Oils and (2) rubbers or elastomers used in the sealing materials in contact with these oils. Since seals may be static or dynamic and may operate over a range of conditions, a complete evaluation of the potential sealing performance of any rubber-oil combination in any service condition usually requires additional tests.

1.3 This test method may be used to determine the compatibility of rubbers and elastomers such as nitrile (NBR), polyacrylate (ACM), fluoroelastomer (FKM) and silicone (VMQ) with Automotive Engine oil. Other candidate rubbers and elastomers as proposed for use in conjunction with any candidate Automotive Engine oil may also be evaluated.

1.4 The testing procedures as described in D412, on stress strain evaluation, in D471, on the effect of rubber immersion in liquids, in D865 on the use of special test tubes for aging evaluation, in D2240, on the measurement of hardness and in D5662, gear oil compatibility with typical oil seal rubbers and elastomers, are all used in the execution of the operations of this standard. The user of this standard should be proficient in the use of these additional cited standards.

1.5 (Standard ASTM safety boilerplate text here)

2. Referenced Documents

2.1 ASTM Standards

2

D412 - Standard Test Method - Rubber Properties in Tension

D471 - Standard Test Method - Rubber Property - Effect of Liquids

D1349 - Standard Practice - Rubber: Standard Temperatures for Testing

D2240 - Standard Test Method - Rubber: Durometer Hardness

D5662 - Standard Test Method - Determining Automotive Gear Oil Compatibility with Typical Oil Seal Elastomers

D865 - Rubber - Deterioration by Heating in Air (Test Tube Enclosure)

3. Terminology

3.1 *Description of Terms Specific for this Standard* - These terms are defined in a sequential order; the simple or more basic definitions are defined first then the more complex terms. The basic terms are then used as a part of later definitions. This produces the most meaningful and succinct definitions.

3.1.1 *compatibility*, n. - a characteristic of a rubber or elastomer 'compound-liquid' combination that signifies a complete or high level of resistance of the compound, to deleterious effects imparted by contact with or immersion in, the liquid.

Discussion: The phrase 'high compatibility' indicates that after contact or immersion, the compound properties are maintained at or near their original level.

3.1.2 *immersion test*, n. - an operation to evaluate compatibility; it determines the effect of a liquid on rubber or elastomer compound test specimens maintained beneath the surface of the liquid for a specified time and temperature.

Discussion: The effect of the liquid is evaluated by the difference in (typical) compound physical test properties before and after immersion.

3.1.3 *reference compound*, n. - a compound prepared using a specified rubber or elastomer (formulation) that has well established immersion test properties with selected oils or liquids, obtained by the use of recognized and accepted testing and documentation procedures.

3.1.4 *reference oil*, n. - an immersion liquid that has well-established properties, obtained by the use of recognized and accepted testing and documentation procedures.

Discussion: The 'established properties' may be chemical or compositional properties or properties as developed from immersion tests using reference compounds.

3.1.5 *candidate compound*, n. - a non-reference or experimental rubber or elastomer compound to be evaluated for compatibility with reference or candidate oils or liquids.

3.1.6 *candidate oil*, n. - a non-reference or experimental oil or liquid to be evaluated for its effect on (compatibility with) reference compounds or experimental compounds.

3.1.7 *Tensile strength*, n - the maximum tensile stress applied in stretching a specimen to rupture.

4. Summary of the Test Method

4.1 Rubbers and elastomers immersed in selected oils are aged at a selected temperature specific for each oil - rubber or elastomer combination, for 14 days or 336 hrs. The performance of the rubber or elastomer is determined by its resistance to change in typical physical properties after immersion such as; stress-strain, elongation at break, tensile strength, hardness (Durometer Type A), and dimensional properties (volume change). The results of the immersion testing are usually expressed in terms of the percent change in the selected properties, (after immersion - before immersion). A negative percent change indicates a reduction in property level.

4.2 Both reference oils and candidate or experimental oils may be evaluated. When comparisons among any series of oils are to be made, the test conditions for all oils (reference and candidate) shall be well controlled especially the concurrent immersion test aging, in the same temperature controlled heating device.

