[Date RR # approved – ASTM to assign]

Committee D02 on Petroleum Products and Lubricants
Subcommittee B on Automotive Engine Oil

Research Report [RR # – ASTM to assign]

The Development of ASTM D7320, Addendum X3, Seq. IIIGB for Phosphorus Retention Measurement

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1. Introduction:

Three-way catalysts have been used in passenger cars to control various exhaust emissions since the 1970s. Phosphorus (P) in engine oils has long been identified to affect the durability of such catalysts. A chemical limit has been in place to restrict the amount of P in engine oils in past engine oil categories. However, chemical limits do not reflect the total mechanism on how P in engine oil affects catalyst durability. Further reduction of P limit also raises wear concerns especially around flat tappet type cam followers. While chemical limits do address the impact of consumed oil on catalyst durability they fail to address the equally significant contribution from volatilized P. A recent field test evaluation clearly indicates that controlling P volatility can substantially improve long term three-way-catalyst durability[1,2]. In that study a GF-4 engine oil containing conventional ZDP was shown to cause greater catalyst degradation over a 100,000 field trail compared to an identical oil using a reduced volatility ZDP. A task force, Emissions System Compatibility Investigation Team (ESCIT), was formed under ILSAC/Oil[3] to develop a test procedure best capable of measuring the contribution of P in engine oils to catalyst degradation. More specifically, ESCIT was charged to find a test that evaluates the amount of volatile P emitted from engine oil while maintaining the correlation with the field performance.

2. Test Method:

A literature review was conducted in the beginning of the activity with the detailed list provided in **Appendix A**. A multitude of tests were proposed during the process. They primarily fall into 3 categories:

1. A dyno test that directly measures catalyst performance

2. A test utilizing used oils generated from an existing lubricant engine dynamometer test

3. A bench test

The primary consideration was given to the technical merits of each proposed test but resource restrictions, especially the need to fit into overall GF-5 timeline, were also a major factor. For example, the dynamometer test under development by Southwest Research Institute was considered very promising and most realistic but it did not progress sufficiently to be considered when the selection was made. The Sequence IIIG test (ASTM D7320) was found most suitable to provide the oil aging process and the Phosphorus Retention (PR) calculation was selected (The higher the PR, the lower the amount of volatile P emitting) as defined by:

$$PR = \frac{Ca_{tI}}{Ca_{t100}} x \frac{P_{t100}}{P_{tI}} x100\% \qquad [1]$$

Where Ca is Calcium concentration in the oil and P is Phosphorus concentration of oil, both in ppm. If Ca is not the highest non-volatile detergent metal in the oil, other non-volatile detergent metals can be used in its place. The designations “tI” and “t100” refer to the used oil samples taken at the beginning of the test (Initial) and at the end (100 hours) of the IIIG test. The 100-hr test duration was chosen over shorter hours (such as 20 and 40 hrs proposed in ESCIT) is to make sure as much volatile P is driven out of the oil as possible. The test is named IIIGB as it utilizes the used oil samples generated from Seq. IIIG. Other tests evaluated during the process are listed in **Appendix B** with limited descriptions. The final recommendation was made by ESCIT via 2 letters to ILSAC/Oil chair based on ESCIT final votes (**Appendix C**). PR measures the amount of volatile P retained after an aging period. It is compared to a baseline using Ca as a non-volatile component in the engine oil to reflect the liquid portion of the oil consumption. That is, PR will be 100% if the only P loss is via liquid oil consumption. The more volatile P retained in the oil (higher PR), the less volatile P emitted to the exhaust to affect catalyst performance. The P contribution to catalyst via liquid oil consumption is addressed through the chemical limit. This test ranks the improved oil (low impact) and the standard performance oil used in the field test in the correct order.

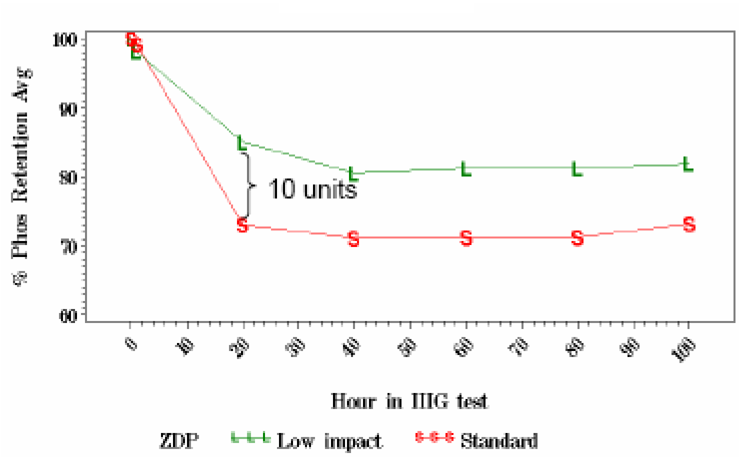

Figure 1 Smoothed average IIIG PR data for the two field test oils

JAMA[4] performed catalyst evaluation in a dynamometer test and confirmed the correlation achieved via IIIG as well. This result is in **Appendix D**.

In addition to the variability of IIIG itself, another source of error was suspected around the measurement procedure of Ca and P in the used oil sample. The following requirements were added by the Surveillance Panel to improve IIIG PR precision:

- The phosphorus and calcium weight percentages are to be determined using ASTM D 5185.
- All samples are to be run sequentially, in duplicate, using the same calibration (i. e. as close in time as practical)

- Background correction, internal standard, and peristaltic pump are required
- Sample dilutions of at least 1:20 are required
- Report the average of the two duplicate determinations

As a result, the IIIGB is basically a test taking used oil samples generated from IIIG and measures the samples with an improved used oil analysis procedure followed by the calculation in equation [1].

