November 15, 2007

Reply to: Fred Gerhart

Southwest Research Institute

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### UNCONFIRMED MINUTES from the SEQUENCE VIII SURVEILLANCE PANEL

### Held in San Antonio, Texas November 14, 2007

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### Welcome

Chairman Fred Gerhart called the meeting to order at 3:00 PM. The agenda was accepted and is included as Attachment 1.

### Membership Changes

The attendance record of the voting membership and guests is included as Attachment 2. Phil Scinto represented Lubrizol for this meeting only. No other changes were noted in the membership.

### **Motion and Action Items**

Bill Buscher III recorded the motion and action items of this meeting, which are included as Attachment 3.

### Approval of Conference Call Minutes from July 11, 2007

The minutes from this conference call were distributed for review and were accepted by a vote of general consent. A short time later, Charlie pointed out that these minutes were not complete. An amended set of minutes was distributed to the panel from the chairman by e-mail after the meeting and is included as Attachment 4. These minutes will also be posted on the TMC web site.

### **Review of TMC Report by Rich Grundza**

The TMC report for the reporting period of April 1, 2007 through September 30, 2007 was distributed to the panel by e-mail on October 9, 2007 by John Zalar as "Memo 07-056 - Sequence VIII Semiannual Report" and is included as Attachment 5.

At the chairman's request, Rich reviewed the current lead leaching rate, the status of industry precision and severity, and the reference oil inventory.

- The highest value of lead during the reporting period was 202 ppm. The last two values reported in this period were 37 ppm and 80 ppm.
- Industry precision is in control. Industry severity is at EWMA warning limits but should correct itself as targets continue to evolve for 1006-2. The industry is currently at n count of 13 for 1006-2 on the 0306 bearings. The next target update is to occur at n count of 20.
- There is an ample supply of reference oil.

The panel accepted the TMC report by a vote of General Consent.

### TEI CPD Report - by Zack Bishop

The CPD report is included as Attachment 6.

There are 2,549 sets of 0306 bearings in Argon storage at TEI.

The latest ICP data from two sets of bearings at TEI show the lead content in the storage oil continuing to increase. Sample PAO1 increased from 98 ppm to 158 ppm. Sample PAO2 increased from 113 ppm to 190 ppm. TEI believes the source of the lead to be from a coating of lead residue left over from broaching and not due to lead leaching from the actual matrix of the 0306 bearing. The change out of bearing storage oil has not started due to supply issues by the vendor of the plastic storage containers. TEI has all other components on hand and will start the change out as soon as the plastic storage containers arrive. The bearings will be cleaned and placed into new containers of EF-411 and will be issued a new lot code of 01A. The change out is scheduled for completion by February 2008.

The annual order for 2007 has been fulfilled. A letter of solicitation for the 2008 annual order will be sent to the laboratories shortly.

There was discussion of the CPD report by the panel.

The panel agreed that laboratories are to change to the lot code 01A bearings at the start of a reference period. Existing referenced stands will be allowed to complete their calibration periods using lot code 01 bearings, which are packed in PAO8.

Sid Clark reviewed data from 0306 bearings inspections that were conducted at GM. In April of 2007, TEI provided one set of bearings that was stored PAO8 with a blanket of Argon gas. The storage oil was clear and the bearings were in perfect condition. The report from the GM metallurgical laboratory is attached as Attachment 7. This particular set of bearings had the matrix intact and showed no signs of leaching. During June 2007, Sid visually inspected a second set of bearings from SwRI on hand inventory. The storage oil for these bearings was not clear and there was a brown ring on the bottom of the container. When placed under a standard microscope, Sid could see areas where the witness marks of the broaching operation were not present indicating evidence of lead leaching from the surface of the bearing. Sid cautioned the group that when preparing surface mount samples for SEM analysis do not polish because the polishing operation will remove evidence of lead leaching and restore the surface. A better method is to

mount cross sections of the bearing, which will allow polishing and will also ensure preservation of any evidence of lead leaching. After much discussion, TEI agreed to inspect cross sections of random samples of 0306 bearings using a SEM.

### Fuel Supplier Report

The fuel supplier provided the chair with a report. The report is included in this document as Attachment 8.

### **Old Business**

Discussion took place about re-introducing 1009 as a reference oil or to pursue introduction of a "GF-5" potential reference oil from Lubrizol. TMC has two drums of this potential new reference oil and has coded it as oil 705. The consensus of the panel was to drop oil 1009 and pursue potential GF-5 reference oils that have performance close to the GF-4 pass limit of 26 mg. The panel requested TMC to provide analytical data of oil 705 for review by the panel.

The current reference interval is 3 months or 12 tests whichever comes first. After discussion, a motion was made to restore the reference interval to 6 months or 15 tests whichever comes first. Motion Passed.

### **New Business**

The chairman called for new business. No new business was presented for panel consideration.

### Review of Scope and Objectives

The panel reviewed the Scope and Objectives. An objective was added for the panel to monitor the progress of the comprehensive review of the different LTMS systems utilized by the PCMO test types and their pros and cons with a completion date of May 2008. The Scope and Objectives are attached as Attachment 9.

### **Adjournment**

By General Consent, the meeting adjourned at 4:20 PM. The next meeting of this panel will be at call of the chair.

### **Sequence VIII Surveillance Panel Agenda**

SwRI in Building 209, Conference Room 103.

### November 14, 2007 (Wednesday)

3:00 PM - 5:00 PM

- 1. Call to Order by Chairman Fred Gerhart
- 2. Membership Changes
- 3. Attendance Sign-in
- 4. Motion and Action Items Recorder
- 5. Approval of Conference Call Minutes from July 11, 2007
- 6. TMC Report Rich Grundza
  - a. Lead leaching rate
  - b. Any industry precision or severity issues?
  - c. Reference oil inventory
- 7. CPD Report TEI
- 8. Old Business
  - a. Introduce New Reference Oil or Keep 1009?
  - b. Restoration of Sequence VIII Reference Interval
- 9. New Business
- 10. Review Scope and Objectives
- 11. Next Meeting At the call of the chairman
- 12. Adjournment

### **ASTM SEQUENCE VIII SURVEILLANCE PANEL VOTING MEMBERSHIP ATTENDANCE RECORD**

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BILL BUSCHEN\_I SWAL

### ASTM SEQUENCE VIII SURVEILLANCE PANEL NON- VOTING MEMBERSHIP and GUESTS ATTENDANCE RECORD

Name	Address	ddress Phone/Fax/Email		
			Attendance	
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	LINGER, 10 7 8 1036			
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D.				

### Sequence VIII Surveillance Panel November 14, 2007 3:00PM – 5:00PM SwRI, Building 209, Conference Room 103 San Antonio, TX

### Motions and Action Items

### As Recorded at the Meeting by Bill Buscher

- 1. Action Item TEI to perform another SEM analysis on a random bearing set from the TEI inventory to check for indications of lead leaching, and to compare to the original SEM analysis.
- 2. Action Item TMC to obtain demonstration data for proposed reference oil 705. At a minimum, TMC will request the test results and bearing batch.
- 3. Motion Restore VIII reference interval back to 15 runs or 6 months from 12 runs or 3 months. Effective November 14, 2007.

### Fred Gerhart / Charlie Leverett / Passed Unanimously with 1 Waive

4. Action Item – TEI will bring a few random bearing sets from the TEI inventory to Intertek, and TEI, Sid Clark and Charlie Leverett will inspect them under a high power microscope.