5. Significance and Use

5.1 Engine oil formulations have been shown to lack compatibility (cause deleterious effects) with certain rubbers and elastomers. These deleterious effects are greatest under two conditions (1) with virgin or new engine oil, i.e.. oil that has not been exposed to an engine's operating environment and (2) when the exposure is at elevated temperatures.

5.2 This method evaluates the relative effects of new or candidate engine oil formulations using four reference compounds as cited in the Scope. See also Annex A2. The performance of the new engine oils is determined by a comparison of the reference compound immersion test data for the candidate oils with the reference compound immersion test data for the reference oils. This comparison permits decisions on the anticipated or predicted performance of the candidate oils in service. The standard also permits an evaluation of candidate compounds with either reference or candidate oils or liquids. This comparison permits decisions on the anticipated or predicted performance of the candidate compounds in service.

5.3 This test method is suitable for specification compliance testing, quality control, referee testing and research and development.

6. Apparatus

6.1 The testing equipment as specified in D412, D471, D865, D2240 and D5662 is required for the use of this standard.

6.1.1 *Hardness Durometer* - See D2240.

6.1.2 *Tension Testing Machine* - See the appropriate sections of D412. The rate of grip separation for the tension testing shall be 500 +/- 50 mm per min (8.5 +/- 0.8 inches per min).

6.1.3 *Glass tubes*, of borosilicate glass if possible, shall be used, having an outside diameter of 38 (+0, -1 mm) and an overall length of 300 mm. Each tube is fitted loosely with an aluminum-covered cork stopper or an equivalent inert sealing device that will not contaminate the immersion oil. An inert (stainless steel) wire rod is hung over the edge of the glass tube and held in place by the stopper. It is used to hold the test specimens submerged in the immersion liquid. See Figure 1 and 2 and see D865, sec 5.

6.1.4 A *Heated Immersion Test Bath or Block*, or equivalent shall be used. This heating device should be capable of maintaining the oil sample in the glass tube within +/- 1 deg C of the test temperature. The

4

immersion test bath or block shall contain a rack or holes which will accept the glass tubes specified in 6.1.3 and hold them in a vertical position.

6.1.5 *Dumbbell cutting die* should use that referenced in ASTM D-412. Die C is required.

7. Reference Materials

7.1 *Reference Oils* - The reference oils are maintained and distributed by the ASTM Test Monitoring Center (TMC), see Annex A1. .

7.1.1 The TMC is responsible for managing a system that ensures the performance and formulation consistency of the reference oils. Reference oils shall be stored in locations where the ambient temperature does not exceed 32 deg C. Under these conditions the shelf life of the reference oils is five years unless otherwise specified by the TMC using documented analysis procedures for a longer projected shelf life.

7.2 *Reference Compounds* - Reference compounds are available from an organization known as the Parts Distributor (PD), see Annex A1. The four reference compounds are (1) a fluoroelastomer (FKM), (2) a polyacrylate material (ACM), (3) a silicone rubber (VMQ) and (4) a nitrile rubber (NBR). See Annex A2 for formulations. A numbering system has been established by the PD of the format: [type] X; where 'type' is either (FKM), (ACM), (VMQ) or (NBR). and X = batch number for the particular formulation. The physical properties as reported in Annex A2 are typical values.

7.2.1 The PD is responsible for (1) maintaining the numbering and tracking system for the reference compounds and (2) for managing the procurement of rubbers and elastomers that meet the specifications of this standard. The reference compounds shall be stored in a location shielded from light, where the relative humidity is in the range 40 to 55% and the temperature in the range of 10 to 25 deg C. Under these conditions the shelf life of the reference compounds is three years from the date of cure. Any immersion test using a reference compound older than three years shall be treated with caution.

7.3 Table 1 lists the reference compounds, the specified immersion test temperatures and the reference oils. As noted above these are available from the Parts Distributor and/or the Test Monitoring Center. See Annex A1.