3. IIIGB Estimated Precision:

Given the IIIG test is an existing test procedure under ASTM Test Monitoring Agency (TMC) monitoring, reference oil data, including used oil results, are already available. Because the existing data were not produced with the final used oil analysis procedure, the precision thus produced could only be used as a preliminary precision. The estimated precision (**Table 1**) is still valuable as it allows assessment whether this test can discriminate between reference oils. TMC provided the IIIG reference oil data from 5 Labs (A, B, D, G, F), all running referenced and monitored IIIG stands. The data are listed in **Appendix E** with PR calculated in the last column. Note the units for Ca is ppm but "%" for P and PR. The three IIIG reference oils cover the range for the two field test oils. It is clear that IIIG PR can discriminate between the two field test oils. The IIIG PR can also discriminate among the three TMC IIIG reference oils (434, 435 and 438). This analysis includes all reported data. The added measures to improve used oil analysis precision will only improve the test precision and discrimination of IIIGB.

Table 1 IIIG PR precision used as the preliminary precision for IIIGB

Reference Oil	Mean	Standard Deviation
tmc-434	74.66	2.25
tmc-435	81.44	2.28
tmc-438	76.96	1.79

It is therefore considered sufficient to implement IIIGB. The addendum X.3 balloted successfully as a TMC information letter via ASTM D02.B is attached in **Appendix F** (TMC Sequence IIIG Information Letter 08-2, Sequence No. 18, November 6, 2008).

4. Precision and Bias Statement:

There was no established precision or bias statement when the final procedure was balloted. The preliminary precision was established using the IIIG PR reference oil data as described in **3**. That is, the statement in **Table 2** does not incorporate the use of modifications to the used oil analysis though the precision is expected to get better than that indicated in **Table 2**. The final precision statement will be established by the Surveillance Panel and documented via an information letter when sufficient reference oil data are produced using the final IIIGB procedure.

Table 2. Preliminary Sequence IIIGB Reference Oil Precision Statisticis[A]

	Intermediate Precision		Reproducibility	
Variable	$s_{i.p.}$ [B]	**i.p.**	s_R [B]	**R**
Phosphorus Retention, %	**2.07**	**5.741**	**2.11**	**5.845**

[A] Based on reference tests used to determine targets in Sequence IIIG.

[B] standard deviation

5. References:

1. Bardasz et. al., “Low Volatility ZDDP Technology: Part 1 Engines and Lubricant Performance in Field Applications”, SAE 2007-01-1990 (2007)

2. Bardasz et. al., “Low Volatility ZDDP Technology: Part 2 Exhaust Catalyst Performance in Field Applications”, SAE 2007-01-4107 (2007)

3. Appendix C, API 1509 EOLCS, 16th ed. (2007)

4. Japanese Automotive Manufacturers Association (JAMA)

Appendix A Literature Review

- SAE 952416---Donald D. Beck, David R. Monroe, Craig L. DiMaggio, John W. Sommers, "Axial Characterization of Lightoff and Underfloor Catalytic Converters Vehicle-Aged on a 5.7 L Corvette", SAE Technical Paper 952416 (1995)
- SAE 940746---Fumio Ueda, Shinichi Sugiyama, Kazutaka Arimura, Shigeki Hamaguchi, Kenyu Akiyama, "Engine Oil Additive Effects on Deactivation of Monolithic Three-Way Catalysts and Oxygen Sensors ", SAE Technical Paper 940746 (1994)
- SAE 2002-01-2680---M.Johnson, R.McCabe,C.Hubbard,m.Riley,C.Kirby,D.Ball,G.Tripp,T.McDonnell,W.Lam, "Effects of Engine Oil Formulations Variables on Exhaust Emissions in Taxi Fleet Service", SAE 2002-01-2680 (2002).
- Greg Guinther, Afton Chemical, Afton Catalyst Test- Researching the Effect of PEI on Phosphorus Related Catalyst Poisoning, 12/15/05 presentation to ESCIT.
- S.A. Culley and T.F. McDonnell, "The Impact of Passenger Car Motor Oil Phosphorus Levels on Engine Durability, Oil Degradation, and Exhaust Emissions in a Field Trial", SAE Technical Paper 952344 (1995).
- S.T. Darr, R.A. Choksi, C.P. Hubbard, M.D. Johnson and R.W. McCabe, "Effects of Oil-Derived Contaminants on Emissions from TWC-Equipped Vehicles", SAE Technical Paper 2000-01-1881 (2000).
- S.A. Culley, T.F. McDonnell, D.J. Ball, C.W. Kirby and S.W. Hawes, "The Impact of Passenger Car Motor Oil Phosphorus Levels on Automotive Emissions Control Systems", SAE Technical Paper 961898 (1998).
- S.A. Culley, T.F. McDonnell, D.K. Walters, D.J. Ball, C.W. Kirby and S.W. Hawes, "The Effect of Passenger Car Motor Oil Phosphorus Levels on Engine Durability, Oil Degradation, and Emissions Control Systems", CEC Paper CEC97-EL22, (1997).
- D.J. Ball and C.W. Kirby, "A Survey of Automotive Catalyst Technologies Using Rapid Aging Test Schedules Which Incorporated Engine Oil Derived Poisons", SAE Technical Paper 973050 (1997).
- D.S. Lafayatis, R. Petrow and C. Bennett, "The Effects of Oil-Derived Poisons on Three-Way Catalyst Performance", SAE Technical Paper 2002-01-1093 (2002).
- S.K. Korcek, M.D. Johnson, R.K. Jensen, C. Stow and J. Bennett, "Emissions Driven Engine Oil Reformulation", *Proceeding of the 14th European Automotive Symposium "Engine, fuels and lubricants: a view for the future"*, Nice, France, Sept. 2001.
- Fumio Ueda, et al., "Engine Oil Additive Effects on Deactivation of Monolithic Three-Way Catalysts and Oxygen Sensors", SAE Technical Paper 940746 (1994).
- W. Chamberlin and F. Zalar, "Balancing Crankcase Lubricant Performance with Catalyst Life", SAE Technical Paper 841407 (1984).
- T.W. Selby, "Development and significance of the Phosphorus Emission Index of Engine Oils", *Proceedings of the 13th International Colloquium Tribology – Lubricants Materials and Lubrication*, Esslingen, Germany, 2002.