### Minutes of Sequence VIII Conference Call July 11, 2007

- Roll Call 8 voting members on the call. See Attachment 1
- 2. Motion and Action Items Recorder
- Preservation of Bearings
  - a. Fred reviewed his email see Attachment 2
  - b. Charlie has problems with new bearings already was 5 ppm is now 33 ppm
  - c. Motion made for TEI to clean and pentane wipe and repack the 0306 bearings into EF411 using new containers. TEI is to also inspect, clean, and repack the bearings into fresh EF411 on an annual basis. Passed
  - The repacked bearings will be identified as batch 0306 lot code 01A.
  - e. During discussion over differences measured in lead content between bearings stored at TEI vs. bearings stored at individual laboratories, the laboratories agreed to start storing their bearings in Argon gas.
- 4. Action Alarm TBWL Severity
  - TMC 704-1 and 1006-2 severities are not equivalent
  - TMC Lead levels do not seem to impact reference results
  - · TMC both labs have experienced severity shifts.
  - Statisticians want to wait and see while the panel works on bearing storage issues.
- 5. Restore Sequence VIII reference interval back to six months or 15 tests.
  - a. Targets have been obtained on 1006-2.
  - Three additional references should occur based on time before fall meeting
  - c. Motion made to change reference interval from 3 months or 8 runs to 3 months and 12 runs. Passed

### ASTM SEQUENCE VIII SURVEILLANCE PANEL ATTACHMENT 1 - VOTING MEMBERSHIP ATTENDANCE RECORD

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### Fred Gerhart

From: Fred Gerhart [fgerhart@swri.org] Sent:

Tuesday, July 10, 2007 8:36 PM

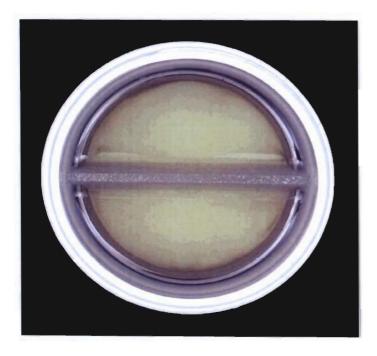
To: Andrew Ritchie; Bill Buscher; Charlie Leverett; Clayton Knight; Dave Glaenzer; Dwight Bowden; George Szappanos; Mark Sutherland; Rich Grundza, Sid Clark, Timothy Caudill; Timothy Miranda

Cc: Ben Weber; Frank Farber; Gordon Farnsworth; Irwin Goldblatt; Jim Carter; Jo Martinez; Johnny De La Zerda; Jon Carlson; Lew Williams; Martin Chadwick; Patrick M Lang; Phil Scinto; Robert Ofree;

William A. Buscher III; Zack Bishop

Subject: Proposal to change bearing storage oil back to EF-411

This set was 11-93 lot 12 changed to PAO8 at TEI on June 2005 per Surveillance Panel request. Please remember that TEI was told by the Surveillance panel to simply remove the bearings from the container of EF-411, allow the bearings to drain, and then place the bearings into a new container filled with PAO8. No cleaning of the bearings was permitted.



When I opened this container the PAO8 had a slight green tint and I saw the brown/tan color displacement at the bottom of the container like we have been occasionally observing on the 0306 bearings.

D5185 metals was Cu of 28 ppm and PB 190 ppm for the fluid immediately around the edge of the bearings at the bottom of the container.

I then swished and stirred the oil and took a homogenous sample from the same container. D5185 metals were Cu 26 ppm and Pb 161 ppm for the homogenous sample.

These bearings had been stored at our facility since the repacking date.

On June 28 of 2007, I started an experiment with a set of 0306 bearings that had been stored at our facility since completion of the prove out matrix. The bearings were removed from the container, identifying marks applied to the back of the bearing tangs, and wiped and cleaned as per the current procedure.

One bearing half was then placed into a 250 ml Pyrex beaker and enough new EF-411 added to cover the bearing completely. The beaker was then placed into a vacuum desiccator at 25 in hg vacuum. On July 7, 2007 this bearing was removed from EF-411 and the bearing half cleaned and weighed again. There was no change in mass.

The other bearing half was also placed into a 250 ml Pyrex beaker but without the benefit of any storage oil. The beaker was then placed into the same vacuum desiccator at 25 in hg vacuum. On July 7, 2007 this bearing was removed from the Pyrex beaker and cleaned and weighed again. The mass increased by 0.7 mg.

Based on this data and the fact that EF-411 has been shown in the past to protect the bearings for about 3 years before things start changing, SwRI is in favor of changing the bearing storage fluid back to EF-411 but only if the bearings are cleaned first with Pentane and then placed into new containers with EF-411 and the bearings are cleaned, inspected, and the EF-411 replaced with new EF-411 on an annual basis.

Thanks for your considerations and time.

### Fred Gerhart

Senior Research Technologist Fuels and Lubricants Research Division Southwest Research Institute

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Memorandum:

07-056

Date:

October 9, 2007

To:

Fred Gerhart, Chairman, Sequence VIII Surveillance Panel

From:

Richard E. Grundza

Subject:

Sequence VIII Semiannual Report: April 1, 2007 to September 30, 2007

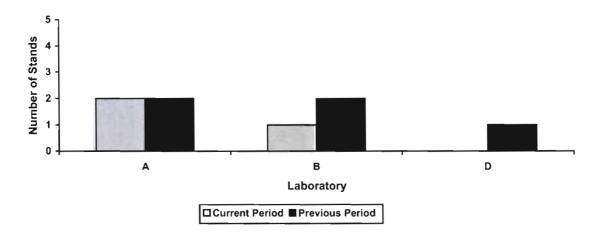
The following is a summary of Sequence VIII reference oil tests that were reported to the Test Monitoring Center during the period from April 1, 2007 to September 30, 2007.

### Lab/Stand Distribution

	Reporting Data	Calibrated as of September 30, 2007
Number of Laboratories:	2	2
Number of Stand/Engine Combinations:	3	2

The following chart shows the laboratory/stand distribution:

### Laboratory/Stand Distribution

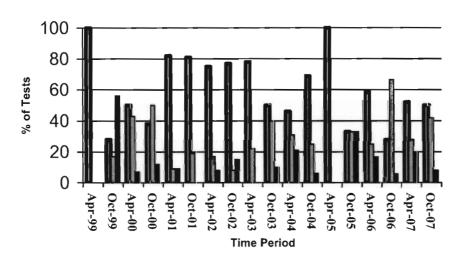


The following summarizes the status of the reference oil tests reported to the TMC:

Calibration Start Outcomes	TMC Validity Code	No. of Tests
Operationally and Statistically Acceptable	AC	6
Operationally Invalid (laboratory judgment)	LC	3
Aborted	XC	2
Statistically unacceptable Calibration Test	OC	1
Total		12

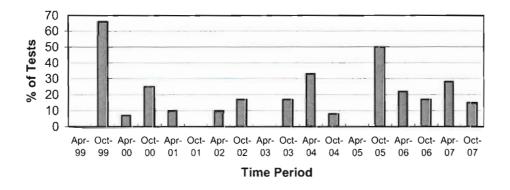
Calibrations per start, lost tests per start and rejection rates are summarized below:

### **Calibration Attempt Summary**



■ Calibrations/Starts
■ Lost Tests/Starts
■ Rejections/Starts

### **Rejected Operationally Valid Tests**



One test failed acceptance criteria during the period, for severe BWL.

There were no LTMS Deviations this period. There have been three deviations from the LTMS to date.

Two lab visits were conducted by the TMC this period.