8. Procedure for Immersion Testing

8.1 *General Background* - The immersion tests for any candidate oil (or oils) shall be conducted on the basis of a 'test series' operation. A test series is a complete evaluation program using the specified physical tests, for any selected number of candidate and reference oils and/or candidate (experimental) or reference compounds. The test results for the specified physical tests obtained for the reference compounds in the candidate oils shall be compared to the test results, for the specified physical tests, for the reference compounds in the reference oils. For this comparison insure that the same compound batch is used for both candidate and reference oils. Refer to Table 1 for the immersion test temperatures and reference oils for each reference compound.

8.2 *Number of Test Specimens* - There are two types of test specimens, (1) dumbbells for stress-strain testing and (2) 25 x 50 mm rectangular sheets for dimensional changes or volume swell and hardness testing. For the dumbbell specimens, twelve (12) test specimens shall be prepared; six (6) for original property testing and six (6) for aged (after immersion) testing. For the rectangular specimens, six (6) test specimens should be prepared; the same specimen is used for original property testing and for aged (immersion) testing. The total number of specimens of both types is determined by the scope of the

5

testing; the number of candidate compounds or candidate oils to be evaluated and the number of selected reference compounds or reference oils in the test series. The assessment of the total number of specimens is based on the use of the standard to evaluate both candidate compounds as well as candidate oils. It is important that all reference compounds in one test set be from the same batch.

8.3 Test Specimen Preparation - Using the total number of specimens as required for each candidate or reference compound for the entire test series as determined in 8.2. , select the number of sheets for each compound as required for the projected testing. This will depend on the number of oils to be used for immersion. Condition the sheets from which test specimens are to be cut for 3 hr at 23 +-2 deg C as specified in D412.

8.3.1 Stress-Strain Testing - For each candidate or reference compound, cut the required number of dumbbell specimens from the sheets. This cutting shall be with Die C as specified in D412, parallel to the grain, using sharp well prepared dies that are unaltered though out the entire cutting operation. Use a die press for the operation to cut only one sheet thickness for all cutting operations. Since two or more sheets will be required for the total number of dumbbells for any compound, it is necessary that each sample of six dumbbells (original and aged sample sets) contain as close as possible an equal number of dumbbells from each of the individual sheets as required for the testing.

8.3.2 Dimensional Properties (Volume Swell) - As specified in D471, cut the number of required number of 25 x 50 x 2.0 +- 0.1 mm (1.0 x 2.0 x 0.08 +- 0.005 inch) specimens from the sheets. Since two or more sheets will be required (to prepare at least 12 specimens) for the total number of specimens for any compound, it is necessary that each sample of six (original and aged sample sets) contain an equal number of specimens from each of the individual sheets as required for the testing.

8.3.3 Hardness Testing - The hardness testing is conducted on the specimens prepared for the volume swell testing.

8.4 Physical Testing: Initial Measurements - Conduct the initial or original physical testing measurements using six of the prepared test specimens. Measurements that must be recorded include: (1) Initial and final (after immersion) modulus, elongation at break and tensile strength, (2) initial and final (after immersion) mass of 25 x 50 specimens in air and water (or other liquid used for the weighing operation) and (3) initial and final (after immersion) hardness using the 25 x 50 specimens.

8.4.1 Stress-Strain - Using the procedure as specified in D412, test six dumbbells, recording for each (1) the elongation at break and (2) the maximum tensile stress (tensile strength) applied to the specimen in stretching the specimen to break. Calculate and record the original average and standard deviations of the six measurements for each of the two properties.

8.4.2 Dimensional Properties: Original (Initial) Mass - Use the water displacement method as described in D471 to conduct the initial mass (weight) measurements for the 25 x 50 mm specimens. Weigh each specimen in air to the nearest 1 mg. This original mass is recorded as M_1 . Immerse the specimen into a 1% solution of Aerosol OT in water before weighing in distilled water, insuring that there are no air bubbles clinging to the specimen. Record this original 'in water' mass as M_2 .

8.4.3 Hardness - Using a Type A Durometer as described in D2240, stack three of the 25 x 50 mm specimens on top of each other for a 6 mm thickness. Take three readings from the topside of the stacked specimens rotate the top specimen over and take additional three readings from the bottom side of the top specimen. The readings are taken 1 sec after the pin make s contact with the rubber.