- Drury, C. & Whitehouse, S., The effect of lubricant phosphorus level on exhaust emissions in a field trial of gasoline engine vehicles; SAE 940745
- Ueda, F, et al.; Engine oil additive effects on deactivation of monolithic 3-way catalysts and oxygen sensors; SAE 1994 trans, 4, 332-341.
- Inoue et al., Effects of Phosphorus and ash contents of engien oils on deactivation of monolithic three-way catalysts and oxygen sensors; SAE 920654.
- Brett, P. S. et al., An Investigation into Lubricant related poisoning of automotive three-way catalysts and lambda sensors, SAE 890490.
- Durbin, T. D., Miller, J.. W., Pisano, J. T., Sauer, C., Rhee, S., Huai, T., "Impact of Engine Oil Sulfur Content on Emissions", CRC Project No. E-61, May 2002.
- Gotta, L., Natoli, G., Salino, P., Barr, D., and Boyer, M., How Modern Engine Oils can impact on Emission Reduction, JSAE20030344, SAE 2003-01-1989.
- Webb, C. C., Bartley, J. J., Bykowski, B. B., Farnsworth, G., and Riley, M., Catalyst Aging Evaluation with Exposure to 0.06 and 0.11 Percent Phosphorus Oils Using the FOCAS Burner System, JSAE 20030269, SAE 2003-01-1999
- Chamberlin, W., Kelley, J., Wilk, M., “The Impact of Passenger Car Motor Oils on Emissions Performance,” SAE Paper No. 2003-01-1988
- Eastwood, P., “Critical Topics in Exhaust Gas Aftertreatment,” Baldock Research Studies Press Ltd. Hertfordshire, England, 2000.
- Crocker, M., Lox, E., Presentation “Deactivation of Automotive Emissions Control Catalysts: An Overview ,” Southwest Research Institute Non-Thermal Catalyst Deactivation (N-TCD) Symposium, January 2001.
- Selby, T., “Development and Significance of the Phosphorus Emission Index of Engine Oils,” Expanded version of paper: 13th International Colloquium Tribology- Lubricants Materials and Lubrication Technische Akademie Esslingen, January, 2002, Stuttgart/Ostfildern, Germany.
- Webb,C., Bykowski, B., “Development of a Methodology to Separate Thermal from Oil Aging of a Catalyst Using a Gasoline-Fueled Burner System,” SAE Paper No. 2003-01-0663.
- Ball, D., Mohammed, A., Schmidt, W., “Applications of Accelerated Rapid Aging Test (RAT) Schedules with Poisons: The Effects of Oils Derived Poisons, Thermal Degradation and Catalyst Volume on FTP Emissions,” SAE Paper No. 972846.
- Natoli, G., Pometto, C., Salino, P., Guerzoni, M., “Three-way Catalyst Deactivation by Lubricants During Fast Aging Engine Test,” ITT 8-48-12-685.
- Bartley, G., discussion regarding catalyst deactivation mechanisms, Southwest Research Institute, 26 April, 2005.
- ASTM Committee D02 on Petroleum Products and Lubricants, Unapproved Minutes, Oil Protection of Emissions System Test (OPEST) II Task Force, 27 September, 2000, Houston, Texas.

Appendix B List of tests proposed during ESCIT

An exit ballot was issued by ESCIT in November, 2008 to narrow down the list of tests being considered. That ballot listed all test methods proposed for ESCIT as follows:

	Proposed Volatility / Retention Test
1	CIBA Proposed Test Method
2	Phosphorous Retention in the Sequence IV A
3	Phosphorous Retention in the Sequence VG
4	Phosphorous Retention in the Sequence VI D.
5	Phosphorous Retention in the Sequence IIIG. (20 Hrs)
6	Phosphorous Retention in Sequence IIIG (EOT)
7	Phosphorous Retention in the Sequence VI B.
8	PEI 165
9	PEI 250
10	ROBO- Phosphorous Retention
11	Southwest Research Institute Engine Dyno Test

A TEOST MHT proposal was also added to the poll. The results released on Jan 9, 2008 showed the following results:
5: 2 votes
6: 7 votes
9: 3 votes
MHT: 3 votes
There was also 1 vote supporting IIIG at 40 hours.

IIIG PR was further developed into IIIGB because the IIIG had the most support after combining all versions (different hours). ESCIT released its final vote on test method and calculation selections in two letters released to ILSAC/Oil chair to document the findings (**Appendix C**).

Appendix C ESCIT Recommendations to ILSAC/Oil

TOYOTA

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January 8, 2008

Dear Chairman, Bob

The Emission System Compatibility Investigation Team (ESCIT) is pleased to be able to deliver a recommendation to ILSAC/Oil on how best to measure the phosphorus impact on the emissions system. We recommend using the Sequence IIIG engine test and evaluating at end of test.

While ESCIT recognizes the benefits of a bench test from both a cost and ease-of-use perspective, precision and accuracy ultimately trump these considerations. At this time the Sequence IIIG is the best choice for measuring the engine oil's catalyst impact.

ESCIT will continue to discuss the most appropriate calculation method. We will notify ILSAC/Oil when this process is complete.

This information will be shared with the entire ILSAC/Oil group at the January 23, 2008 meeting.

Best Regards,

Hannah Murray
ESCIT Chair

TOYOTA

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April 18, 2008

Dear Mr. Chairman, Bob

The Emission System Compatibility Investigation Team (ESCIT) is pleased to be able to deliver a second recommendation to ILSAC/Oil on how best to measure the phosphorus impact on the emissions system. We previously recommended using the Sequence IIIG engine test and evaluating at end of test; a recommendation we still support.