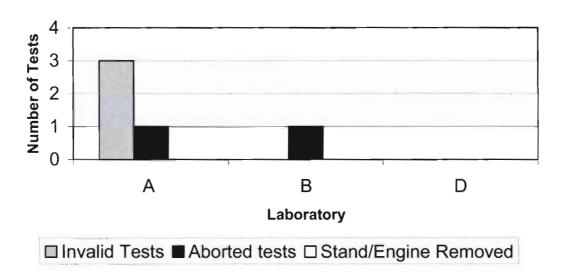
### Lost Test Summary

Five tests were lost this period. The reasons for the lost tests are tabulated below:

Reasons for Lost Test(s)	Number
High mechanical wear	3
Heater malfunction	1
High oil consumption	1

Aborts and operationally invalid tests, reported by laboratory, are summarized in the following chart:

### **Lost Test Distribution**



### Information Letters

No information letters were issued this period.

### Severity and Precision Analysis

Below is a summary of the average  $\Delta$ /s, pooled standard deviation, and average  $\Delta$  in reported units for the tests reported during this period. Also below is a summary of the average  $\Delta$ /s values for all laboratories reporting data during this period.

Industry Severity Summary					
Parameter	Average Δ/s	Pooled standard deviation (degrees of freedom)	Average Δ, in reported units		
BWL	1.079	3.039 (df=6)	3.28 mg		
SVIS	-0.143	0.086 (df=6)	-0.01 cSt		

Average ∆/s by Laboratory				
Lab	BWL	SVIS		
A	0.324	-0.952		
В	1.644	0.464		
D	-	-		

### Bearing Weight Loss (BWL)

The industry control charts for severity have been in warning or action alarm for the entire period. Precision control charts were in control for the period. (see Figure 1).

The Industry BWL mean  $\Delta$ /s is 1.079 severe for this report period (see Figure 3). This equates to a shift of 3.28 mg in reported units. The pooled standard deviation for the period is 3.039 mg (see Figure 4), which is essentially unchanged with respect to the previous period and compares well with historical estimates.

Figures 7 and 8 graphically illustrate the lead content, in ppm, versus test severity in delta/s. The highest concentration of lead reported this period with the 03-06 batch of bearings was 202 ppm.

### Stripped Viscosity (SVIS)

The industry control chart for severity began the period in warning alarm, but after the second test, the charts were within control limits for the remainder of the period. With the exception of a warning alarm at the end of the period, precision was in control for the period (see Figure 2).

The Industry SVIS mean  $\Delta$ /s is -0.143 severe for this report period (see Figures 2 & 5), and equates to a shift of -0.01 cSt in reported units. The pooled standard deviation for the period is 0.086 cSt (see Figure 6), which is comparable to historical performance.

### Hardware

All tests reported during this period were run on the 03-06 Batch bearings.

### TMC Memorandum

No TMC Memoranda were generated this report period.

### Reference Oils

Oil	TMC Inventory, TMC Inventory, Laboratory Inventory,		Laboratory Inventory,	Estimated Life	
	In gallons	In tests	in tests		
704-1	326	163	4	5+ years	
1006	43	21	1	3 months <sup>1</sup>	
1006-2	4,492	2,246	5	3+ years <sup>1</sup>	
1009	669	334	5	3+ years <sup>1</sup>	

<sup>&</sup>lt;sup>1</sup> Multiple test area reference oil; total TMC inventory shown

### REG/reg

### Attachments

c: F. M. Farber, TMC

Sequence VIII Surveillance Panel

ftp://ftp.astmtmc.cmu.edu/docs/gas/sequenceviii/semiannualreports/VIII-10-2007.pdf

Distribution: Electronic Mail

### List of Figures

- Figure 1 graphically presents the Industry control charts for BWL and also the CUSUM delta/s plot (by count in completion date order) of bearing weight loss for operationally valid tests.
- Figure 2 graphically presents the Industry control charts for SVIS and also the CUSUM delta/s plot (by count in completion date order) of bearing weight loss for operationally valid tests.
- Figure 3 graphically presents a historic perspective for BWL mean delta/s by report period.
- Figure 4 graphically presents a historic perspective for BWL pooled standard deviations by report period.
- Figure 5 graphically presents a historic perspective for SVIS mean delta/s by report period.
- Figure 6 graphically presents a historic perspective for SVIS pooled standard deviations by report period.
- Figure 7 graphically presents a comparison of Total Bearing Weight Loss (Delta/s) vs. the amount of lead content, in ppm, in the bearing storage oil.
- Figure 8 graphically presents the amount of lead content, in ppm, in the bearing storage oil by completion date order (Sequence VIII and L-38 data combined).
- Figure 9 is the Sequence VIII Timeline, created to track changes in test hardware and operations.

