ASTM RESEARCH REPORT

Development of the Sequence IIIG Engine Oil Test

By
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Background

The Sequence IIIF test was developed by General Motors for inclusion in the International Lubricants Standardization and Approval Committee (ILSAC) GF-3 oil category. However, concerns about the oxidation discrimination and imprecise cam and lifter wear were raised after the precision matrix was conducted on the original test. General Motors agreed to rework the Sequence IIIF test procedure and hardware to address these concerns. To do this they formed a task force with PerkinElmer Automotive Research (PEAR) and Southwest Research Institute (SwRI). They succeeded in increasing the oxidation severity of the test but virtually eliminated cam and lifter wear discrimination.

For development of a new Sequence test that would be used in ILSAC GF-4, a task force was again formed with GM Powertrain, PerkinElmer Automotive Research, Southwest Research Institute, and OH Technologies (OHT).
The objectives were to:

- Increase oxidation severity to approximately twice that of the IIIF test.
- Increase piston deposit severity to a level that would require detergency performance equivalent to API CD or ACEA B1 oils.
- Increase cam & lifter wear severity, with improved precision; over the IIIF test so that oils could be separated in terms of their high temperature wear performance.

Two separate tests considered

The initial thoughts of the ILSAC committee were to separate the Sequence III test into a wear test and an oxidation and deposits test. GM Powertrain was to develop the wear test. Initial work started in January of 2001. Daimler Chrysler was to develop the oxidation and deposit test and started work at about the same time. DaimlerChrysler and Lubrizol did start development of a test using the Chrysler 2.7 liter V-6 engine. The test conditions for the proposed 2.7-liter test resembled those of the Sequence IIIF test except that the engine was operated at higher speed, load, and temperatures. The test length was also increased to 100 hours and the forced oil additions reduced. In May 2001 ILSAC decided to recombine the tests into one test, the Sequence IIIG . Similar test conditions to those developed for the DaimlerChrysler test were incorporated into the new test.

New engine considered

Early in 2001, GM Powertrain thought that it would be best if the Sequence III were switched from a production engine to a custom Chevrolet small-block V-8 crate engine supplied by GM Performance Parts. This engine would be based on one of the semi-custom 350 CID blocks so widely used in racing. Supply of such an engine would not be dependent on current production and could be assured indefinitely.

Unfortunately this plan had several drawbacks. One was that running such an engine would produce too much horsepower for the dynamometers in place at the labs for the Sequence IIIF test as well as consuming substantially more fuel. It would also require a custom sourced camshaft as castings for service camshafts introduce to much variability in camshaft lobe width and linear positioning for precision wear testing.
In view of the above and the limited time available for developing a new test, it was decided by the development group to stay with the 3800 Series II engine used in the Sequence IIIF test.

Lifter foot metallurgy

Both the Sequence IIID and IIIE tests used four grams per gallon leaded fuel so that significant wear and oxidation severity could be obtained in a reasonably short test length. Attempts to run Sequence IIID and IIIE tests with unleaded fuel resulted in little or no measurable wear. In addition, the D-500 lifters used in the Sequence IIID and IIIE tests were no longer available. After an exhaustive study, a lifter with a 52100 steel foot was selected for use in the Sequence IIIF test. During the original Sequence IIIF Precision Matrix, low wear (approximately 7 microns average camshaft and lifter wear) was measured with all the matrix oils.

After the Sequence IIIF Precision matrix was completed , the Passenger Car Engine Oil Classification Panel (PCEOCP) decided that the original Sequence IIIF test was not severe enough and suggested that the test conditions needed to be changed to increase oil oxidation severity. During redevelopment testing , random catastrophic camshaft and lifter wear was encountered. Given the very short time available for redeveloping the Sequence IIIF test and knowing the pitfalls awaiting anyone attempting to unravel the causes of random catastrophic wear, the development group decided to switch to service production alloy cast iron (ACI) lifters.

ACI lifters were identified in the 1950's as being extremely wear resistant and almost universally used in all North American manufactured engines until the advent of roller followers. D500 lifters were cast from the same material as the ACI lifters. However, the grain structure in the D500 lifter foot was finer and more importantly the cast iron material did not extend to the edge of the lifter foot. Switching from steel lifters to the ACI lifters greatly reduced the occurrence of random catastrophic wear but also greatly reduced the test sensitivity to wear. It was expected that all oils would be well under 20 microns average wear, so a failsafe limit of 20 microns was set. One of the objectives in developing the Sequence IIIG test was to increase the wear severity so that the performance of oils could again be separated.

Cam and lifter wear

While it was expected that switching to ACI lifter feet in the redeveloped Sequence IIIF test would result in extremely low wear, and a virtual elimination of single lobe catastrophic wear, it did not in all cases. To lower the impact of single lobe catastrophic wear occurrence on test results, a method of "screened average cam and lifter wear" was instituted where the cam and lifter positions with the highest and lowest combined wear were disregarded and the remaining 10 positions were averaged. However, it was also noticed that some oils produced uniform high wear across all cam and lifter positions. Significant wear could be experienced using typically wear-resistant ACI lifters and "as ground" (non-phosphate coated) cast iron camshafts. One theory was that the increase in lifter wear was related to the increase in test severity from the original Sequence IIIF to the redeveloped Sequence IIIF. If this were true, the further increase in test severity of the Sequence IIIG should result in even more camshaft and lifter wear.

Given the unpredictable results obtained when the steel lifters were used in the redeveloped Sequence IIIF test, and warnings in various materials application handbooks that the 52100 steel used in the original Sequence IIIF test lifters was subject to random catastrophic failures at higher temperatures, no alternative to using ACI lifters in the Sequence IIIG test was considered

Camshaft and lifter wear data generated during Sequence IIIG test development and in the Precision Matrix program presented in this document is reported as the average of the combined wear (cam + lifter) of all twelve positions.

Operating conditions

The main oil gallery inlet supply temperature in the Sequence IIIF test had been raised from 149°C in the Sequence IIIE to 155°C to increase the test severity. Mr. Stephen Korcek, of Ford's Scientific Research Lab, felt that this operating temperature was too high and could lead to abnormal ZDP depletion. Based on his suggestion, the oil gallery inlet temperature of the Sequence IIIG test was set at 150°C, closer to the temperature in the Sequence IIID and IIIE tests. The coolant temperature was also reduced from 122°C in the Sequence IIIF to 115°C, the same as in the Sequence IIID and IIIE tests. The test length, load, and inlet air temperature were all increased while the new oil additions were decreased to reflect the 2.7L DaimlerChrysler test operating conditions. These conditions remained constant throughout the entire development program, and are summarized in Table 1.

Table 1 – IIIF and IIIG Test Conditions

Parameter	IIIF	IIIG
Test Length (hours)	80	100
Oil Level Block (hours)	10	20
New Oil Additions (Total ml)	3304	1880
Load (Nm)	200	250
Oil Temp. °C	155	150
Coolant Temp. °C	122	115
Inlet Air Temp °C	27	35

First test

The first Sequence IIIG development test was run at SwRI in June of 2001 on ASTM Reference Oil 433-1. The piston ring gaps were set the same as in the Sequence IIIF test. The ring gapping strategy used in the Sequence IIIF test is called reverse gapping, where the second ring has a smaller gap than the gap on the top ring. Using this gapping strategy, the end of test viscosity increase for this test was 6467%, camshaft and lifter average wear was 132 microns, and the weighted piston deposits (WPD) were 2.21 merits (See Lab/Run #SR01 in Table 2). Clearly the severity had increased relative to the Sequence IIIF test.

Conventional Ring gapping

Understanding that conventional ring gapping reduces blow-by and test severity, and that the development groups desire was to have the test hardware and clearances as close to production as possible, the next two tests, one at SwRI and one at P&E, were run with conventional ring gapping i.e., 0.025 inch top and 0.042 inch second. These tests were again run on ASTM Reference Oil 433-1. The results were still more severe than Sequence IIIF test results, but much less severe on all parameters than the first Sequence IIIG test (See Lab/Run #'s SR02 and PE01 in Table 2). As a result of this testing, the conventional ring gapping strategy was retained throughout the remainder of the Sequence IIIG development program.

Primary ZDP gives lower wear than secondary ZDP

A cooperating additive supplier was asked to supply a modern analog of the Sequence IIID ASTM Reference Oil 403; i.e., it was to contain the same primary ZDP package as the original oil , but with a more current DI system. Reference oil 403 was a borderline wear oil in the Sequence IIID test, and a failing wear oil in the Sequence IIIE test. When tested under Sequence IIIG test conditions, the oil became too viscous to measure (TVTM), had terrible Weighted Piston Deposits (WPD), and only generated 14 microns average camshaft and lifter wear (See Lab/Run #SR03 in Table 2). Another additive supplier was asked to give us a fortified version of ASTM Reference Oil 1006. Once again the oil broke and became very viscous, and the wear was only 27 microns average (See Lab/Run #SR04 in Table 2).

These results were puzzling, as many additive and oil experts had expected that any oil formulated with primary ZDP would generate high wear. However, a review of the original ASTM Reference Oil 403 fleet test data (also known as Reference Oil 200) clearly stated that oil 403 generated high wear in Chevrolet 350 V-8 engines, only when run on leaded fuel. When run with unleaded fuel the wear results were much lower. The Sequence IIIG as well as the IIIF use unleaded fuel. Thus low wear should be expected with the primary ZDP oil, and indeed low wear was observed.