Regarding the phosphorus calculation method, ESCIT supports the usage of a phosphorus retention calculation method.

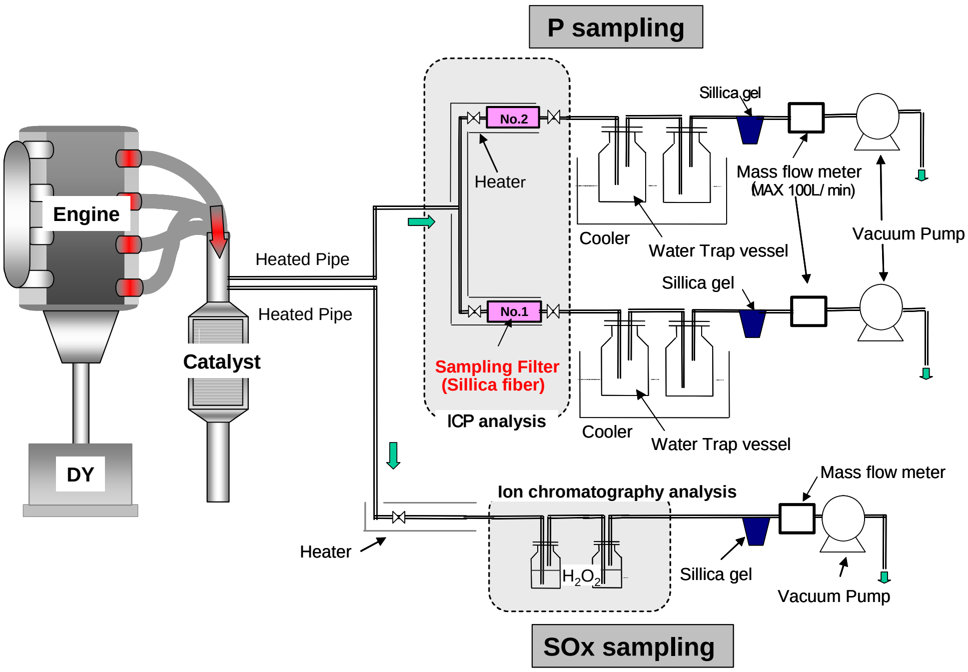
Several ESCIT members have outlined various steps to improve the actual measurement of phosphorus. These actions, while beneficial to the industry and the GF-5 category, will be addressed in other industry forums.

We look forward to future discussions within ILSAC/Oil on the appropriate limit for this test.

Best Regards,

Hannah Murray
ESCIT Chair

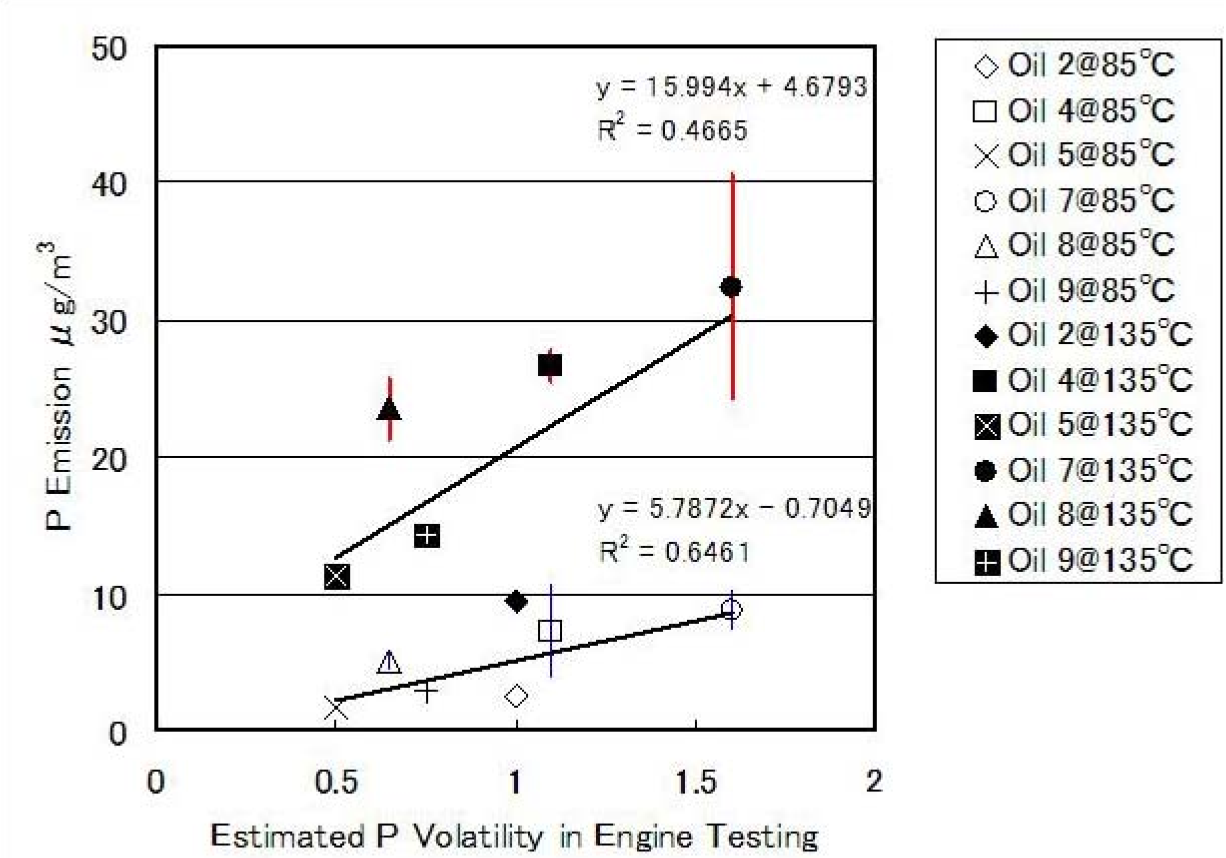
Appendix D Dynamometer Catalyst Evaluation vs. IIIG Data Provided by JAMA[4]