Figure 1

### SEQUENCE VIII INDUSTRY OPERATIONALLY VALID DATA

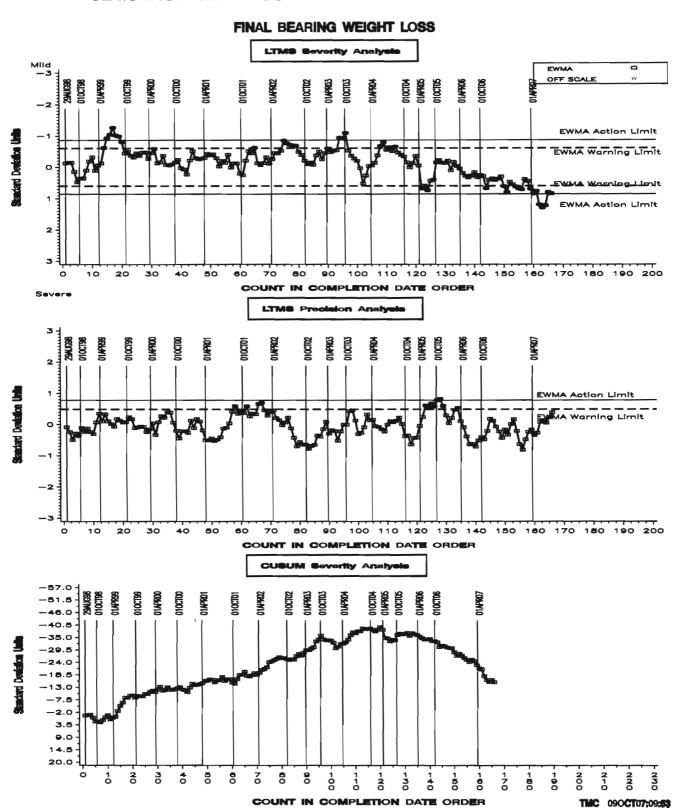


Figure 2

### SEQUENCE VIII INDUSTRY OPERATIONALLY VALID DATA

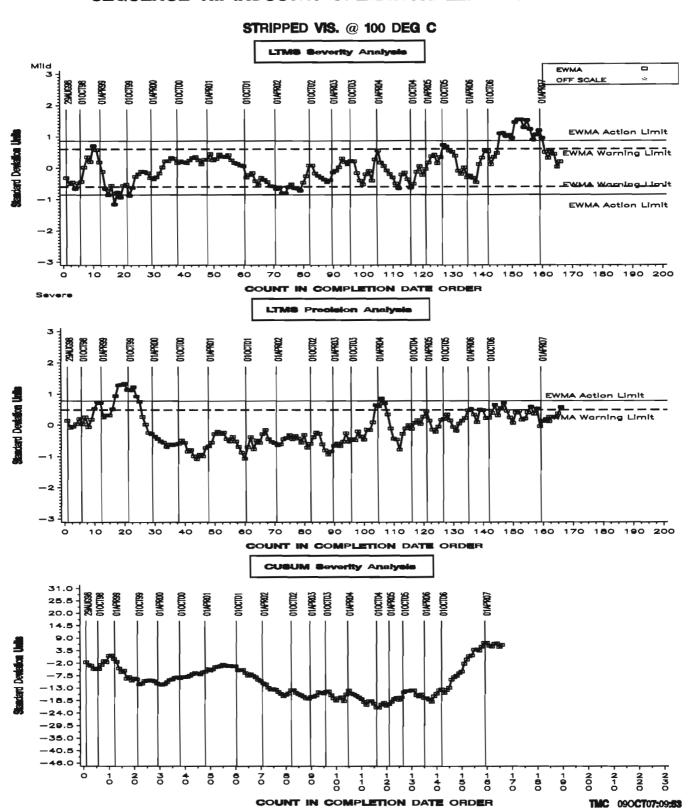


Figure 3 - Sequence VIII Reference Oil Data Bearing Weight Loss

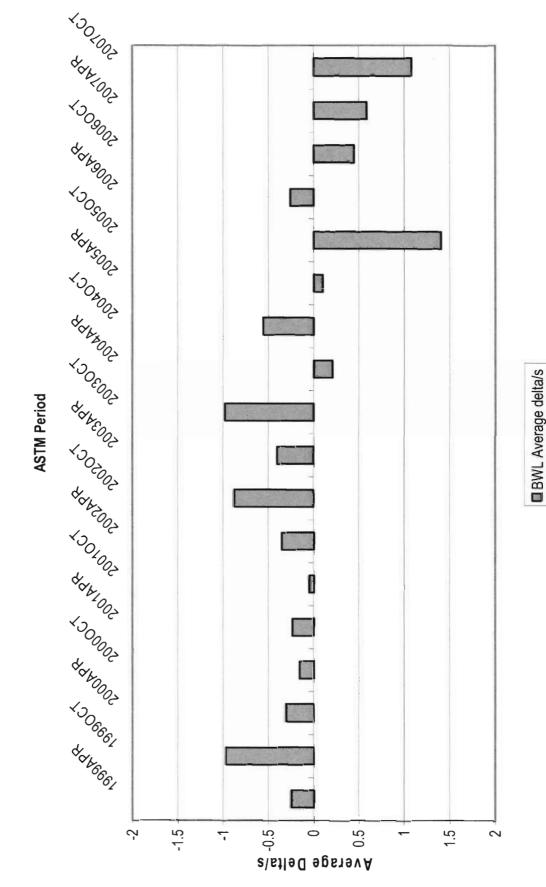


Figure 4 - Sequence VIII Reference Oil Data Bearing Weight Loss

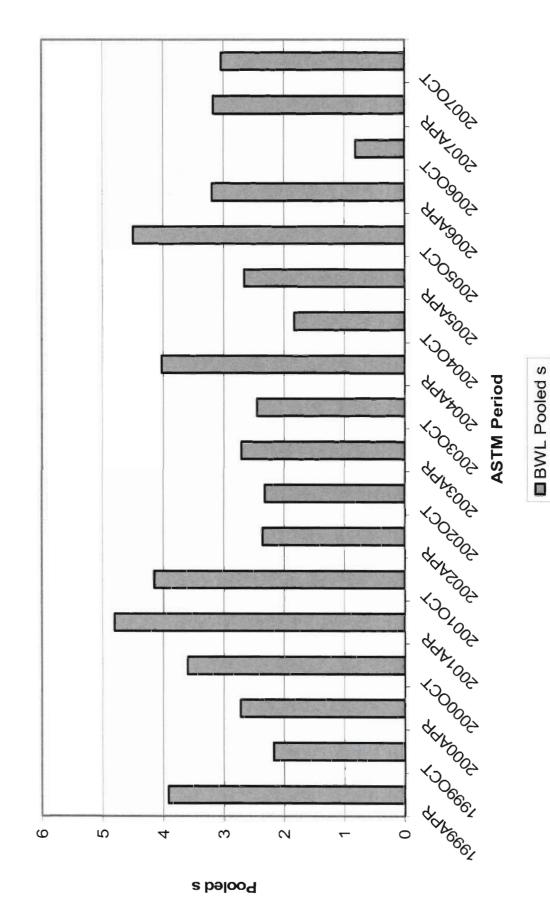


Figure 5 - Sequence VIII Reference Oil Data Stripped Viscosity

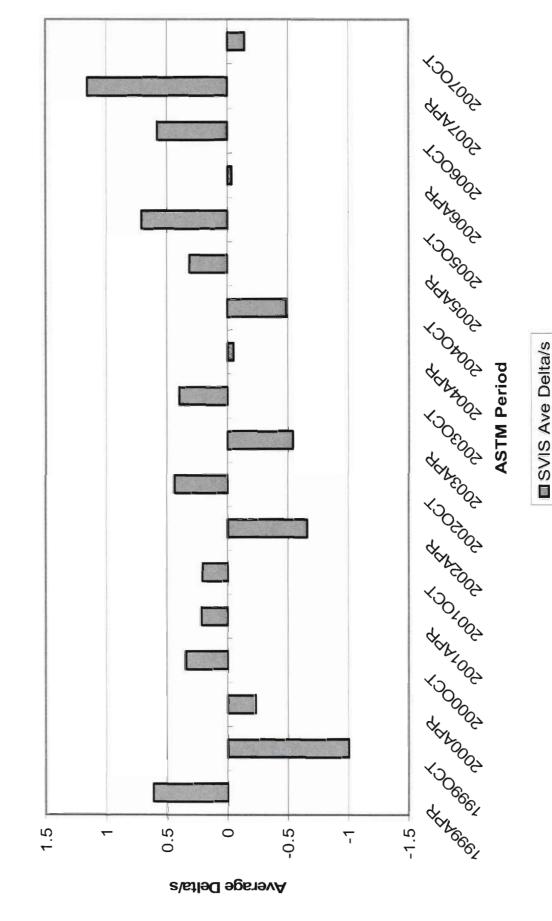
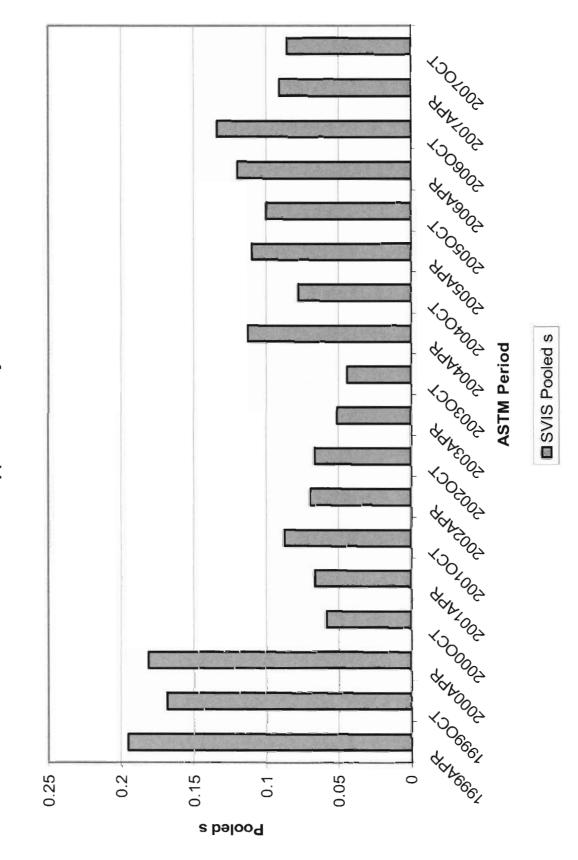