Runs with 0.03% phosphorus give high wear

Another oil was then tested that contained 0.03% phosphorus from secondary ZDP. It was expected to generate high wear. Two tests were run and both tests did produce high wear (See Lab/Run #'s SR05 and SR06 in Table 2).

Spring tension increased

A run was then made with the same oil as used in the previous two runs, only with the ZDP boosted to a phosphorus level of 0.05%. The Sequence IIIG test clearly showed a response to the increase in phosphorus content by generating lower wear results.
Up to this point all testing had been conducted with Sequence IIIF valve springs calibrated to an open load of 180 lbs. which is considered abnormally low for pushrod engines. These special low load springs were originally developed for the 52100 steel valve lifters used in the Sequence IIIF test and were retained when that test was redeveloped. The redevelopment included the switch to ACI lifters.

At this point the valve springs were switched to production 205 lb. springs. This change was made for two reasons. One was to more closely match flat tappet pushrod valve train design practice, and the second was to increase wear severity.

Wear appears to be dialed in

Tests were then run with the 0.05% phosphorus oil and the same oil boosted all the way to 0.095% phosphorus (Lab/Run #'s PE03 and PE04, respectively, in Table 2). The 0.05% phosphorus oil gave high wear and the 0.095% phosphorus oil gave low wear. The test appeared to be responding to ZDP as expected.

Table 2 – Initial Non-Phosphated Camshaft Results

Lab/Run#	Viscosity	Test Oil	Spring Load	% Vis. Inc.	Avg. Wear	WPD	PSV	Oil Cons.
SR/01	5W-30	433-1	180	6467	132	2.21	7.87	4.43
SR/02	5W-30	433-1	180	287	28	2.62	7.96	3.85
PE/01	5W-30	433-1	180	130	37	2.82	8.32	4.09
SR/03	5W-30	403 Reform	180	TVTM	14	1.6	8.2	TVTM
SR/04	5W-30	1006 Reform	180	1077	27	2.36	8.62	3.61 @ 80h
SR/05	5W-30	0.03 Phos.	180	105	105	3.92	8.4	3.74
SR/06	5W-30	0.03 Phos.	180	156	267	2.85	7.79	3.7
PE/02	5W-30	0.05 Phos.	180	130	26	3.16	8.69	3.78
PE/03	5W-30	0.05 Phos.	205	133	153	3.32	8.52	3.99
PE/04	5W-30	0.095 Phos.	205	176	16	3.23	8.84	4.55

The importance of scuff rediscovered

It appeared that the test conditions were fairly close to being finalized, and preliminary testing of prototype GF-4 oils and licensed GF-3 oils began. The results were somewhat confusing and not entirely as expected. By this point some of the task force members had became convinced that high camshaft and lifter wear observed in some of the early testing was solely attributable to break-in type scuffing. PerkinElmer ran a series of ten minute oil leveling tests, on various oils using a new cam and lifter set each test to prove that break-in scuffing was a problem and determine if this early failure mechanism could be overcome. OH Technologies (CPD) suggested phosphating the camshaft, as was done in the Sequence IIID to reduce the tendency of scuffing during break-in. GM resisted phosphating the camshaft because of the difficulties encountered with the Sequence IIID test due to variations in camshaft phosphate coatings which resulted in variations in wear severity and precision. Additional testing to reduce scuffing included running in the camshaft with a camshaft break-in lube (GM Engine Assembly Prelube Pt.# 1052367), using an oiling bar to squirt oil directly onto the camshaft, and switching to a higher carbide camshaft material. In the end GM agreed that it would be necessary to phosphate camshafts to eliminate scuffing.

Phosphated camshafts

In looking back at the earlier runs, it was apparent that many runs had encountered break-in scuffing. Tests that encountered scuffing seemed to exhibit elevated iron concentrations at the end of the ten-minute timing runs. The decision to phosphate the camshafts to reduce break-in scuffing was reported to the ILSAC/OIL Committee and no one disagreed. Since the early 1950s, all production camshafts have been phosphate coated to reduce scuffing. With this change, the valvetrain of the Sequence IIIG test engine more closely represented late 1980s production push rod engines, with no modifications to increase test severity other than setting the first and second piston ring gaps at the high end of the production limits to increase crankcase blow-by.

Ten tests were then run with phosphated camshafts on various oils to look at wear, viscosity increase, and weighted piston deposits. These test results are shown in Table 3. The viscosity increases varied between 107% to TVTM, the weighted piston deposits varied from 2.37 to 5.36, and wear varied from 12.8 to 66.0 microns. Three of the tests were run with the same oil but with varying amounts of ZDP. The run with 0.03% phosphorus gave an average cam and lifter wear of 51 microns while at 0.05% and 0.095% phosphorus the wear was about 18 microns. It appeared that the test was progressing well and meeting expectations.

Table 3 – MK Phosphated Camshaft Results

Lab/Run#	Viscosity	Test Oil	% Vis. Inc.	Avg. Wear	WPD	PSV	Oil Cons.
PE/08	5W-30	0.03 Phos.	170	51.3	3.53	9.07	3.78
SR/15	5W-30	0.05 Phos.	107	17.7	3.30	9.26	3.32
PE/10	5W-30	0.095 Phos.	166	17.6	3.35	9.45	6.09
SR/16	15W-40	CI-4 (Gr.I) (80hrs.)	TVTM	66.0	3.97	8.86	2.79
SR/17	15W-40	CI-4 (Gr.II)	1657	16.1	5.36	9.56	3.95
PE/11	10W-30	GF-3	175	17.6	3.24	8.21	3.40
SR/14	5W-20	GF-3 TMC Ref. 538	118	12.8	3.50	9.16	4.20
PE/12	5W-20	GF-3 TMC Ref. 538	117	14.2	3.70	8.93	3.86
SR/18	5W-30	TMC Ref. 433-1	150	62.0	2.37	7.19	3.51
PE/13	5W-30	TMC Ref. 433-1	228	35.9	2.76	8.52	4.36

NF-190 camshafts

The test development group reviewed the phosphating process on site, was not completely satisfied, and asked the supplier for improved process control. The camshaft supplier readily agreed to make the required changes to their process. The camshafts from this new process were designated NF-190. Wear with these NF-190 camshafts was greatly increased over the first phosphated camshafts tested. These results were not completely unexpected as it was recognized that the heavy phosphating used on the Sequence IIID camshafts was a prime factor in the wear severity of that test. The development group wanted camshafts with a consistent fine grain, light phosphate coating that would protect from scuffing while not contributing to higher wear severity as in the Sequence IIID test. The CPD responded to this request by working with an alternate facility which specializes in phosphate coatings. Together they developed an alternate process, which very closely monitors every step including time and chemical concentrations thereby assuring uniform coatings.

NF-200 camshafts

The camshafts phosphated using this alternate process were identified as NF-200 camshafts. Twenty runs were made with NF-200 camshafts as shown in Table 4. These included oils supplied by various additive suppliers as potential oils for inclusion in the precision matrix. Wear ranged from below 20 microns for oil 538 to about 75 microns for an oil formulated with an aryl ZDP (GM-2). In virtually all of the early Sequence III camshaft and lifter wear investigations, oils formulated with aryl ZDP were the only ones identified as truly poor field performers. An aryl ZDP oil was the failing oil in the

Sequence IIID test, which was the last Sequence III test which used a phosphated camshaft. NF-200 camshafts run on GF-3 oils typically generated wear results of about 40 microns. Additional NF-200 testing showed, very good oils generated wear results near 20 microns, good oils generated results of 40 microns, and poor oils generated results of 75 microns. Thus, the test appeared to be providing good wear discrimination.

Table 4 – NF-200 Phosphated Camshaft Results

Lab/Run#	Viscosity	Test Oil	% Vis. Inc.	Avg. Wear	WPD	PSV	Oil Cons.
OHT/PE	5W-30	TMC Ref. 433-1	191	37.7	2.94	8.46	4.09
SR/19	5W-30	TMC Ref. 433-1	TVTM	98.9	3.13	8.51	4.31
PE/20	5W-30	TMC Ref. 433-1	153	37.8	3.14	8.64	4.13
OHT/SR	5W-20	GF-3 TMC Ref. 538	91.6	17.9	2.90	8.73	3.80
SR/20	5W-20	GF-3 TMC Ref. 538	92.7	19.3	2.89	8.25	3.12
PE/16	5W-20	GF-3 TMC Ref. 538	118.9	16.8	3.30	9.04	4.61
PE/17	5W-20	GF-3 TMC Ref. 538	101.2	15.8	2.64	8.10	3.29
PE/18	5W-30	0.03 Phos.	114	36.7	3.24	8.48	3.66
SR/21	5W-20	Cand. Ref Oil A-1	106	44.6	3.74	8.46	3.50
PE/19	5W-30	Cand.Ref Oil B-1	91	21.0	4.21	8.70	3.67
SR/22	5W-30	Cand.Ref Oil B-1	155	42.2	4.06	8.66	N/A
SR/23	5W-20	Cand.Ref Oil C-1	159	43.2	2.97	7.88	3.73
PE/21	5W-20	Cand.Ref Oil C- 2	166	45	3.40	8.38	3.88
SR/24	5W-20	Cand.Ref Oil C-1	133	41.2	3	8.31	3.54
SR/25	5W-30	Cand.Ref Oil B-1	TVTM	56.1	2.82	8.62	4.19
PE/22	5W-30	Cand.Ref Oil B- 2	148	38.4	4.37	9.20	4.64
SR/25A	5W-30	Cand.Ref Oil B- 2	157	41.8	3.67	8.8	3.89
SR/26	5W-20	GM-2	168	69.9	2.84	7.6	3.64
PE/23	5W-20	GM-2	146	79.5	3.29	8.59	3.57
PE/XX	5W-20	Cand.Ref Oil C	228	32.8	3.19	8.96	4.30

0.03% phosphorus oil now passes on wear

The secondary ZDP 0.03% phosphorus test oil switched from a failing wear oil to a passing wear oil when the test cams were switched to phosphated camshafts. This demonstrates that ZDP is a very powerful anti-scuff additive and very effective in protecting non-phosphated camshafts during break-in. The Sequence IIIG test will be one of two wear tests in the ILSAC GF-4 specification, and it is almost certain that the 0.03% phosphorus oil would not pass the Sequence IVA test. It is important to remember that the Sequence IIIG test measures high temperature wear and not low temperature or scuffing wear. Some may classify the high temperature wear in the Sequence IIIG test as chemical etching wear.