Cylinder		In-line 4
Valve Train		DOHC 16 valve
Displacement	cm^3	1998
Compression Ratio		10.0
Rated Power	kW / (r/ min)	110 / 6000
Max. Torque	Nm / (r/ min)	182 / 4500
Fuel Supply		EGI
Oil Pan Volume	L	4.6

		Conditon H	Conditon M
Engine Operating Condition		**High Oil Temp.**	**Middle Oil Temp.**
Rev.	**r/min**	**4000**	**2500**
Torque	**N.m**	**150**	**100**
Coolant outlet temp.	**℃**	**105**	**80**
Oil pan temp.	**℃**	**135**	**85**
Test Fuel		**Premium Gasoline (S:2ppm)**	

Oil Code	Level	P %	PEI	ZDTP	Test Condition		
Oil 2	5W30 GF-5	0.08	Typical	A	Conditon H	High Oil Temp.	(n=2)
					Conditon M	Middle Oil Temp.	(n=2)
Oil 4	Oil 2 changed	0.08	Typical	B	Conditon H	High Oil Temp.	(n=2)
					Conditon M	Middle Oil Temp.	(n=2)
Oil 5	Oil 2 changed	0.08	Typical	C	Conditon H	High Oil Temp.	(n=1)
					Conditon M	Middle Oil Temp.	(n=1)
Oil 7	Oil 4 +ZDTP	0.12	Typical	B	Conditon H	High Oil Temp.	(n=2)
					Conditon M	Middle Oil Temp.	(n=2)
Oil 8	Oil 4 -ZDTP	0.05	Typical	B	Conditon H	High Oil Temp.	(n=2)
					Conditon M	Middle Oil Temp.	(n=2)
Oil 9	Oil 2 changed	0.05	High	D	Conditon H	High Oil Temp.	(n=1)
					Conditon M	Middle Oil Temp.	(n=1)