6

8.4.3.1 After this set of 6 measurements, rotate the bottom specimen to the top and the top specimen to the bottom. Take another set of 6 readings as above.

8.4.3.2 After this set of 6 measurements, rotate the bottom specimen to the top and the top to the bottom. Take another set of 6 readings as above. Record all 18 measurements from this stack of three specimens.

8.4.3.3 Repeat the set of 18 hardness readings for the second set of 3 rectangular specimens. Calculate an average for the original hardness from all 36 readings.

8.5 *Initiating the Immersion Tests* - All immersion tests for a test series, shall be conducted concurrently in the same heated immersion test block.

8.5.1 Fill the immersion test tubes with $150 \pm 5 \text{ cm}^3$ of the candidate or reference oil as appropriate. Four test tubes are required for each compound - oil combination. In each tube suspend three rectangular specimens or three dumbbell specimens from a stainless steel wire hanger, see Figure 1. Inert (to oil or rubber) spacers should be located between each test specimen to prevent specimens from touching each other or the test tube wall. See Figure 2. Each test tube shall be covered with a stopper as specified in 6.1.3.

8.5.2 Insert the test tubes into the heating block that has been set to the temperature required for the evaluation, on a random basis and age for a period of 336 ± 0.5 hrs. Insure that no specimen touches another specimen or the test tube wall. Such an occurrence will invalidate the test. To insure that aging conditions are equal for candidate and reference oil tests, the final endpoint of the 336 hr aging for either oil should be the same with an allowed tolerance or difference of 8 hrs.

8.6 *Terminating the Immersion Tests* - At the end of the aging period, remove the specimens from the test tubes and place them (while on the wire hanger) on a clean absorbent towel or surface. Allow for a cooling period while on the hanger of no more than 30 minutes.

8.6.1 Remove the specimens from the wire hanger and place them on a new clean absorbent towel or surface. Remove the excess oil with a clean absorbent towel. Begin the 'after immersion' testing approximately 30 to 60 minutes after removal from the test tube. Complete the final 'after immersion' testing within 2 hrs from time of removal from the test tube.

8.7 *Physical Testing: After Immersion Measurements* - The response of the immersed compound test specimens to the effects of the oil or liquid is assessed on the basis of the change in properties, before immersion Vs after immersion, expressed as a percent with the exception of hardness which is expressed in points. Using the procedures as set forth in 8.4.1, 8.4.2 and 8.4.3 repeat all of the stress-strain, dimensional property mass measurements (in air and water or other suitable liquid) and hardness measurements. Record these measurements as described above in 8.4. It is suggested that the hardness measurement be taken before the volume measurement to insure that the Durometer tip is kept free of moisture.

8.8 *Percent Change in Properties* - The percent change in stress strain properties is given by Equations 1, and 2 for elongation at break and tensile strength. The percent change in the volume of the test specimen is given by Equation 4 and the percent change in hardness is given by Equation 5.

$$\% \Delta E = [(E_A - E_O) / E_O] 100 \quad (1)$$

% ΔE = percent change in elongation at break

E_O = original elongation at break

7

E_A = aged (after immersion) elongation at break

$$\% \Delta TS = [(TS_A - TS_O) / TS_O] 100 \quad (2)$$

% Δ TS = percent change in tensile strength

TS_O = original tensile strength

TS_A = aged (after immersion) tensile strength

Equation 3 is given for test specimen weighing in air and in water, which has a density of 1.00, for liquids other than water use alternative Equation 3A.

$$\% \Delta V = [\{ (M3 - M4) - (M1 - M2) \} / (M1 - M2)] 100 \quad (3)$$

% Δ V = percent change in volume

M1 = original mass in air, g

M2 = original mass in water, g

M3 = mass in air after immersion test, g

M4 = mass in water after immersion test, g

$$\% \Delta V = [(M3 - M1) / \{ d (M1 - M2) \}] 100 \quad (3A)$$

M1 = original mass in air, g

M2 = original mass in liquid, g

M3 = mass in air after immersion test, g

d = density of liquid, g/cm³

$$\Delta H = (H_A - H_O) \quad (4)$$

Δ H = point change in hardness

H_O = original hardness

H_A = aged (after immersion) hardness

9. Report

9.1 For each of the percent change in property parameters and point change for hardness as evaluated in 8.8, calculate the average and standard deviation. Appendix X1 contains recommended data forms to report the results of the immersion testing in the evaluation of candidate oils. These may be modified (1) for use with compounds other than the four reference compounds as listed and (2) for use in evaluating candidate rubber or elastomer compounds with selected reference oils or candidate oils.