Oil filters

Early on during the development of the test it was discovered that future supply of AC PF47 oil filters could contain different types of filter media. It was decided that the existing inventory should be used only in the Sequence IIIF test. The CPD found an alternate filter source assuring the use of consistent filter media, and these filters were incorporated into the testing. During the twenty runs made with the NF-200 camshafts, sporadic oil filter plugging was encountered. Oil filter plugging is easily identified in Sequence IIIF and IIIG testing through the advent of a CPD developed, externally mounted by-pass valve with instrumentation designed to detect oil flow through the by-pass valve. This issue was extensively investigated, but no conclusive finds were made. For the matrix runs a last minute change was made, and filters from the same supplier but with a larger nominal micron rating (similar construction to the AC PF-47 filter) were used. The plugging problem was not encountered during the precision matrix, and these larger nominal pore size filters were incorporated into the test.

Precision matrix (IIIG Operating Procedure Draft 2D)

GM originally suggested that the precision matrix be run on GF-3, GF-3+, and GF-4 prototype oils. A task force was formed under the Sequence III Surveillance Panel to select the matrix oils. Mr. Gordon Farnsworth, Chairman of the task force, felt that it was important to have two different prototype oil chemistries included in the matrix. Thus the GF-3 oil was removed from the matrix. The GF-3+ oil selected was ASTM Reference Oil 538 (SAE 5W-20), later renamed for the Sequence III as ASTM Reference Oil 438. The second oil was identified as ASTM Reference Oil 434 (SAE 5W-30) that in initial Sequence IIIG testing was a good performer. The third oil was identified as ASTM Reference Oil 435 (SAE 5W-20) that in initial Sequence IIIG testing was a borderline performer.

Each oil was scheduled to run 4 times on 2 stands at both SwRI and PEAR. Two separate manganese phosphate batches of camshafts were also scheduled into the matrix. A task force under the Sequence III Surveillance Panel and the Passenger Car Engine Oil Classification Panel designed the matrix. A statistical analysis of the Precision Matrix results was conducted under the guidance of Mr. John Zalar of the ASTM Test Monitoring Center (TMC) and a partial summary of their report is included in this report as Attachment 1. The full report is available from the TMC website.

A summary of the matrix results is shown as Table 5. The data are arranged in the following order, test oil and run sequence by oil. The data include laboratory code, oil code, ACLW (average camshaft and lifter wear), VIS (percent viscosity increase at end of test), WPD (weighted piston deposits, merits), APV (average piston varnish deposits, merits), and OIL CONS (end of test total oil consumption, liters). The table also includes, standard deviation, range, and overall average for each parameter.

Table 5 – Precision Matrix Summary

RUN SEQ.	LAB	OIL	ACLW	VIS	WPD	APV	OIL CONS
4	G	434	41.1	133.3	3.15	8.61	3.86
5	A	434	26.2	89.9	5.83	9.43	3.98
7	G	434	43.7	127.6	3.39	8.81	4.23
11	A	434	37.1	249.5	4.77	8.74	4.65
13	G	434	33.1	99.2	4.32	8.76	3.90
20	A	434	39.1	86.7	4.42	8.83	3.47
21	G	434	40.2	185.7	3.83	8.36	4.39
23	A	434	34.2	62.8	4.99	9.04	3.73
		ST.Dev.	**5.55**	**61.34**	**0.88**	**0.31**	**0.38**
		Range	**17.5**	**186.67**	**2.68**	**1.07**	**1.18**
		AVERAGE	**36.84**	**129.33**	**4.34**	**8.82**	**4.03**
1	A	435	45.8	172.2	3.26	8.84	3.74
2	G	435	30.9	163.4	2.90	8.57	3.86
9	A	435	31.6	222.2	3.31	7.98	4.22
10	G	435	26.8	279.0	3.30	8.76	4.30
15	G	435	48.7	304.8	4.12	8.11	4.31
18	A	435	33.0	176.4	3.92	8.32	4.25
19	G	435	34.6	230.2	2.97	8.63	4.21
22	A	435	46.8	167.7	3.28	8.36	3.79
		ST.Dev.	**8.47**	**54.33**	**0.43**	**0.31**	**0.24**
		Range	**21.9**	**141.4**	**1.22**	**0.86**	**0.57**
		AVERAGE	**37.28**	**214.48**	**3.38**	**8.45**	**4.09**
3	A	438	14.4	102.3	3.04	8.96	3.62
6	G	438	16.8	132.6	3.68	9.39	4.27
8	A	438	21.2	111.7	3.14	8.86	3.47
12	G	438	15.3	143.2	2.85	8.91	4.33
14	G	438	20.8	120.6	3	8.26	3.87
16	G	438	15.6	91.7	4.17	8.65	3.41
17	A	438	22	88.6	3.08	9.2	3.32
24	A	438	21.4	90.5	3.26	8.82	3.28
		ST.Dev.	**3.20**	**20.57**	**0.44**	**0.34**	**0.42**
		Range	**7.6**	**54.6**	**1.32**	**1.13**	**1.05**
		AVERAGE	**18.44**	**110.15**	**3.28**	**8.88**	**3.70**

Industry statisticians from Chevron Oronite, Infineum, and the Lubrizol Corporation performed the statistical evaluation of the Precision Matrix data. Twenty-four operationally valid tests were used in the evaluation. Three tests were invalidated by one of the testing laboratories due to high oil consumption. Initial model factors for the evaluation included laboratory, stand, oil, and camshaft phosphate batch. After evaluation, the statisticians did not believe there was enough statistical evidence to include stand within laboratories or camshaft phosphate batch in the final model. Therefore the final model fit for all parameters includes only laboratory and oil.

Two methods for comparing percent viscosity increase were used to analyze the data, natural log and adjusted natural log for oil consumption. Two methods for comparing weighted piston deposits were used to analyze the data, the normal weighting factor method and a new weighting factor method. A comparison of the two weighting methods is shown on page 31 of the statistical evaluation attachment.

During review of the Precision Matrix data at the Sequence III Surveillance Panel meeting on June 10, 2003, the panel members moved to accept the results of the Precision Matrix, deciding to retain the standard methods for calculating percent viscosity increase and weighted piston deposits. The panel members also moved to recommend the Sequence IIIG test for inclusion into GF-4 to the Passenger Car Engine Oil Classification Panel.

To address concerns about oil consumption correlation to all other parameters, a task force was formed under guidance of the American Chemistry Council (ACC). Their task was to investigate the need for a correction equation based on oil consumption correlations to percent viscosity increase and MRV test results. During their investigation, the Sequence III Honing Task Force Chairman, Charlie Leverett, identified enhancements to the honing process that improved the oil consumption variability at PEAR. The information was provided to the Test Sponsor and the Test Monitoring Center who after reviewing the process enhancements decided that they were within the procedural guidelines and better classified as refinements to the honing process. The refinements were outlined and presented to the Sequence III Operations and Hardware Subpanel on October 28, 2003. The panel moved to adopt the refinements and forward their recommendation to the Sequence III Surveillance Panel on October 29, 2003. As a result of this information, the ACC task force concluded that "the effort by the test sponsor, the test labs, the TMC, and the Sequence IIIG Surveillance Panel and industry stakeholders have sufficiently improved the OC precision of the Sequence IIIG to make an equation unnecessary at this time."

To further address all concerns, the Sequence III Surveillance Panel moved to have all CV-616 industry honers calibrated on-site by a qualified Sunnen technician followed by an industry honer specific training workshop and implement the honing refinements into the Sequence III procedure. The honing workshop was conducted at Lubrizol on December 9 and 10, 2003 and the test laboratories were to bring the honing refinements into their laboratory with a successful reference.

Publication of this report officially completes the activities of the IIIG Development Group. All future investigative activity will be at the direction of the Sequence III Surveillance Panel and / or the Passenger Car Engine Oil Classification Panel under ASTM D02.B0.01.

General Motors would like to thank Southwest Research Institute, PerkinElmer Automotive Research, and OH Technologies for their dedicated assistance and engineering expertise during the development of the IIIG test. General Motors would also like to thank the many others i.e., additive suppliers, oil companies, parts suppliers, committee members, and many others that contributed to the successful completion of this test.