Figure 6 - Sequence VIII Reference Oil Data Stripped Viscosity

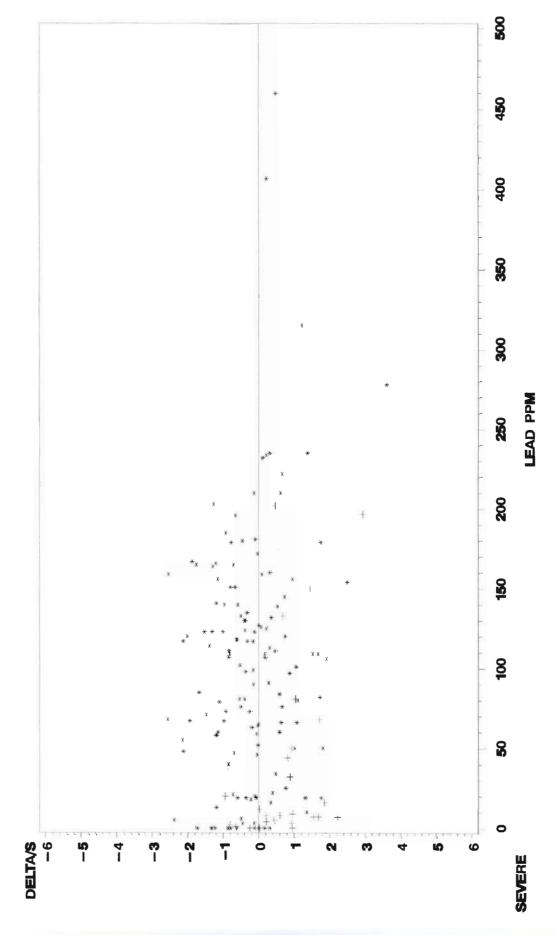


Memo 07-056 Page 13

Figure 7

M

## SEQUENCE VIII BWL DELTA/S VS LEAD PPM



(") BEARING BATCH 11-93 (+) BEARING BATCH 03-06

Memo 07-056 Page 14

Figure 8

# BEARING OIL STORAGE LEAD PPM VS COMPLETION DATE

All LTMS Data

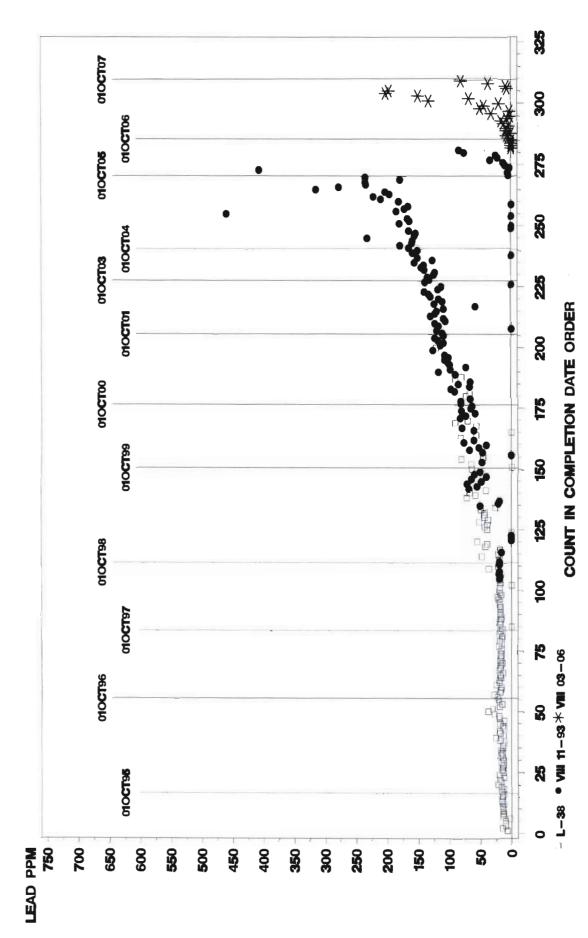


	Figure 9 - Sequence VIII Timeline				
Date	Topic	Information Letter			
2/10/1999	NEW PISTON RING BATCH APPROVED FOR USE IN SEQUENCE VIII TESTING	00-1			
4/16/1999	DRAFT 3.1 OF THE SEQUENCE VIII TEST PROCEDURE ISSUED	99-1			
5/19/1999	REMOVAL OF RING BATCH REPORTING REQUIREMENTS	00-1			
5/19/1999	NEW OIL FILTER (RAYCOR LFS-62) IMPLEMENTED INTO TESTING	00-1			
11/16/1999	TEST ENGINEERING INC. NEW TEST PARTS SUPPLIER	00-1			
1/28/2000	PISTON CLEANING PROCEDURE FOR REUSING PISTONS IN SEQUENCE VIII TESTING	00-1			
6/15/2002	REVISED STAY-IN-GRADE PROCEDURE IMPLEMENTED	02-1			
11/18/2002	EDITORIAL REVISIONS TO D6709-01	02-2			
1/1/2004	NEWINERAL SPIRITS SPECIFICATION	03-1			
1/26/04	BILLET CRANKSHAFT APPROVED FOR USE IN SEQUENCE VIII TESTING				
12/9/2004	CLARIFIED SOLVENT SPECIFICATION	04-1			
12/9/2004	REVISED FUEL FLOW SPECIFICATION	04-1			
12/9/2004	REQUIREMENTS FOR BUILDS WITH OVERSIZE PISTONS	04-1			
6/23/05	DELETED ROCKER COVER INLET TEMPERATURE AND PRESSURE SENSORS, UPDATED PRECISION STATEMENT	05-1			
9/20/06	FIRST TEST ON 03-06 BEARINGS				
10/24/06	REVISED BEARING CLEANING PROCEDURE IN ANNEX A9	06-1			
3/12/07	TARGET UPDATE, REFERENCE OIL 1006-2				

03-06 Batch of Rod Bearings

Part number 1000341

Number of bearings produced 5,098 (2,549 sets)

TEI currently has 2,289 sets stored in Argon (10+ year supply)



## Analysis of rod bearing 03-06 storage oil

Sample date	<u>late</u>	Lead (ppm)
11/01/06	(PAO 1)	12
08/02/07	(PAO 1)	86
11/12/07	(PAO 1)	158
08/02/07	(PAO 2)	113
11/12/07	(PAO 2)	190
08/02/07	(EF-411 Lot 15)	12
11/12/07	(EF-411 Lot 15)	18



Storage Oil Changeover Request (8-22-2007 email to TEL from S.P.)

Replace PAO with EF-411.

Project is in progress – estimate completion by mid February 2008.

Bearings will be cleaned per procedure outlined by the surveillance panel (see next slide).



## Surveillance Panel Recommended Cleaning Procedure

- scratching the bearing surface. Always use latex gloves and protective tongs when handling bearings (note: TEI will also wear cotton gloves). 1.) Exercise care when handling the bearings to prevent nicking or
- Soak bearing halves in a container of mineral spirits for a minimum of 5 minutes to remove all traces of PAO oil from both the front and back sides of the bearing. 7
- Dip the bearing halves in pentane and allow to air dry.
- Wipe bearings with a soft paper towel soaked in pentane (note: TEI will use a soft cotton cloth in lieu of paper).
- Place bearing halves into new containers filled with EF-411 and store in Argon cabinet. 5.