9.2 The following data and information should be in the report for each liquid or oil evaluated:

9.2.1 Rubber or elastomer (batch, date and code), test date, test number

9.2.2 Test temperature, deg C

9.2.3 Test duration, hrs

9.2.4 Percent volume change (D471)

8

9.2.5 Change in Durometer hardness, points (D2240)

9.2.6 Percent change in tensile strength, (D412)

9.2.7 Percent change in elongation at break, (D412)

9.2.8 Aging block or bath identification

9.2.9 If requested, report to the TMC the data and information on the reference materials from the test

10. Precision and Bias

10.1 The precision and bias for this method has not been evaluated. A program to evaluate the precision will be undertaken as soon as possible after the adoption of this standard.

11. Key Words

11.1 compatibility, rubber, elastomer, automotive oil, heavy duty engine oil, seal,

Annex A1

A1.1 *Reference Materials* - Reference oils may be obtained from the ASTM Test Monitoring Center or TMC located at 6555 Penn Avenue, Pittsburgh PA 15206; phone 412-365-1010; Fax 412-365-1047. In order to receive reference oils individual laboratories shall agree to furnish the TMC with immersion test result data developed with these reference oils

Note: Unless otherwise specified, these oils are not Committee D11 IRMs.

A1.2 *Reference Rubbers or Elastomers* - Cured prepared sheets of reference rubbers or elastomers may be obtained from an organization known as the Parts Distributor or PD. The company currently functioning in this capacity is; OH Technologies Inc., Attn Jason Bowden, PO Box 5039, 9300 Progress Parkway, Mentor OH 44060; phone 440-354-7007, Fax 440-354- 7080; email: jhbowden@ohtech.com

Note: Unless otherwise specified, these oils are not Committee D11 IRMs

Annex A2

Elastomer materials, formulations, and expected properties of cured materials:

Elastomer	Ingredients	Parts	Points Hardness	Tensile, Mpa	Elongation, %	Specific Gravity
Fluoroelastomer (FKM)	Viton A-275C or Fluorel FC-2123	100.00	71	13.3 min	270	1.84
	Maglite D	3.00				
	N-990 Carbon Black	30.00				
	Calcium Hydroxide - Reagent Grade	6.00				
	Press Cure:	10 min. @ 177°C				
Post Cure:	16 hrs. @ 232°C					

9

Viton is a registered trademark of Dupont Dow
Elastomers, Fluorel is a registered trademark of 3M

Polyacrylate (ACM)	HyTemp 4051 EP	100.00	66	11.9	175	1.31
	N-550 Carbon Black	65.00				
	Stearic Acid	1.00				
	Naugard 445	2.00				
	TE-80	2.00				
	Sodium Stearate	4.00				
	HyyTemp NPC-50	2.00				

Press Cure: 12 min. @ 170°C

HyTemp is a registered trademark of Zeon Chemicals

Silicone (VMQ)	Dow Corning Product ID.24122V-BLK					
	Cure:	vulcanized 5 minutes @ 370°F				
	Pots Cure:	4 hours @ 200°C				

Nitrile (NBR)	Nipol DN3350	100.00	68	19.6	290	1.25
	Zinc Oxide	5.00				
	Stearic Acid	2.00				
	Stangard 500	2.00				
	N-774 Carbon Black	70.00				
	Thiokol TP-95	5.00				
	Varox DCP40KE	3.00				

Press Cure: 12min. @ 170°C

Nipol is a registered trademark of Zeon Chemicals

Manufacturer shall mark each elastomer sheet to designate the direction of the grain of the material.