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Statistical Summary of the Sequence IIIG Matrix

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June 6th, 2003

Outline

- Sequence IIIG Matrix Summary
- Matrix Design
- The data
- Correlations among parameters
- Statistical Analysis of the matrix data by parameter
- Summary of Means and Standard Deviations by Oil
- Appendix
  - Transformation Analysis and Residual Plots by Time Order
  - Plots of the data for each parameter by Lab and by Stand
  - Summary of unusual observations by parameter
  - Correlation of MRV and PVIS:
    - Used Oil MRV over Fresh Oil MRV versus PVIS
    - Used Oil MRV over Fresh Oil MRV versus PVIS by Oil

Sequence IIIG Matrix Summary

- Matrix included 24 operationally valid tests
  - Three other tests were invalid due to high oil consumption
- Model factors considered for all analysis include
  - Lab (A,G),
  - Stand within Lab,
  - Oil (434, 435, 438)
  - Cam/Phosphate Batch (30203, 30422)
- Final model fits for all parameters include Lab and Oil
  - There was not enough Statistical Evidence to Include Stand within Lab or Cam/Phosphate Batch in the final models

Sequence IIIG Matrix Summary

- Parameters of study include
  - Percent Viscosity Increase (VIS),
  - Adjusted Percent Viscosity Increase (NVIS),
  - Average Camshaft plus Lifter Wear (ACLW),
  - Average Piston Varnish (APV),
  - Weighted Piston Deposits (WPD),
  - New Weighted Piston Deposits (NWPD),
  - Oil Consumption (OC),
  - Cold Crank Simulator Viscosity (CCS),
  - MRV Viscosity (MRV)

Sequence IIIG Matrix Summary

- Transformations to stabilize the variance
  - Natural Log Transformations indicated by Box-Cox Analysis for VIS (but not NVIS) and ACLW;
  - Inverse Square Root Transformation indicated for MRV

- There is a general high correlation among VIS, OC and MRV

- There are Lab effects in OC

- There is also an indication of a Lab effect in VIS and MRV.

- There is a Lab by Oil Interaction for Weighted Deposits

Sequence IIIG Matrix Summary

- Ep is Below the ACC Precision Target for Percent Viscosity Increase, WPD, NWPD (but closer), and CCS
- Ep is Above the ACC Precision Target for Adjusted Percent Viscosity Increase and ACLW
- Ep is just at the ACC Precision Target for APV and MRV (Depends)

Sequence IIIG Matrix

- 24 runs, 2 labs, 6 stands, 4 tests/stand
- Degrees of Freedom (Nested model)
  - Oil 2
  - Lab 1
  - Lab(Stand) 4
  - Cam batch 1
  - Mean 1
  - Error 15
- Variance Inflation Factor
  - Lab 3.00
  - Lab1(Stand2) 1.72
  - Lab1(Stand3) 1.72
  - Lab2(Stand2) 1.72
  - Lab2(Stand3 1.72
  - Oil2 1.42
  - Oil3 1.42
  - Cam Batch 1.00

Run	Laboratory	Stand	Oil	Cam Batch
1	SWRI	1	1	1
2	SWRI	1	2	1
3	SWRI	1	3	2
4	SWRI	1	1	2
5	SWRI	2	1	2
6	SWRI	2	2	1
7	SWRI	2	3	1
8	SWRI	2	2	2
9	SWRI	3	1	1
10	SWRI	3	2	2
11	SWRI	3	3	2
12	SWRI	3	3	1
13	PE	1	1	2
14	PE	1	2	2
15	PE	1	3	1
16	PE	1	1	1
17	PE	2	1	1
18	PE	2	2	2
19	PE	2	3	2
20	PE	2	2	1
21	PE	3	1	2
22	PE	3	2	1
23	PE	3	3	1
24	PE	3	3	2

TESTKEY	OIL	LAB	STAND	CAMBATCH	SAEVISC	PVIS	ACLW	APV	WPD	OILCON	CCS	MRV
47905	435	G	3	NF	5W20	163.4	30.9	8.57	2.9	3.86	11370	84800
47888	435	A	3	NF	5W20	172.2	45.8	8.84	3.26	3.74	16160	84500
47893	438	A	1	NF	5W20	102.3	14.4	8.96	3.04	3.62	10200	19300
47901	434	G	5	NJ	5W30	133.3	41.1	8.61	3.15	3.86	17200	48900
47884	434	A	2	NJ	5W30	89.9	26.2	9.43	5.83	3.98	15640	31900
47910	438	G	2	NF	5W20	132.6	16.8	9.39	3.68	4.27	9080	23700
47900	434	G	3	NF	5W30	127.6	43.7	8.81	3.39	4.23	23640	49200
47894	438	A	3	NJ	5W20	111.7	21.2	8.86	3.14	3.47	9920	20500
47906	435	G	5	NJ	5W20	279	26.8	8.76	3.3	4.3	8530	210700
47889	435	A	1	NF	5W20	222.2	31.6	7.98	3.31	4.22	19370	300200
47911	438	G	2	NJ	5W20	143.2	15.3	8.91	2.85	4.33	15400	30400
47902	434	G	3	NJ	5W30	99.2	33.1	8.76	4.32	3.9	19480	45600
47886	434	A	2	NF	5W30	249.5	37.1	8.74	4.77	4.65	22500	86400
47891	435	A	3	NJ	5W20	176.4	33	8.32	3.92	4.25	14040	91900
47913	438	G	3	NJ	5W20	91.7	15.6	8.65	4.17	3.41	9180	19000
47908	435	G	2	NJ	5W20	304.8	48.7	8.11	4.12	4.31	17540	400000
47883	434	A	1	NJ	5W30	86.7	39.1	8.83	4.42	3.47	19000	34200
47907	435	G	5	NF	5W20	230.2	34.6	8.63	2.97	4.21	17200	294000
47895	438	A	2	NF	5W20	88.6	22	9.2	3.08	3.32	8320	16700
47885	434	A	3	NF	5W30	62.8	34.2	9.04	4.99	3.71	20600	29000
47896	438	A	1	NJ	5W20	90.5	21.4	8.82	3.26	3.28	8550	18000
47914	438	G	5	NF	5W20	120.6	20.8	8.26	3	3.87	10530	20500
47890	435	A	2	NJ	5W20	167.7	46.8	8.36	3.28	3.79	15600	110100
48605	434	G	2	NF	5W30	185.7	40.2	8.36	3.83	4.39		

Sequence IIIG Correlations

Vis*	0.75	0.40	-0.58	-0.25	-0.27	0.74	0.16	**0.84**
0.50	NVIS	0.34	-0.55	-0.42	-0.41	0.12	-0.16	-0.64
0.10	0.48	ACLW*	-0.42	0.21	0.23	0.30	0.70	-0.71
-0.20	-0.24	-0.34	APV	0.28	0.32	-0.28	-0.20	0.57
-0.05	-0.20	-0.36	0.18	WPD	0.99	0.12	0.40	0.01
-0.10	-0.20	-0.36	0.26	0.99	NWPD	0.09	0.40	0.00
0.76	-0.16	-0.24	0.01	0.17	0.13	OC	0.45	-0.66
0.33	-0.06	0.31	-0.41	-0.18	-0.26	0.42	CCS	-0.49
-0.88	-0.37	-0.03	0.29	0.06	0.13	-0.74	-0.58	MRV*

Raw Data Correlations on Upper Triangle; Partial Correlations on Lower Triangle

Percent Viscosity Increase (VIS)

- Analyzed on Natural Log Scale
- Root Mean Squared Error=0.291911 (20 df)
- Some Statistical Evidence that the Labs Differ
- Strong Statistical Evidence that the Oils Differ

Percent Viscosity Increase (VIS)

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	434	435	438		
Oil 434		0.003	0.828	118.26	95.36 to 146.67
Oil 435	0.003		0.001	208.86	168.40 to 259.03
Oil 438	0.828	0.001		108.52	87.50 to 134.59

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	Lab A	Lab G			
Lab A		0.063		123.56	103.64 to 147.30
Lab G	0.063			156.17	131.00 to 186.18

Adjusted Viscosity Increase (NVIS)