## **Annual Parts Order**

Annual orders for 2007 have been completed.

 TEI will issue a letter in the next few weeks soliciting lab orders for 2008 annual parts requirements.



CG190 (Rev. AUG/2003)





### **GM Evaluation Report**

Page 1 of 6

1348-01 Mechanical

accreditation.

107	0-02 Officiality						
Platform	Project/EWO No.	Requirement No.	Procedure No.	VPP/VIA/UPC	Model Year	Model No.	PER/Report No.
Engine 3.8 V6 SC	N/A	N/A	N/A	N/A	N/A	na	24729

Reference Issue Report (IR) No. NA	Category MAT
	Function <b>x</b> Development Validation  Method: Math Based <b>x</b> Hardware Based
Date: 04/20/2007	
Title: Report for STR # 24729	☐ Interim No.
	Reissue Date of Reissue:
To: (Requestor) Sidney L. Clark	Date of Request: 04/11/2007
From: Patrick M. Hanley	Dates of Evaluation: 04/11/2007 - 04/19/2007
<ul> <li>The results published within this report relate o</li> <li>This report shall not be reproduced except in full laboratory manager or his/her designate.</li> </ul>	

**Objective:** Characterize Cu/Pb/Sn bearings used for oil evaluation sequence testing. Verify bearing composition, look for contaminants, other potential impurities on surface, check metal content in PAO storage fluid by ICP to

Tests identified with an asterisk are not included in the laboratory's current A2LA scope of

**Conclusions:** A lead/tin phase was detected in the copper matrix of a sintered powdered metal copper-lead-tin alloy from the bearing surface into the bearing cross section by Energy Dispersive Spectroscopy (EDS). Evidence of lead leaching from the bearing surface was not observed. Polyalphaolefins from bearing storage jars are free of elemental impurities, there is no evidence of lead, tin, or copper leaching from the bearings. Bearing surface composition is about 78%Cu, 20%Pb, 2%Sn. White flake-like films/floaties found in PAO storage media were identified as polyethylene/vinyl acetate copolymer, mostly polyethylene.

Based on the results of our analyses the bearings are in their original condition, we found no evidence of degradation.

Design Evaluated: GMUTS Rating: n/a Veh. Mileage:

determine if any evidence of Pb, identify floaties/flakes (possibly jar top insulation/gasket).

	omo i o i tattingi. i ii a	3
Part/Test Object Name(s)	Veh/Buck/PT/Part No./RPO	Revision Date/Level
Engine - Bearings	npn	n/a
Material	Vehicle/Engine/Transmission Number	er
Cu/Pb/Sn 80/10/10 - FederalMogul	3800	

Cu/Pb/Sil 60/10/10 - Federa	aliviogui	3000
Distribution:		
Name: Sidney L. Clark Name: Name: Name: Name: cc.	Loc: 480-106-160 Loc: Loc: Loc: Loc:	

Author: Patrick M. Hanley (Title/Phone) Lead Analyst / (248) 753 (Location) GMNA Materials Lab - Pon	
Peer Review: Suzette Kandow Test Engineer/248-753-6 GMNA Materials Lab - F	
Approver: Patrick M. Hanley Lead Engineer, Chemistry/2 GMNA Materials Lab - Pont	





1348-01 Mechanical

Platform	form Project/EWO No. Requirement No. Procedure		Procedure No.	VPP/VIA/UPC	Model Year	Model No.	PER/Report No.	
Engine 3.8 V6 SC	N/A	N/A	N/A	N/A	N/A	na	24729	

Reference Issue Report (IR) No.

NA

Category

MAT

Function

x Development

Validation

Method:

Math Based

Hardware Based

Recommendations: Not provided. Requirements: Not provided

### Background:

Sample Condition as received: New bearings in PAO fluid, plastic separator?, orange/rust colored bearing side in plastic jar

Sample History: Experimental Test bearings, made in Mexico, suspect Pb leach on storage, bunch of

bearings are made for testing, on storage characteristics appear to change

Unusual Circumstances: New storage PAO versus mineral, white floaties/flakes

Previous Performance: None noted.

### Assumptions/Limitations:

-Sample homogeneity (i.e. sample sufficiently homogeneous and representative of material/part).

-For Semi-quantitative X-ray Fluorescence, the concentrations reported were obtained with a standardless semiquantitative x-ray fluorescence analysis program, Uniquant. They are estimated normalized concentrations (to 100%) based on elements detected. The results do not include low atomic number elements (H, He, Li, Be, B, C, N, or O), which are not detected with this method, and may or may not comprise a substantial portion of the material analyzed (e.g. as oxides, hydrates, C based etc.).

-Results are semi-quantitative/qualitative, estimated uncertainty is +/- 1/3 to 3 times the amount reported for representative specimen analyzed.

See results text for additional assumptions/limitations.

### Equipment:

Requestor Defined Chemistry Testing (List In Detailed Test Instructions)\* was accomplished utilizing Personal Computer.

Sample Preparation (Chemistry)\* was accomplished utilizing Mettler PR2003 Balance (ME000357).

Semi-Quantitative X-ray Fluorescence (UniQuant)\* was accomplished utilizing ARL 9400 X-Ray Fluorescence Spectrometer (XRF) (ME000289).

(ASTM D5185-05) Additives/Wear metals by ICP was accomplished utilizing Thermo Electron - IRIS Intrepid II XDL ICP-AES (ME000856).

FTIR Analysis of Oil\* was accomplished utilizing PE Spectrum BX-II FTIR Spectrometer (ME000251). Sample Preparation (Oil Chemistry)\* was accomplished utilizing Marvel Series 8 Bandsaw 824282-W (ME000390). (WI-6001) EDS Analysis (Energy Dispersive Spectroscopy) was accomplished utilizing Jeol 5900 Scanning Electron Microscope with a Noran Vantage 1.5 Digital Analyzer (ME000054).

### Procedure/Method:

Requestor Defined Chemistry Testing (List In Detailed Test Instructions)\* was done by Patrick M. Hanley.

Sample Preparation (Chemistry)\* was done by Patrick M. Hanley.

Semi Quantitative X-ray Fluorescence (UniQuant)\* was done by Patrick M. Hanley.

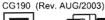
(ASTM D5185-05) Additives/Wear metals by ICP was done by Patrick M. Hanley.

FTIR Analysis of Oil\* was done by Patrick M. Hanley.

Sample Preparation (Oil Chemistry)\* was done by Patrick M. Hanley.

(WI-6001) EDS Analysis (Energy Dispersive Spectroscopy) was done by Suzette Kandow.

Detailed Test Instructions: Verify bearing composition, look for contaminants, other stuff on surface, check metal content in PAO storage fluid by ICP, identify floaties/flakes (possibly jar top insulation/gasket).







1348-01 Mechanical

134	0-02 Chemicai							
Platform	m Project/EWO No. Requirement No. Procedu		Requirement No. Procedure No. VPP/VIA/UPC Model Year		Model Year Model No.		PER/Report No.	
Engine 3.8 V6	N/A	N/A	N/A	N/A	N/A	na	24729	
SC								

Reference Issue Report (IR) No.

NA

Category

~

MAT

x Development

Validation

Function Method:

Math Based

x Hardware Based

NOTE: The specific procedure followed for each test is that described in the relevant GMNA Materials Laboratory test instruction. When standard test methods have been modified, it is labeled as "mod" or "modified" in the test list and the specific nature of such modifications is stated in the test instructions.