Appendix E TMC Data Used to Estimate Preliminary IIIGB Precision

TESKEY	OIL	LAB	OILLH020	OILLH040	OILLH060	OILLH080	OILLH100	CA_INI	CA_020	CA_040	CA_060	CA_080	CA_EOT	PH_INI	PH_020	PH_040	PH_060	PH_080	PH_EOT	PR
47883	tmc-434	A	441	780	981	1114	1107	0.2157	0.2449	0.2761	0.3007	0.3323	0.3483	0.0831	0.0725	0.0777	0.0851	0.095	0.1013	75.49
47884	tmc-434	A	763	1102	1268	1433	1619	0.2094	0.2501	0.2891	0.3115	0.3428	0.367	0.0806	0.0709	0.0778	0.0841	0.0933	0.102	72.21
47885	tmc-434	A	448	586	756	1026	1365	0.2078	0.2446	0.2732	0.2989	0.3243	0.3441	0.0812	0.072	0.0771	0.0847	0.0933	0.0996	74.07
47886	tmc-434	A	824	1423	1778	2063	2291	0.2116	0.2493	0.2932	0.3214	0.3632	0.401	0.0818	0.0728	0.0826	0.0919	0.1047	0.1173	75.67
47900	tmc-434	G	648	1084	1379	1605	1875	0.2011	0.2521	0.2911	0.3303	0.345	0.3697	0.0749	0.0679	0.0746	0.0871	0.0931	0.1006	73.06
47901	tmc-434	G	412	788	990	1222	1506	0.2154	0.2594	0.2849	0.306	0.34	0.3762	0.076	0.0681	0.0726	0.082	0.0899	0.1014	76.39
47902	tmc-434	G	399	836	1099	1326	1535	0.2008	0.246	0.2841	0.3039	0.3316	0.3836	0.0734	0.0673	0.0744	0.0828	0.0917	0.1025	73.10
47916	tmc-434	B	858	1259	1650	2000	2640	0.1887	0.2405	0.278	0.3186	0.352	0.4222	0.0731	0.0697	0.0776	0.0879	0.0913	0.1181	72.21
47917	tmc-434	B	441	611	679	1278	1933	0.1951	0.2318	0.2586	0.2967	0.3141	0.3358	0.0764	0.0673	0.0722	0.0822	0.0878	0.0956	72.70
47924	tmc-434	F	946	1550	1973	2292	3198	0.18	0.22	0.26	0.29	0.33	0.36	0.07	0.065	0.071	0.081	0.093	0.1	71.43
47925	tmc-434	F	605	848	1054	1358	1907	0.191	0.223	0.249	0.272	0.3	0.334	0.0704	0.0641	0.0692	0.0763	0.0851	0.0975	79.20
48581	tmc-434	G	340	850	1017	1183	1497	0.207	0.2275	0.2611	0.2983	0.3292	0.3526	0.0744	0.0641	0.0706	0.0824	0.092	0.1001	78.99
48582	tmc-434	G	342	720	889	1056	1150	0.1926	0.2304	0.2542	0.3017	0.3134	0.3472	0.0692	0.0637	0.0673	0.0795	0.0838	0.0939	75.27
48584	tmc-434	A	583	989	1222	1451	2008	0.1855	0.2314	0.2543	0.2825	0.3398	0.3507	0.0726	0.0686	0.0728	0.0816	0.0983	0.1032	75.19
48605	tmc-434	G	603	1017	1456	1948	2029	0.1985	0.2381	0.2838	0.3065	0.3426	0.3964	0.0736	0.0656	0.075	0.0811	0.0957	0.1123	76.41
49073	tmc-434	B	451	828	997	1265	1486	0.195	0.2405	0.2745	0.3067	0.3734	0.3607	0.0761	0.0681	0.0762	0.0861	0.1011	0.1022	72.60
49510	tmc-434	G	551	1059	1488	1778	2137	0.1922	0.2286	0.269	0.3193	0.3435	0.3942	0.0698	0.0619	0.0687	0.0819	0.0892	0.1042	72.79
49705	tmc-434	A	203	480	787	888	1082	0.1902	0.2283	0.2487	0.2694	0.291	0.3148	0.0729	0.0691	0.0707	0.0773	0.0844	0.0915	75.83
49706	tmc-434	A	414	688	1093	1292	1510	0.1946	0.2325	0.2617	0.286	0.3232	0.3451	0.073	0.0672	0.0708	0.0775	0.0874	0.095	73.38
50450	tmc-434	B	344	1026	1520	1905	2045	0.198	0.2313	0.2693	0.296	0.3292	0.3567	0.0751	0.0705	0.0782	0.0856	0.0969	0.1044	77.17
47890	tmc-435	A	489	833	1003	1238	1429	0.1764	0.2061	0.2304	0.2532	0.272	0.291	0.0813	0.0796	0.0855	0.0933	0.1006	0.1083	80.75
47891	tmc-435	A	530	875	1214	1514	1893	0.1811	0.2109	0.2346	0.2636	0.2749	0.3063	0.0831	0.0822	0.0877	0.0982	0.1021	0.1139	81.04
47892	tmc-435	A	824	1093	1390	1746	2045	0.1748	0.1984	0.2337	0.2616	0.2861	0.3112	0.0797	0.0762	0.0868	0.0971	0.1057	0.1147	80.84
47905	tmc-435	G	406	780	1218	1256	1504	0.1722	0.2113	0.2392	0.2668	0.2859	0.2969	0.0737	0.0747	0.0814	0.0907	0.0973	0.1077	84.76
47906	tmc-435	G	614	984	1347	1637	1936	0.1707	0.2165	0.2312	0.259	0.2887	0.3214	0.0726	0.0759	0.0801	0.0902	0.0991	0.1108	81.06
47907	tmc-435	G	376	886	1285	1577	1848	0.165	0.1935	0.2321	0.262	0.2813	0.3109	0.0736	0.0733	0.0812	0.0913	0.0981	0.1067	76.94
47908	tmc-435	G	464	849	1428	1759	1946	0.1745	0.2049	0.224	0.2514	0.2911	0.3229	0.0731	0.0737	0.081	0.091	0.1017	0.1126	83.24
47909	tmc-435	G	587	961	1327	1621	2491	0.1713	0.2049	0.2327	0.2741	0.3172	0.3443	0.0743	0.0752	0.0823	0.0968	0.1116	0.1191	79.75
47918	tmc-435	B	133	446	583	787	1392	0.1643	0.1943	0.2134	0.2329	0.2502	0.2702	0.0768	0.0764	0.0777	0.0865	0.091	0.1015	80.36
47919	tmc-435	B	446	787	1122	1287	2277	0.1615	0.1937	0.2219	0.2446	0.2704	0.2876	0.075	0.0748	0.0822	0.09	0.0996	0.1048	78.47
47920	tmc-435	B	205	414	552	688	824	0.1609	0.1894	0.2096	0.2309	0.2513	0.2665	0.076	0.0758	0.0786	0.0848	0.0931	0.0969	76.98
47927	tmc-435	F	429	709	985	1291	2063	0.15	0.17	0.19	0.21	0.23	0.25	0.067	0.067	0.071	0.077	0.087	0.094	84.18
47928	tmc-435	F	254	572	816	885	956	0.15	0.175	0.195	0.209	0.225	0.243	0.0656	0.0668	0.0705	0.076	0.0818	0.0881	82.90
47938	tmc-435	D	341	751	1020	1251	1373	0.1688	0.1984	0.2264	0.2511	0.2747	0.296	0.0734	0.0736	0.0808	0.0895	0.0983	0.1063	82.59
48579	tmc-435	G	274	448	688	790	923	0.1741	0.2014	0.2274	0.2395	0.2738	0.2904	0.0737	0.0731	0.0793	0.0856	0.0919	0.0979	79.64