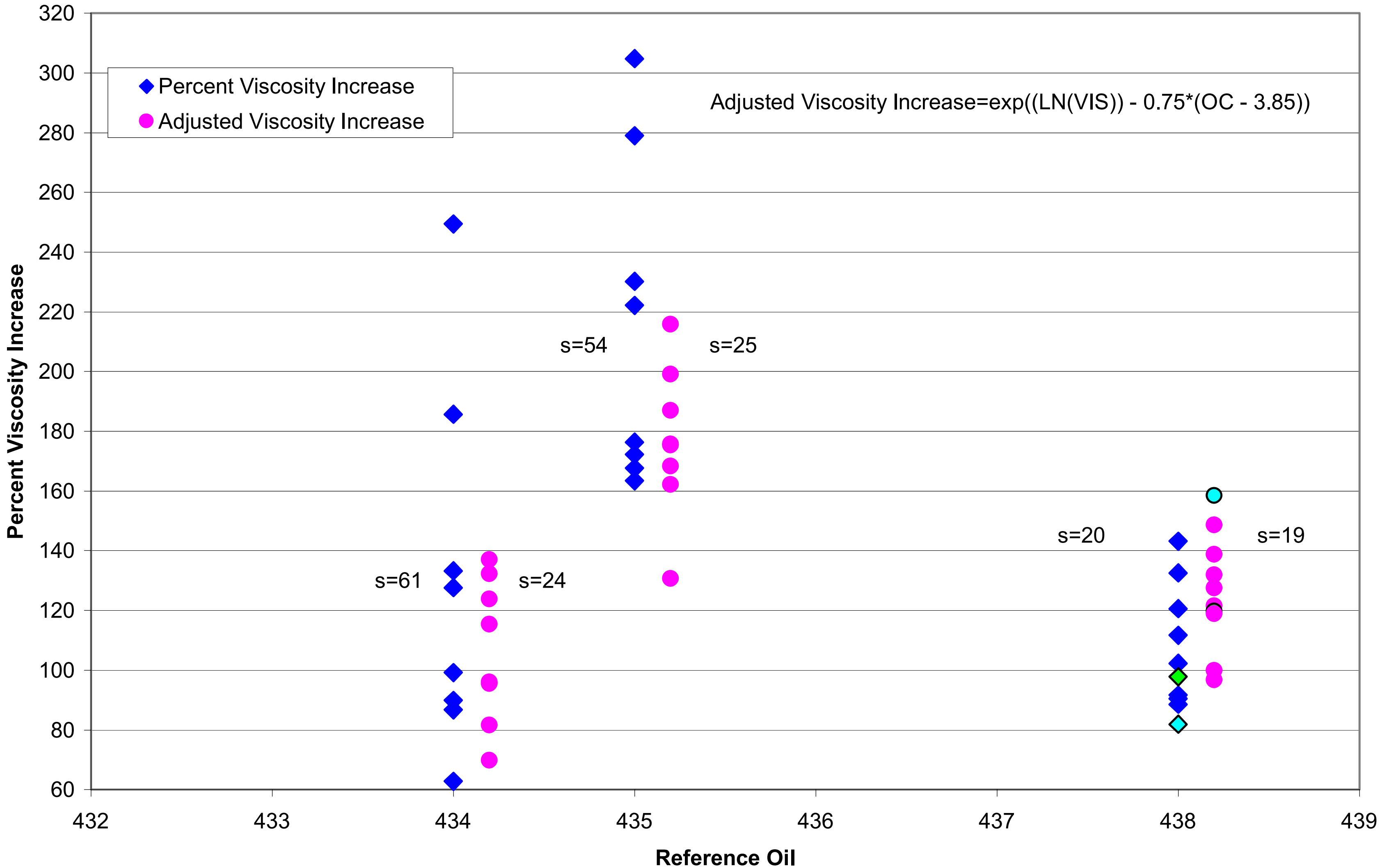
- Test Results Adjusted for Oil Consumption
- Root Mean Squared Error=23.34226 (20 df)
- Marked Improvement in Test Precision
- No Statistical Evidence that the Labs Differ
- Strong Statistical Evidence that the Oils Differ

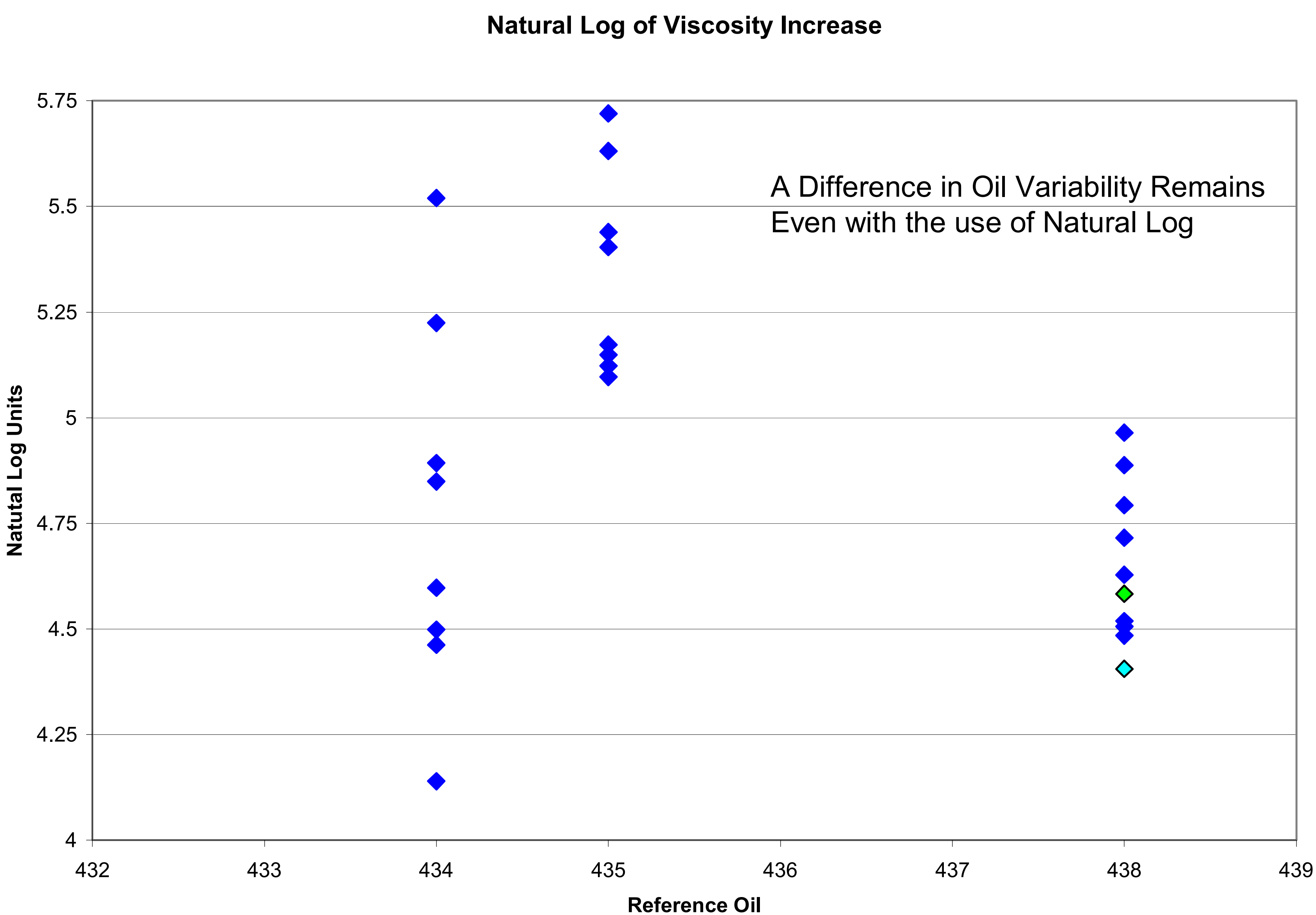
Adjusted Viscosity Increase (NVIS)

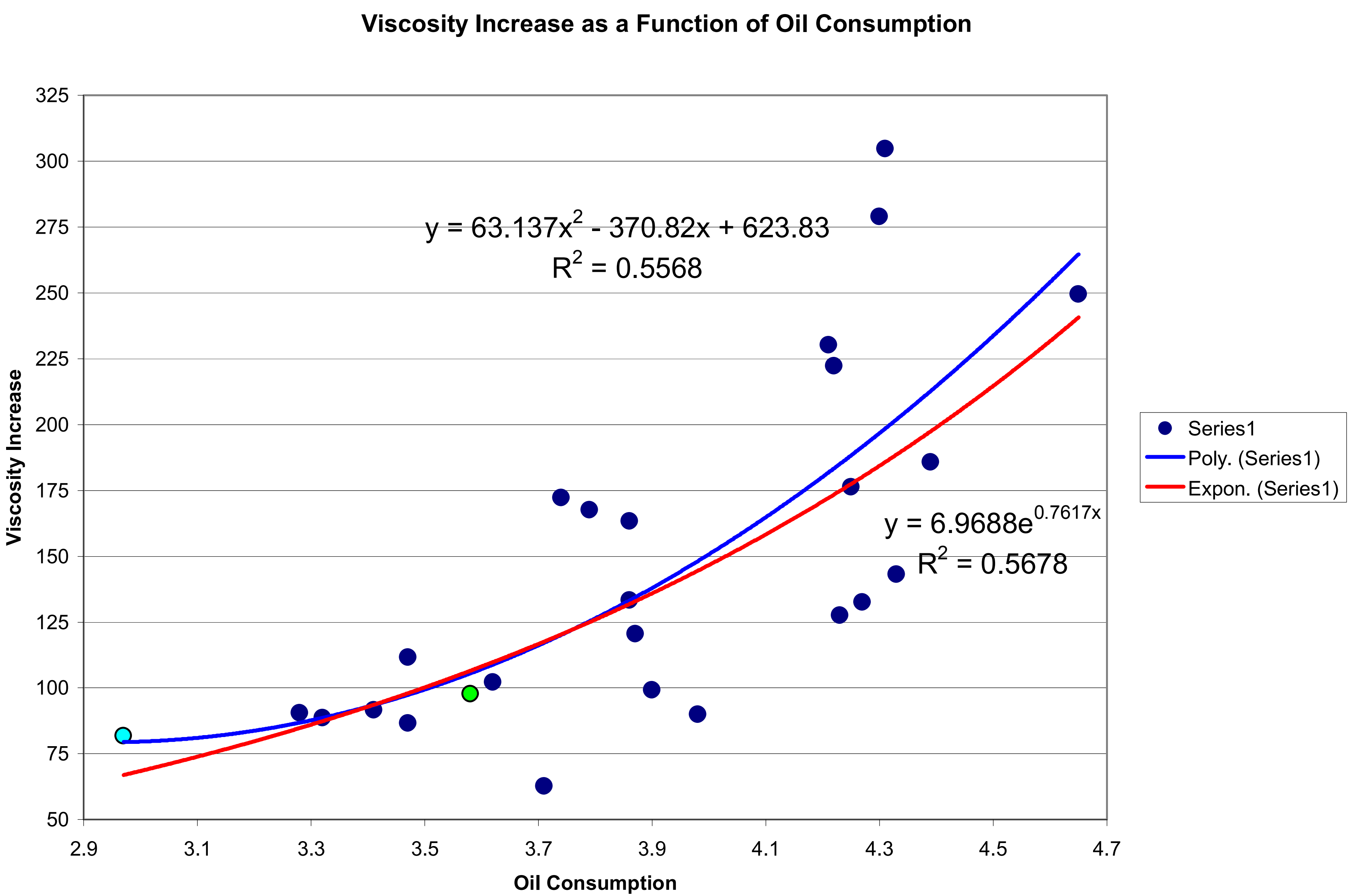
p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	434	435	438		
Oil 434		0.000	0.350	106.40	89.18 to 123.61
Oil 435	0.000		0.001	176.79	159.58 to 194.00
Oil 438	0.350	0.001		122.97	105.75 to 140.18