### Results:

(WI-6001) EDS Analysis (Energy Dispersive Spectroscopy)

STR 24729 Date: 04/19/2007

### Conclusions based on EDS Analysis:

A lead/tin phase was detected in the copper matrix of a sintered powdered metal copper-lead-tin alloy from the bearing surface into the bearing cross section by Energy Dispersive Spectroscopy (EDS). Evidence of lead leaching from the bearing surface was not observed.

### **Assumptions / Limitations:**

- Homogeneity of the sample relative to sample size and area designated for analysis.
- SEM/EDS verification of resolution is performed quarterly.
- CAUTION: Due to the type of analysis technique employed, these values are relative in nature and limited to
  their degree of accuracy and statistical validity. Energy Dispersive Spectroscopy (EDS) semi-quantitative
  (standardless) results may not approximate those found by other conventional quantitative analysis techniques
  used in bulk material analysis.
- EDS semi-quantitative analysis of the sample was performed using both a polished metallographic mount (bearing cross section) and the as submitted sample (bearing surface).
- Any micron markers in photos denote feature size relationships. Magnification values listed for the photos are
  for reference only and any use related to feature size relationships is not recommended due to photo size
  adjustments made for publication purposes.
- Any cutting or other machining that occurred as part of sample preparation prior to evaluation was done in such a manner as to have no effect on the microstructure present.

### Results:

Scanning Electron Microscopy with Backscatter Imaging (BEI) and Energy Dispersive Spectroscropy (EDS X-ray)

Qualitative and Semi -Quantitative Analysis

A polished metallographic mount containing a cross section of a copper alloy running surface clad to a steel backing and a piece from the same bearing was submitted to the SEM area to determine if lead is leaching from the bearing surface.

The metallographic mount was re-polished with 1 micron diamond followed by a 0.1 micron alumina, rinsed in water then dried with methanol. It was then ultrasonically cleaned in hexane, dried and attached to an aluminum holder via a copper grounding strap and inserted in to the SEM. Scanning electron microscopy using backscatter electron imaging techniques to display atomic number contrast of the copper, lead and tin elements was conducted at 25kv acceleration voltage and various magnifications. Energy dispersive spectroscopy (EDS) x-ray and semi-





1348-01 Mechanical 1348-02 Chemical

Platform	Project/EWO No.	Requirement No.	Procedure No.	VPP/VIA/UPC	Model Year	Model No.	PER/Report No.
Engine 3.8 V6 SC	N/A	N/A	N/A	N/A	N/A	na	24729

Reference Issue Report (IR) No.

NA

Category

Function

Development

Validation

Method:

Math Based

¥ Hardware Based

quantitative analysis was performed at this same acceleration voltage. The cross section of the bearing displays evidence of a lead/tin phase in the copper matrix of a sintered powdered metal copper-lead-tin alloy from the running surface into the cross section. Figure A depicts the BEI image at the left and the corresponding x-ray composite map to the right. In the BEI photo copper is displayed as the dark gray constituent in the running surface and the white areas are the lead/tin rich sites. The dark solid gray area to the right is the steel backing. Figure A1 is a representative EDS scan of one of the fields of view used for semi-quantitative analysis that is summarized in Table A.

A piece from the same bearing was ultrasonically cleaned in hexane, dried and attached to an aluminum holder and inserted in to the SEM. The same imaging and EDS techniques used on the mount were used on the as submitted bearing surface. The bearing surface displays evidence of a lead/tin phase in the copper matrix of a sintered powdered metal copper-lead-tin alloy. Figure B depicts the BEI image at the left and the corresponding xray composite map to the right. In the BEI photo copper is displayed as the dark gray constituent and the white areas are the lead/tin rich sites. Figure B1 is a representative EDS scan of one of the fields of view used for semiquantitative analysis that is summarized in Table B.

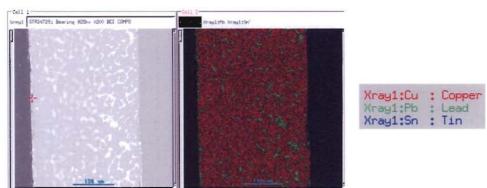


Figure A: Bearing Cross Section in Mount - BEI COMPOSITION and X-Ray Composite Map @25kv X200 (The bearing surface is at the left edge (red cross cursor).)

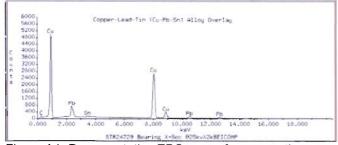


Figure A1: Representative EDS scan of cross section.

Table A: Cross Section	Semi-Quantit	tative Analysis	by EDS met	no			
Semi-Quantitative	Element						
POINT	Pb Sn Cu						
<unitless></unitless>	Wt.%	Wt.%	Wt.%				
Average	18.1	1.4	80.5				
Min	13.3	1.1	77.4				
Max	21	1.6	85.2				
			,	-			

Average of 5 fields of view





1348-01 Mechanical

	6-02 Chemical						
Platform	Project/EWO No.	Requirement No.	Procedure No.	Procedure No. VPP/VIA/UPC Model Year		Model No.	PER/Report No.
Engine 3.8 V6	N/A	N/A	N/A	N/A	N/A	na	24729
SC							

Reference Issue Report (IR) No.

NA

Category MAT

Function

x Development

Validation

Method:

Math Based

Hardware Based

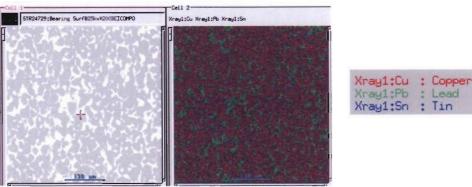


Figure B: As Submitted Bearing Surface – BEI COMPOSITION and X-Ray Composite Map @25kv X200 (The red cross cursor denotes a lead/tin rich area near a copper area.)

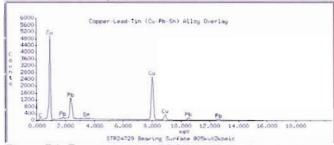


Figure B1: Representative EDS scan of surface.

Table B: Surface Semi-Quantitative Analysis by EDS method									
Semi-Quantitative		Element							
POINT Pb Sn Cu									
<unitless></unitless>	Wt.%	Wt.%	Wt.%						
Average	37.5	1.1	61.4						
Min	21.9	1	51.7						
Max	47.2	1.5	76.6						
Average of 5 fields of vie	Average of 5 fields of viewy								

### (ASTM D5185-05) Additives/Wear metals by ICP

PAO's from bearing storage jars are free of elemental impurities. There is no evidence of lead, tin, or copper leaching.

	ICP (ASTM all units		g (mag)						
SAMPLE	P	Ca	Zn	Ba	Mg	Na	K	S	В
24729-1	<20	<10	<1	<5	<10	<10	<10	178	<2
24729-2	<20	<10	<1	<5	<10	<10	<10	209	<2

SAMPLE	Fe	Al	Si	Cu	Pb	Mn	Ni	Cr	Mo	Sn
24729-1	<5	<3	<5	<5	<3	<5	<2	<2	<5	<5
24729-2	<5	<3	<5	<5	<3	<5	<2	<2	<5	<5

### Assumptions/Limitations:

- Fluid (oil/atf) is homogeneous,
- Elements reported are soluble,
- Particle size effects are assumed to be zero. Accuracy decreases as particle size increases. <1micron = no effect, 1-5microns = minimal effect, >5microns = large effect.