48580	tmc-435	G	169	413	756	959	1154	0.1645	0.2027	0.2542	0.2478	0.2695	0.289	0.0707	0.0711	0.0807	0.0858	0.0937	0.1019	82.04
48587	tmc-435	A	346	622	894	1129	1255	0.1591	0.2014	0.2076	0.2297	0.2533	0.2755	0.073	0.0786	0.077	0.0851	0.0939	0.1028	81.32
49067	tmc-435	D	173	557	729	899	1032	0.1653	0.1923	0.2118	0.2324	0.2493	0.268	0.0724	0.0723	0.077	0.0828	0.0909	0.098	83.49
49074	tmc-435	B	171	484	792	961	1223	0.164	0.1937	0.2177	0.2417	0.2642	0.2843	0.0754	0.0747	0.0809	0.09	0.0964	0.1049	80.25
49076	tmc-435	A	135	654	688	688	890	0.1562	0.1872	0.207	0.226	0.2458	0.262	0.0702	0.0715	0.0754	0.08	0.0879	0.0926	78.64
49512	tmc-435	G	134	482	620	586	1022	0.1734	0.2037	0.2244	0.2402	0.2635	0.2768	0.0738	0.0738	0.0779	0.0833	0.0916	0.0968	82.17
50457	tmc-435	A	203	446	651	719	953	0.1622	0.18	0.221	0.2387	0.2651	0.2776	0.0706	0.0673	0.0818	0.0883	0.0982	0.1046	86.57
51017	tmc-435	G	413	959	1682	2219	2381	0.1794	0.2067	0.2363	0.2709	0.3049	0.3432	0.0755	0.0741	0.0816	0.0936	0.1064	0.1193	82.60
51018	tmc-435	G	169	482	654	891	1088	0.1751	0.2067	0.2309	0.2529	0.2623	0.2852	0.0728	0.0743	0.0789	0.0856	0.0911	0.0996	84.00
51027	tmc-435	A	344	586	959	1093	1382	0.1656	0.1983	0.223	0.2514	0.2761	0.2982	0.0733	0.0748	0.0792	0.0891	0.0973	0.1056	80.00
51752	tmc-435	G	344	688	790	925	1187	0.1711	0.2009	0.2199	0.2394	0.2653	0.2841	0.0729	0.072	0.076	0.0831	0.0928	0.1002	82.78
52628	tmc-435	A	414	654	824	892	957	0.1645	0.1912	0.2123	0.2311	0.2474	0.2685	0.0728	0.0729	0.0777	0.0842	0.0899	0.0969	81.55
47893	tmc-438	A	416	760	964	1065	1259	0.1457	0.1681	0.1839	0.2017	0.2208	0.2321	0.0972	0.088	0.0924	0.1018	0.1118	0.1176	75.95
47894	tmc-438	A	441	713	848	948	1107	0.1474	0.17	0.1865	0.2022	0.2207	0.2333	0.0979	0.0875	0.0931	0.1012	0.1115	0.1193	76.99
47895	tmc-438	A	380	656	792	860	959	0.1462	0.1637	0.1808	0.1936	0.2068	0.2285	0.0978	0.0857	0.0902	0.098	0.105	0.1178	77.07
47896	tmc-438	A	344	552	688	756	923	0.1452	0.1615	0.1766	0.1946	0.207	0.2211	0.0967	0.0852	0.09	0.0995	0.1066	0.1144	77.69
47897	tmc-438	A	275	414	517	586	1252	0.1389	0.1547	0.1715	0.1858	0.2025	0.2163	0.0937	0.0813	0.0874	0.0938	0.1036	0.1112	76.21
47910	tmc-438	G	616	1020	1285	1609	1911	0.1445	0.1652	0.1879	0.2125	0.2323	0.2401	0.0889	0.0816	0.087	0.0973	0.1091	0.114	77.18
47911	tmc-438	G	648	1183	1509	1701	1967	0.1391	0.1686	0.1926	0.2188	0.2313	0.267	0.0882	0.08	0.0879	0.1008	0.1126	0.1261	74.48
47913	tmc-438	G	167	410	684	853	1049	0.1447	0.1571	0.1711	0.1873	0.2147	0.2322	0.0899	0.0797	0.0833	0.091	0.1001	0.1081	74.93
47914	tmc-438	G	238	549	720	1056	1506	0.1381	0.1559	0.1785	0.1936	0.211	0.2276	0.0901	0.0754	0.0841	0.0915	0.1005	0.1099	74.01
47921	tmc-438	B	96	164	266	300	1080	0.1359	0.1513	0.164	0.1762	0.1903	0.1991	0.0916	0.0805	0.084	0.0905	0.1031	0.1038	77.35
47922	tmc-438	B	0	167	167	202	749	0.1367	0.1511	0.1629	0.1778	0.1879	0.1975	0.097	0.0808	0.0864	0.0982	0.1034	0.1035	73.85
47923	tmc-438	B	30	241	276	415	420	0.1404	0.1589	0.1744	0.1845	0.1901	0.2007	0.0902	0.0806	0.0852	0.0913	0.0962	0.1022	79.26
47930	tmc-438	F	468	956	1229	1432	2004	0.13	0.15	0.17	0.18	0.19	0.2	0.084	0.076	0.083	0.089	0.095	0.1	77.38
47932	tmc-438	F	287	638	811	880	1726	0.12	0.14	0.16	0.17	0.18	0.19	0.08	0.075	0.079	0.086	0.092	0.098	77.37
47941	tmc-438	D	103	419	419	1102	866	0.1378	0.1529	0.169	0.1815	0.1922	0.2051	0.0895	0.0794	0.0835	0.089	0.0955	0.1031	77.40
48577	tmc-438	G	239	586	992	1520	1827	0.1507	0.163	0.1807	0.2008	0.2425	0.2672	0.0893	0.0808	0.0862	0.0959	0.1125	0.1292	81.60
48585	tmc-438	A	209	385	489	559	699	0.1418	0.1611	0.1747	0.1859	0.1971	0.2096	0.0957	0.0861	0.0901	0.0952	0.1018	0.1087	76.84
48586	tmc-438	A	310	688	756	1026	1187	0.1245	0.1497	0.1662	0.1829	0.1992	0.2125	0.0848	0.0797	0.0834	0.092	0.101	0.1089	75.24
49064	tmc-438	D	100	136	346	450	658	0.1366	0.1536	0.1657	0.1785	0.1909	0.2017	0.0888	0.0799	0.0828	0.089	0.0955	0.1024	78.10
49069	tmc-438	B	207	451	624	624	762	0.1382	0.1593	0.1748	0.1858	0.1975	0.2125	0.0914	0.0847	0.0903	0.0956	0.102	0.1106	78.70
49070	tmc-438	B	0	275	483	620	655	0.14	0.1591	0.1701	0.1858	0.2008	0.2138	0.093	0.0828	0.0854	0.0924	0.1025	0.11	77.45
49075	tmc-438	A	135	414	517	586	622	0.1344	0.1567	0.1715	0.182	0.1931	0.1941	0.0868	0.0778	0.0827	0.0872	0.0923	0.0967	77.14
49515	tmc-438	G	208	453	626	729	832	0.1431	0.1572	0.1808	0.1975	0.2125	0.2237	0.0858	0.0784	0.0835	0.0932	0.1005	0.1066	79.48
49516	tmc-438	G	374	749	951	1050	1110	0.1453	0.1617	0.1787	0.199	0.2154	0.2307	0.0882	0.0805	0.0855	0.0931	0.1015	0.1099	78.48
49708	tmc-438	A	136	451	589	589	862	0.1401	0.1586	0.174	0.1917	0.1999	0.2252	0.0948	0.0841	0.0893	0.0966	0.1013	0.1144	75.07
50458	tmc-438	A	136	382	486	555	660	0.1452	0.1626	0.1804	0.1956	0.2193	0.2245	0.0952	0.0826	0.0894	0.0959	0.1099	0.1113	75.62