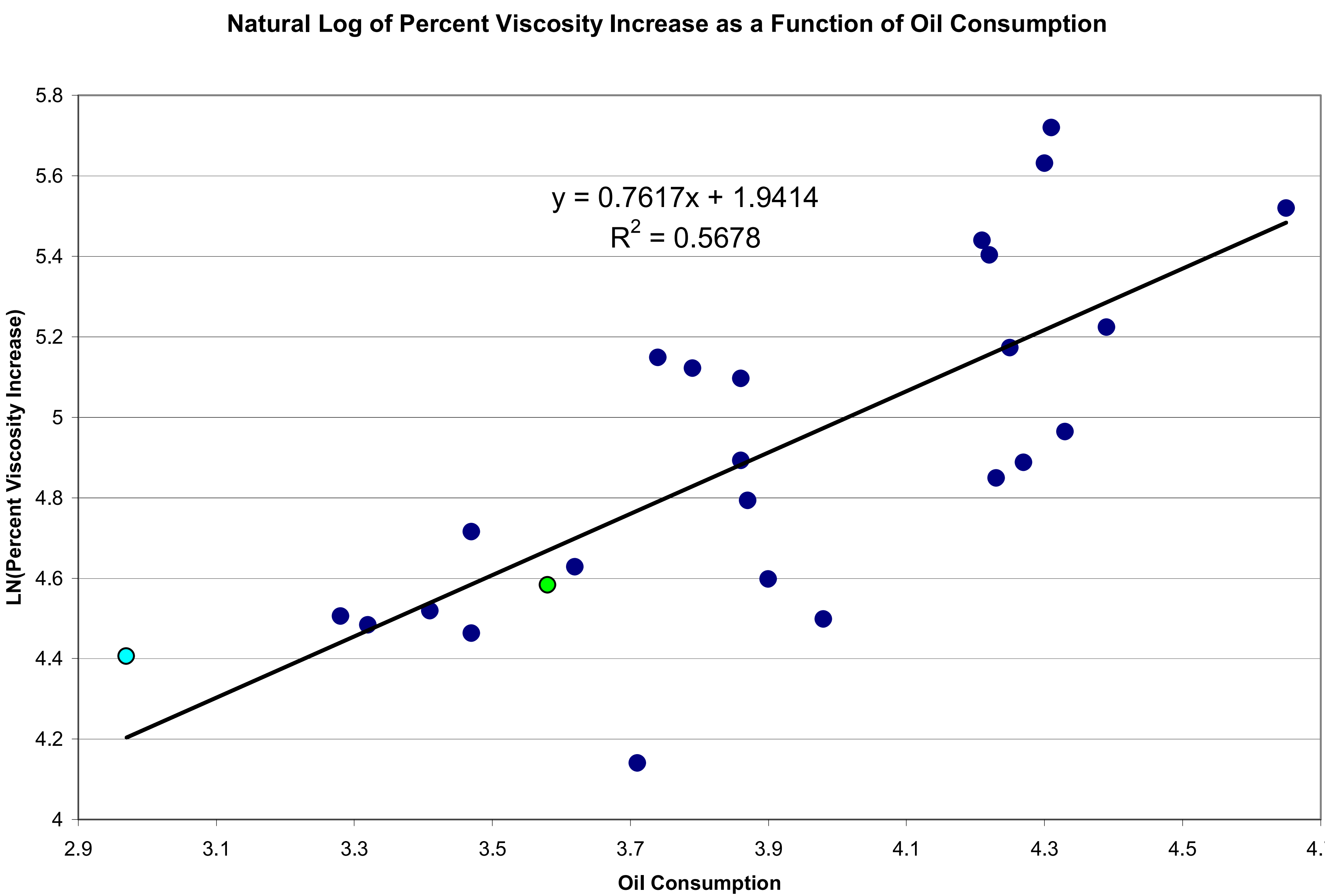
p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	Lab A	Lab G			
Lab A		0.744		133.81	119.75 to 147.86
Lab G	0.744			136.96	122.91 to 151.02

Comparison of Viscosity Increase Calculation Methods

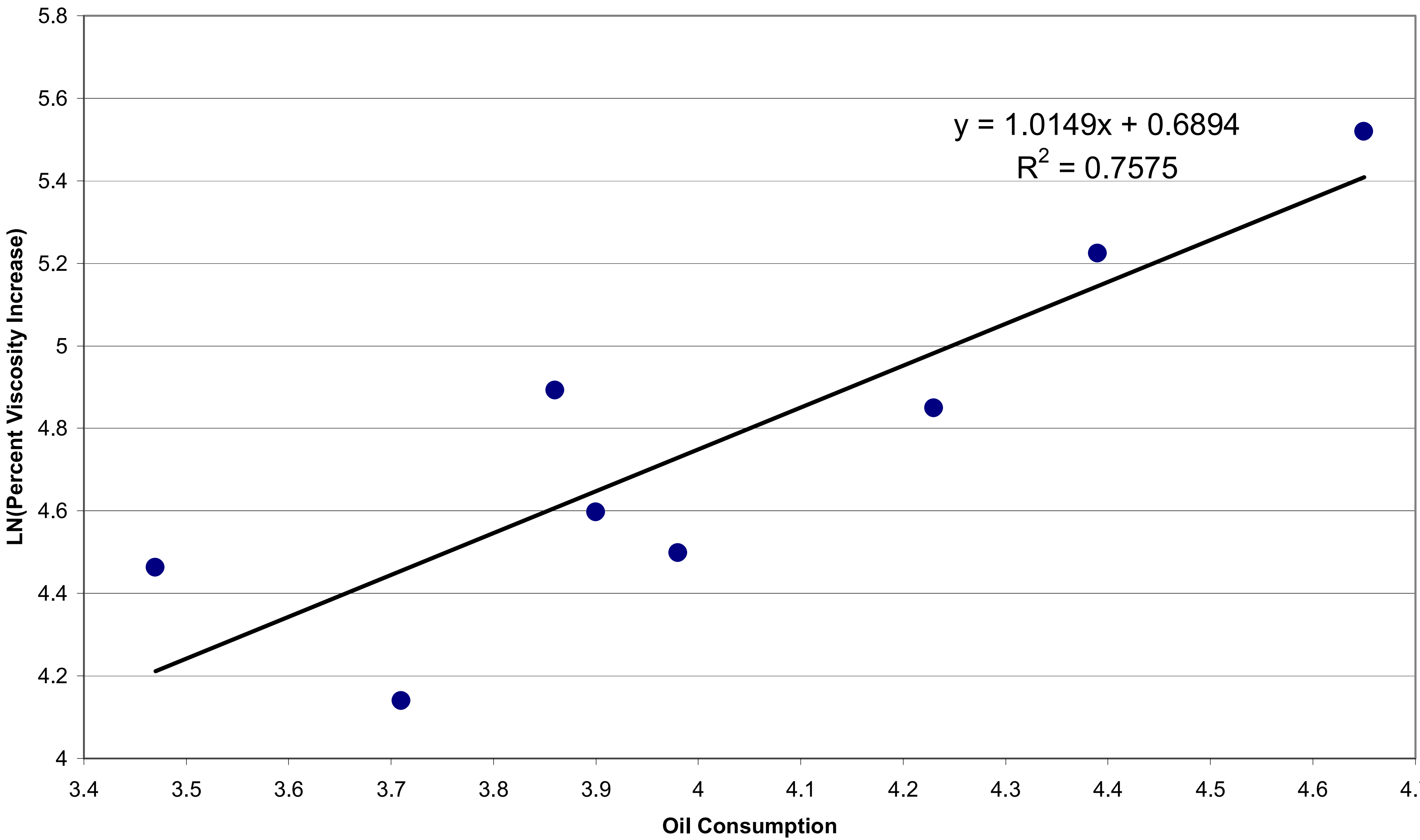



Natural Log of Viscosity Increase
A Difference in Oil Variability Remains
Even with the use of Natural Log
Natutal Log Units
5.75
5.5
5.25
5
4.75
4.5
4.25
4
432
433
434
435
436
437
438
439
Reference Oil