1348-01 Mechanical

1348-02 Chemical								
Platform	Project/EWO No.	Requirement No.	Procedure No.	VPP/VIA/UPC	Model Year	Model No.	PER/Report No.	
Engine 3.8 V6 SC	N/A	N/A	N/A	N/A	N/A	na	24729	

Reference Issue Report (IR) No.

NA

Category

MAT

Function Method:

x Development

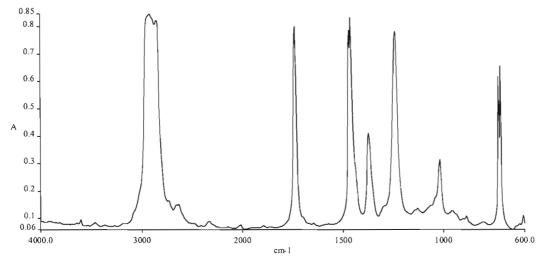
Validation

Math Based

Hardware Based

### FTIR Analysis of Oil\*

Infrared Analysis - Identification of white flake-like floaters in PAO bearing storage oil.



### STR24729

Infrared spectrum of white flake-like films/floaties found in PAO storage media. Spectrum indicates white flake-like floaties are polyethylene/vinyl acetate copolymer, mostly polyethylene. Spectrum taken as thin film on ZnTe cystal.

### Semi Quantitative X-ray Fluorescence (UniQuant)\*

Bearing surface composition was found to be 78%Cu, 20%Pb, 2%Sn (+/-1/3 to 3 times amount reported), based on examination of and average over a 15mm diameter area of the bearing

### References/File Storage:

Original reports are stored in the Engineering Document Management database.

All raw data files are stored in the laboratory or the Global Product Development Information Center (GPDIC) and can be made available upon request.

Measurement uncertainty values are available upon request.

### **Calibration Information:**

All relevant calibration reports can be made available upon request.

HALTERMANN

November,

2007

**FUEL REPORT** 

PRODUCT:

**KA24E TEST FUEL** 

PRODUCT CODE:

HF008

Seq. IV & VIII

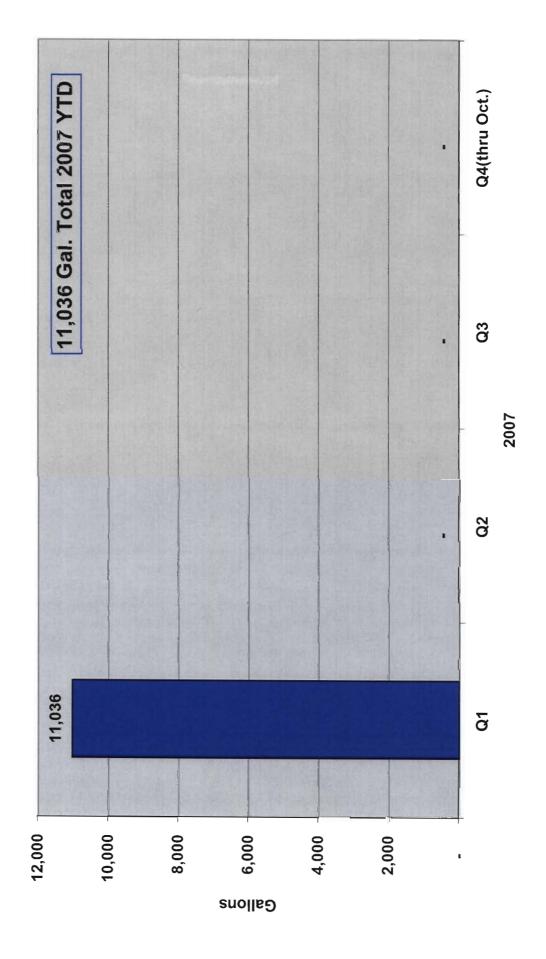
TMO No.: MTS MTS
Tank No.: TK58 676

**Batch No.:** VJ1121GP01 VC0921LS03

Analysis Date: 10/29/2007 3/15/2007

TEST	METHOD	UNITS	SPECIFICATIONS			RESULTS	RESULTS
			MIN	TARGET	MAX		
Distillation - IBP	ASTM D86	°F	75		95	91.6	85
5%		°F				114.1	112
10%		°F	120		135	126.3	125
20%		°F				147.9	147
30%	]	°F				174.2	173
40%		°F				203.9	203
50%		°F	200		230	221.4	222
60%		°F				230.7	231
70%		°F				239.4	241
80%		°F				254.8	260
90%		°F	300		325	313.9	321
95%		°F				340.5	345
Distillation - EP		°F	385		415	398.3	400
Recovery		vol %		Report		98.0	97.6
Residue		vol %		Report		0.9	1.0
Loss	]	vol %		Report		1.1	1.4
Gravity	ASTM D4052	°API	58.7		61.2	59.7	59.9
Density	ASTM D4052	kg/l	0.734		0.744	0.7398	0.7393
Reid Vapor Pressure	ASTM D323	psi	8.8		9.2	9.1	9.1
Carbon	ASTM E191	wt fraction	0.8580		0.8667	0.8622	0.8651
Carbon	ASTM D3343	wt fraction		Report		0.8646	0.8643
Sulfur	ASTM D4294	wt %	0.01		0.04	0.0233	0.016
Lead	ASTM D3237	g/gal			0.05	< 0.01	< 0.01
Phosphorous	ASTM D3231	g/gal			0.005		
Oxygen	ASTM D4815	wt %			0.05	< 0.05	< 0.05
Composition, aromatics	ASTM D1319	vol %			35.0	28.1	28.0
Composition, olefins	ASTM D1319	vol %	5.0		10.0	5.8	7.0
Composition, saturates	ASTM D1319	vol %		Report		66.1	64.2
Oxidation Stability	ASTM D525	minutes	1440			>1440	>1440
Copper Corrosion	ASTM D130				1	1	1
Gum content, washed	ASTM D381	mg/100ml			5	< 0.5	<1
Research Octane Number	ASTM D2699		96.0		97.5	97.0	97.1
Motor Octane Number	ASTM D2700			Report		88.2	87.0
R+M/2	D2699/2700			Report		92.6	92.1
Sensitivity	D2699/2700		7.5			8.8	10.1
Net Heat of Combustion	ASTM D240	btu/lb		Report		18342	18458
Color	Visual			Green		Green	Green

KA24E, HF-0008 Seq. IV & VIII



### SEQUENCE VIII SURVEILLANCE PANEL SCOPE AND OBJECTIVES

### **SCOPE**

The Sequence VIII Surveillance Panel is responsible for the surveillance of the Sequence VIII test procedure (ASTM D 6709-01). This panel works in conjunction with Test Engineering Inc. (TEI) who supplies the test hardware. Improvements in the test operation, test monitoring, and test validation will be accomplished through continual communications with the test hardware supplier, the ASTM Test Monitoring Center, the Technical Guidance Committee, and the ACC Monitoring Agency.

The panel will maintain an up-to-date evaluation of the precision of the VIII reference oils and will report this precision and test severity levels to D02.BO.01 Oil Classification Panel and section.

These combined efforts will help to assure that the Sequence VIII test method will continue to provide the industry with a precise method for evaluating a lubricant's ability to protect against copper-lead bearing weight loss and to evaluate the viscosity stability of multi-viscosity-grade oils.

### **OBJECTIVES**

Continue monitoring of lead leaching rate in storage oil
Request reference oils with performance close Ongoing to 26 mg TBWL
Continue to look at improved bearing storage Ongoing methods
Monitor the progress of the comprehensive review of the different LTMS systems utilized by the PCMO test types and their pros and cons