Figures

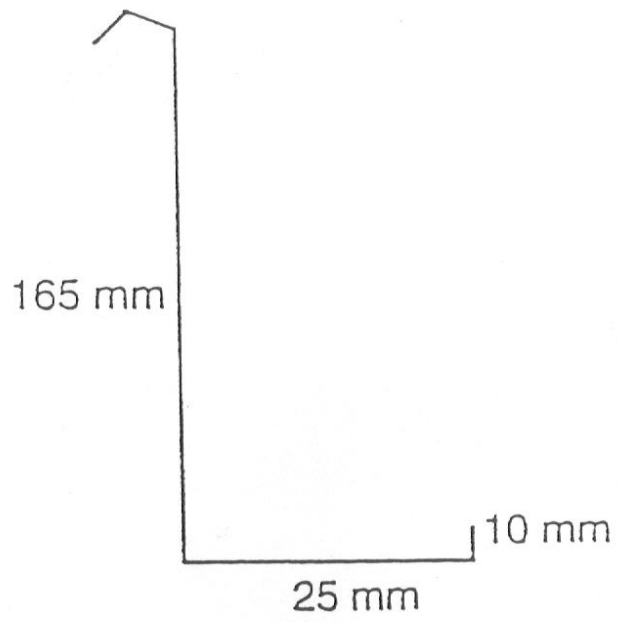


FIG. 1 Wire Hanger

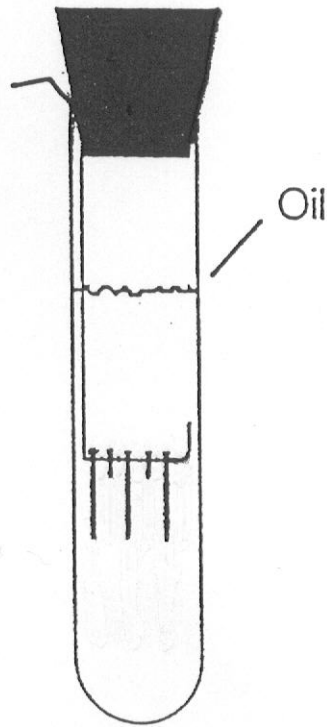


FIG. 2 Test Tube Arrangement

Appendix

X1 Recommended Report Forms

REPORT FORMS

PC-9 OIL SEAL COMPATIBILITY TEST						
Candidate Oil Test						

Sample Code:

Rubber or Elastomer (Batch)	Test Temperature, °C	Test Duration, Hours	Volume Change, %	Points Hardness Change	Tensile Strength Change, %	Elongation Change, %
Nitrile (NBR) (NBR0500S)	100	336				
Average						
Standard Deviation						

Polyacrylate (ACM) (ACM1199S)	150	336				
Average						
Standard Deviation						

Fluoroelastomer (FKM) (FKM1199S)	150	336				
Average						
Standard Deviation						

Silicone (VMQ) (SIL1199S)	150	336				
Average						
Standard Deviation						

Signature Block

PC-9 OIL SEAL COMPATIBILITY TEST

Reference Oil Test

Reference Oil Code:

Rubber or Elastomer (Batch)	Test Temperature, °C	Test Duration, Hours	Volume Change, %	Points Hardness Change	Tensile Strength Change, %	Elongation Change, %
Nitrile (NBR) (NBR0500S)	100	336				
Average						
Standard Deviation						

Reference Oil Code:

Polyacrylate (ACM) (ACM1199S)	150	336				
Average						
Standard Deviation						

Reference Oil Code:

Fluoroelastomer (FKM) (FKM1199S)	150	336				
Average						
Standard Deviation						

Reference Oil Code:

Silicone (VMQ) (SIL1199S)	150	336				

14

Average						
Standard Deviation						

Signature Block

PC-9 Elastomer Test

Proposed Limits

Proposed Limits

Elastomer	Volume Change	Hardness	Tensile Strength	Elongation
Nitrile	+5 / -3	+TMC 1006 /-5	+10/TMC 1006	+10/-TMC1006
Silicone	+TMC 1006/-3	+1/ -TMC 1006	+10/-45	+20/-30
Polyacrylate	+5/-3	+8/-5	18/-15	10/-35
FKM	+5/-2	+TMC 1006/-5	+10/-TMC 1006	+10/-TMC 1006