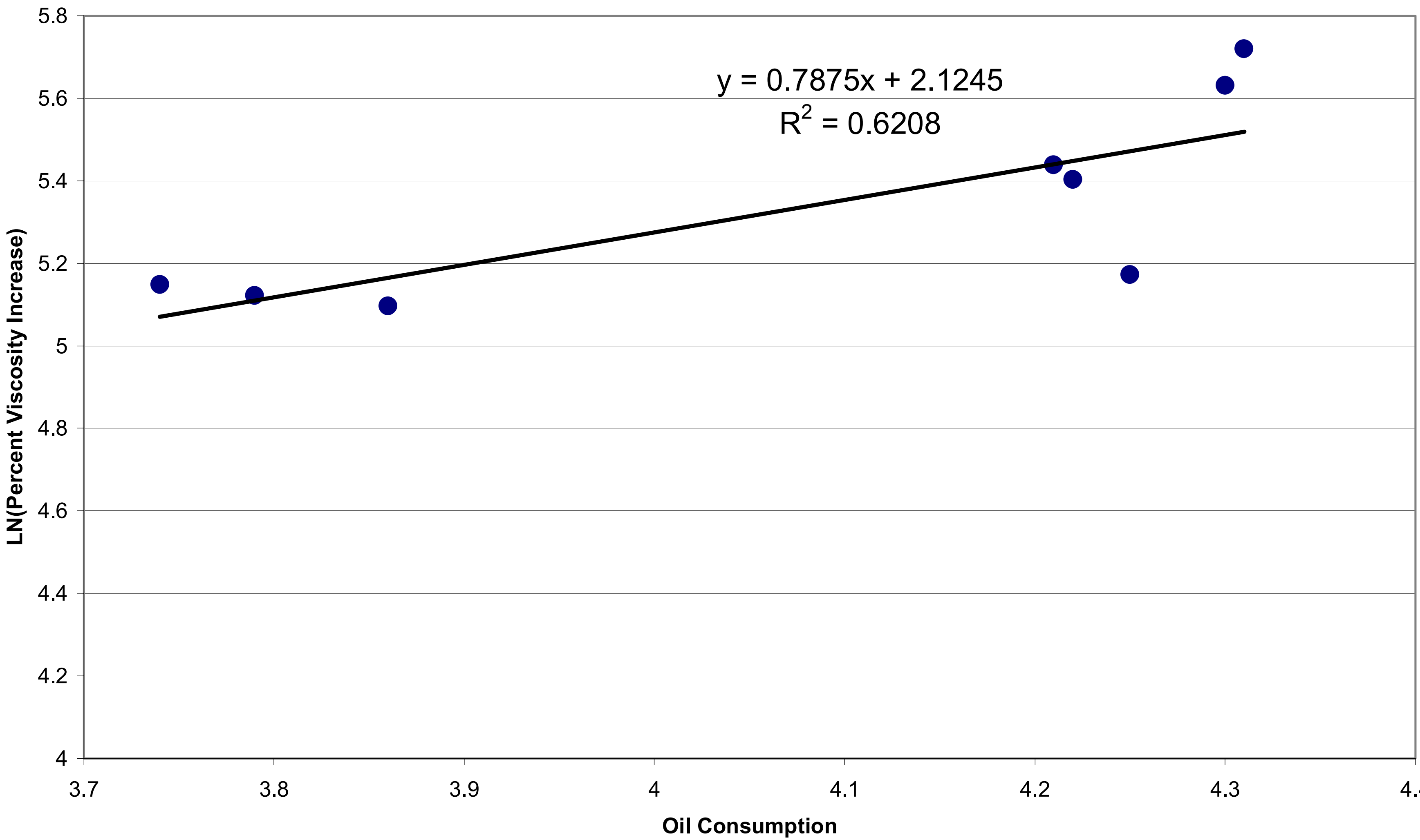

Viscosity Increase as a Function of Oil Consumption
y = 63.137x2 - 370.82x + 623.83
R2 = 0.5568
y = 6.9688e0.7617x
R2 = 0.5678
Viscosity Increase
325
300
275
250
225
200
175
150
125
100
75
50
2.9
3.1
3.3
3.5
3.7
3.9
4.1
4.3
4.5
4.7
Oil Consumption
Series1
Poly. (Series1)
Expon. (Series1)

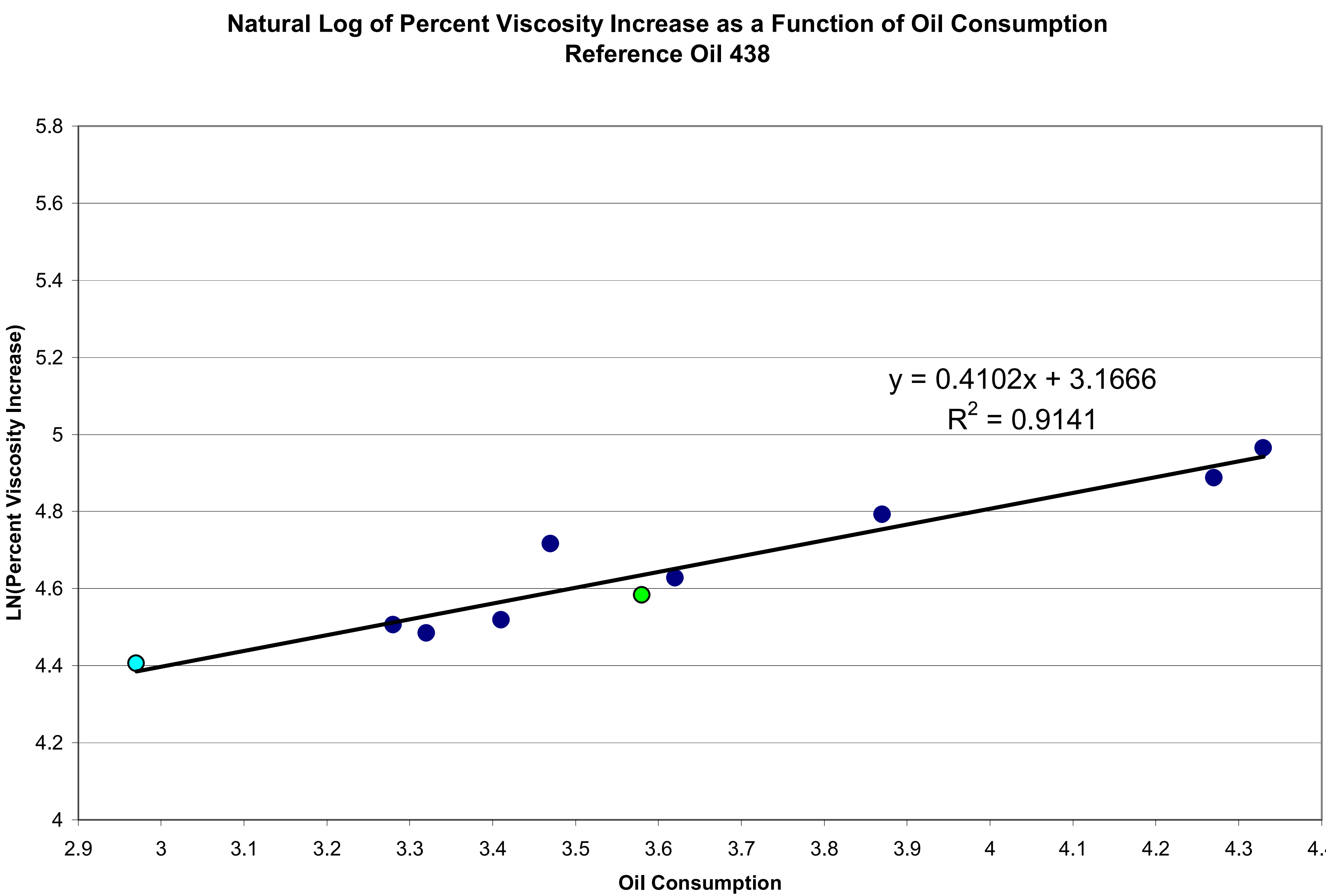

Natural Log of Percent Viscosity Increase as a Function of Oil Consumption
y = 0.7617x + 1.9414
R2 = 0.5678
LN(Percent Viscosity Increase)
5.8
5.6
5.4
5.2
5
4.8
4.6
4.4
4.2
4
2.9
3.1
3.3
3.5
3.7
3.9
4.1
4.3
4.5
4.7
Oil Consumption

Natural Log of Percent Viscosity Increase as a Function of Oil Consumption
Reference Oil 434



Natural Log of Percent Viscosity Increase as a Function of Oil Consumption
Reference Oil 435