Appendix F TMC Information Letter Defining The Final Procedure of IIIGB

TMC
A Program of ASTM International

Test Monitoring Center
Carnegie Mellon University
6555 Penn Avenue, Pittsburgh, PA 15206, USA
http://astmtmc.cmu.edu
412-365-1000

Sequence IIIG Information Letter 08-2
Sequence No. 18
November 6, 2008

Approved by ASTM D02.B on October 29, 2008

TO: Sequence III Mailing List

SUBJECT: Sequence IIIGB Test Creation

The Sequence III Surveillance Panel approved, via electronic ballot, the creation of a new version of the Sequence IIIG test measuring only percent phosphorus retention. Appendix X3 has been added to Test Method D7320 to define the requirements for conducting this procedure, referred to as the Sequence IIIGB. Sections 1.1.1, 1.4, and 2.1 have also been updated. This change is effective the date of this information letter.

Bruce Matthews
Engine Oil Test Development and Support
GM Powertrain Materials Engineering

John L. Zalar
Administrator
ASTM Test Monitoring Center

Attachment

c: ftp://ftp.astmtmc.cmu.edu/docs/gas/sequenceiii/procedure_and_ils/IIIG/IL08-2.pdf

Distribution: Electronic Mail

APPENDIX

X3. SEQUENCE IIIGB TEST PROCEDURE

X3.1 *Overview*

X3.1.1 The Sequence IIIGB supplement to the Sequence IIIG test was developed to generate used oil samples to measure the phosphorus retention of a test lubricant after 100 h of Sequence IIIG test operation. No parts ratings or measurements are required in the Sequence IIIGB test. A separate Sequence IIIGB Report Form Set is available from the TMC for reporting Sequence IIIGB test results. Do not use the Sequence IIIG Report Form Set to report Sequence IIIGB test results. The oil samples used for measurement of the phosphorus retention in the Sequence IIIGB test are the initial oil sample, removed from the engine following the initial run-in, and the end-of -test 100-h oil sample. The phosphorus retention calculation is:

$$\text{Phosphorus Retention} = (Ca_{t1} / Ca_{t100}) \times (P_{t100} / P_{t1}) \times 100$$

where Ca_{t1} and P_{t1} are analytical results from the initial oil sample and Ca_{t100} and P_{t100} are analytical results from the end-of-test oil sample. Use Test Method D 5185 to measure calcium and phosphorus concentrations. For oils where calcium is not the highest concentration detergent metal, substitute the highest concentration detergent metal into the equation for calcium

X3.2 *Preparation of Apparatus* – Prepare the Sequence IIIGB test engine in the same manner as a Sequence IIIG or IIIGA test engine; except that the pre-test camshaft and lifter measurements are not required. No special preparations are required or permitted on test engines for Sequence IIIGB testing.

X3.3 *Calibration*

X3.3.1 There is no stand-alone calibration system for the Sequence IIIGB test. A stand that is calibrated for the Sequence IIIG is also calibrated for Sequence IIIGB testing. Conduct a Sequence IIIIGB test simultaneously with each Sequence IIIG reference oil test.

X3.3.2 No special calibration of stand instrumentation is required for Sequence IIIGB testing.

X3.3.3 A Sequence IIIGB test counts as one run against the Sequence IIIG calibration period in which it was run. A test run as a combined Sequence IIIG, IIIGA, or IIIGB test counts as only one run against the calibration period.

X3.4 *Test Procedure* – The Sequence IIIG/B test can be conducted in one of two ways:

X3.4.1 *Stand-alone Sequence IIIGB Test* – If only a Sequence IIIGB test result is needed, conduct the test in the normal manner as detailed in Test Method D 7320 with the exception of ratings, wear measurements, or assessment of stuck rings. At the end of test, report all results as a stand-alone IIIGB test using the Sequence IIIGB Report Form Set.

X3.4.2 *Combined Sequence IIIG, IIIGA, IIIGB Sequence Test* – If Sequence IIIG, IIIGA, and IIIGB test results are desired on a non-reference oil, conduct the test in the normal manner as listed in Test Method D 7320, identify the test as a Combined Sequence IIIG, IIIGA, IIIGB on Report Form 1 and complete all forms in the standard Sequence IIIG, IIIGA, and IIIGB Report Form Sets including all ratings, measurements, and used oil analyses.

X3.5 *End-of-Test Oil Sample Testing*- The phosphorus and calcium elemental concentrations for all oil samples are to be reported in mg/kg as determined using Test Method D 5185. All samples, initial and end-of-test, are to be run sequentially, in duplicate, using the same calibration (i.e. as close in time as practical). Background correction, internal standard, and peristaltic pump are required. Use sample dilutions of at least 1:20. Once a dilution is established, use it for all samples from a test. Report the average of the two determinations as the final result. If the duplicate determinations are outside the repeatability calculations shown in Table 2 of Test Method D 5185, follow the procedure in Test Method D 3244 Section 6.2.

X3.6 *Quality Index* – Calculate quality index results for a Sequence IIIGB test in the same manner as for the Sequence IIIG.

X3.7 *Test Reporting* – Report Sequence IIIGB test results using the standard IIIGB Report Form Set, available from the TMC.

X3.8 *Precision and Bias* – The precision and bias of this test procedure for measuring phosphorus retention has yet to be determined.