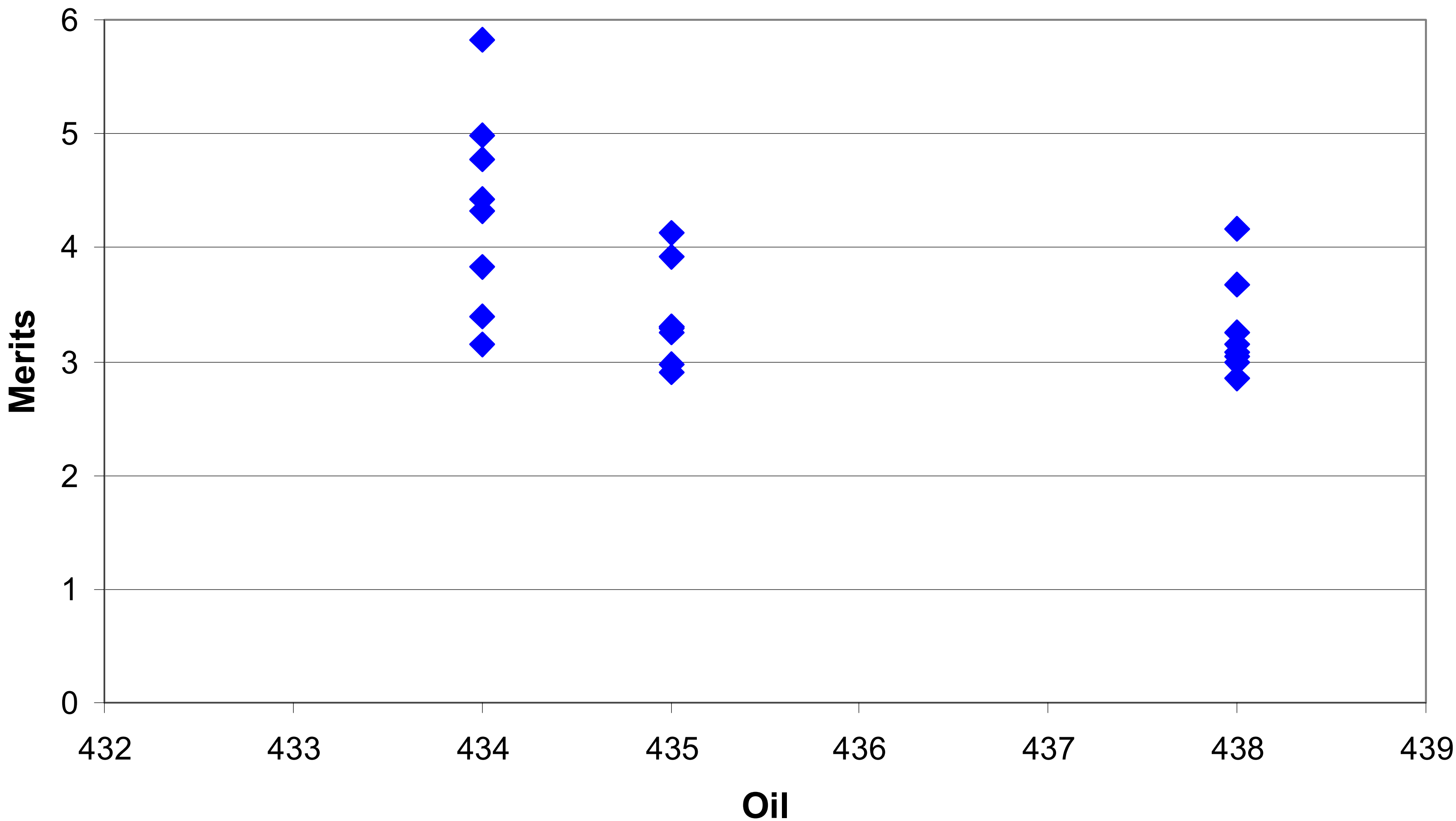

Natural Log of Percent Viscosity Increase as a Function of Oil Consumption
Reference Oil 438
y = 0.4102x + 3.1666
R2 = 0.9141
LN(Percent Viscosity Increase)
Oil Consumption
5.8
5.6
5.4
5.2
5
4.8
4.6
4.4
4.2
4
2.9
3
3.1
3.2
3.3
3.4
3.5
3.6
3.7
3.8
3.9
4
4.1
4.2
4.3
4.4

Weighted Piston Deposits (WPD)

- Root Mean Squared Error=0.597072 (20 df)
- There is Evidence of a Lab Difference ONLY for Oil 434
- Strong Statistical Evidence that the Oils Differ in Lab A, But No Evidence that Oils Differ in Lab G

LAB G Data Only p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	434	435	438		
Oil 434		0.667	0.813	3.67	3.03 to 4.31
Oil 435	0.667		0.964	3.32	2.68 to 3.96
Oil 438	0.813	0.964		3.43	2.79 to 4.06

WPD by Reference Oil



Weighted Piston Deposits (WPD)

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	434	435	438		
Oil 434		0.012	0.005	4.34	3.90 to 4.78
Oil 435	0.012		0.934	3.38	2.94 to 3.82
Oil 438	0.005	0.934		3.28	2.84 to 3.72

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	Lab A	Lab G			
Lab A		0.130		3.86	3.50 to 4.22
Lab G	0.130			3.47	3.11 to 3.83

New Weighted Piston Deposits (NWPD)

- This is one Example of a Weighting Change
- Root Mean Squared Error=0.466414 (20 df)
- There is Evidence of a Lab Difference ONLY for Oil 434
- Strong Statistical Evidence that the Oils Differ in Lab A, But No Evidence that Oils Differ in Lab G

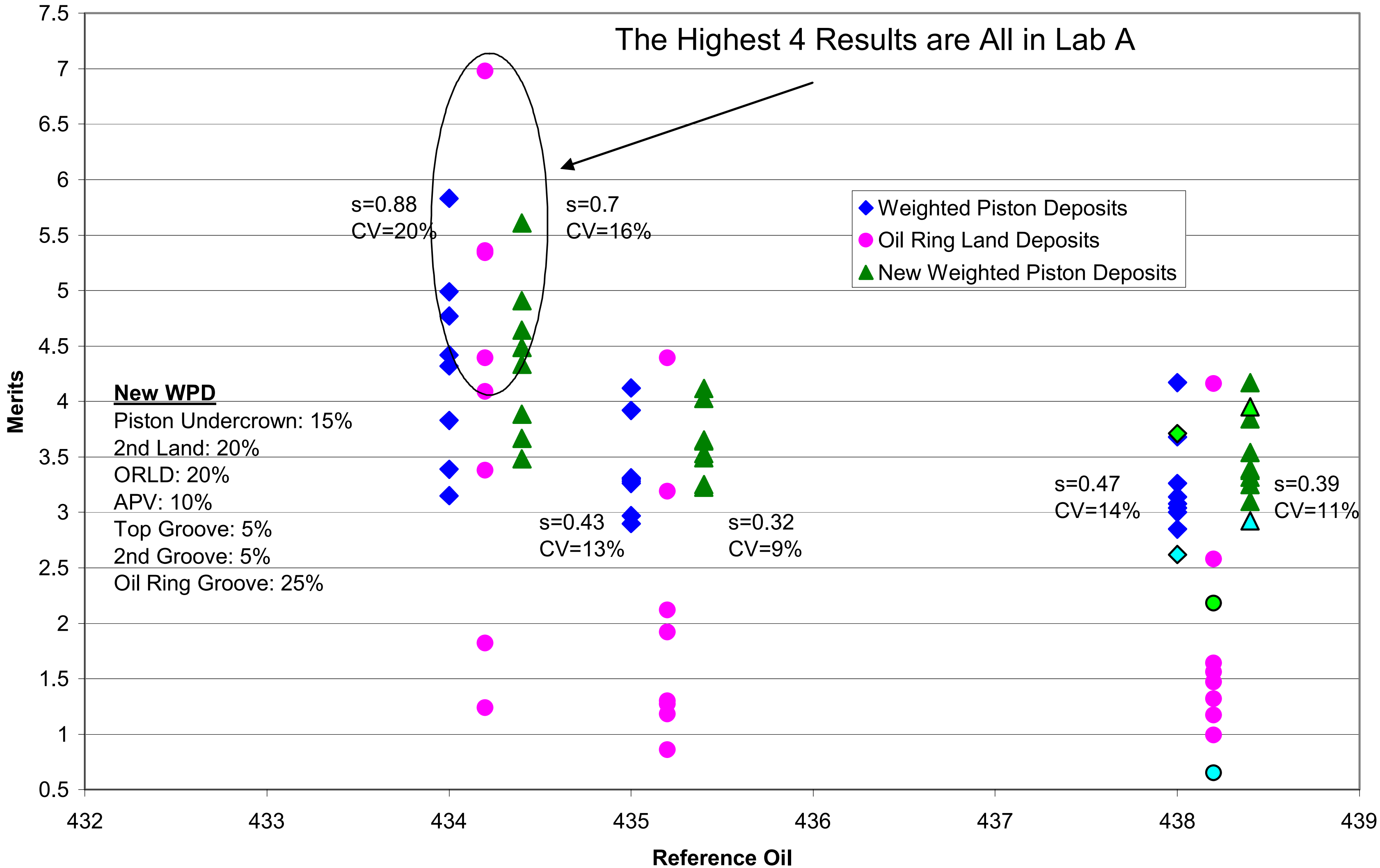
LAB G Data Only p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	434	435	438		
Oil 434		0.644	0.698	3.84	3.35 to 4.33
Oil 435	0.644		0.995	3.56	3.07 to 4.05
Oil 438	0.698	0.995		3.59	3.10 to 4.08

New Weighted Piston Deposits (NWPD)

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	434	435	438		
Oil 434		0.011	0.003	4.38	4.03 to 4.72
Oil 435	0.011		0.868	3.62	3.27 to 3.96
Oil 438	0.003	0.868		3.50	3.15 to 3.84

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	Lab A	Lab G			
Lab A		0.096		4.00	3.72 to 4.28
Lab G	0.096			3.67	3.38 to 3.95

Comparison of Weighted Deposits



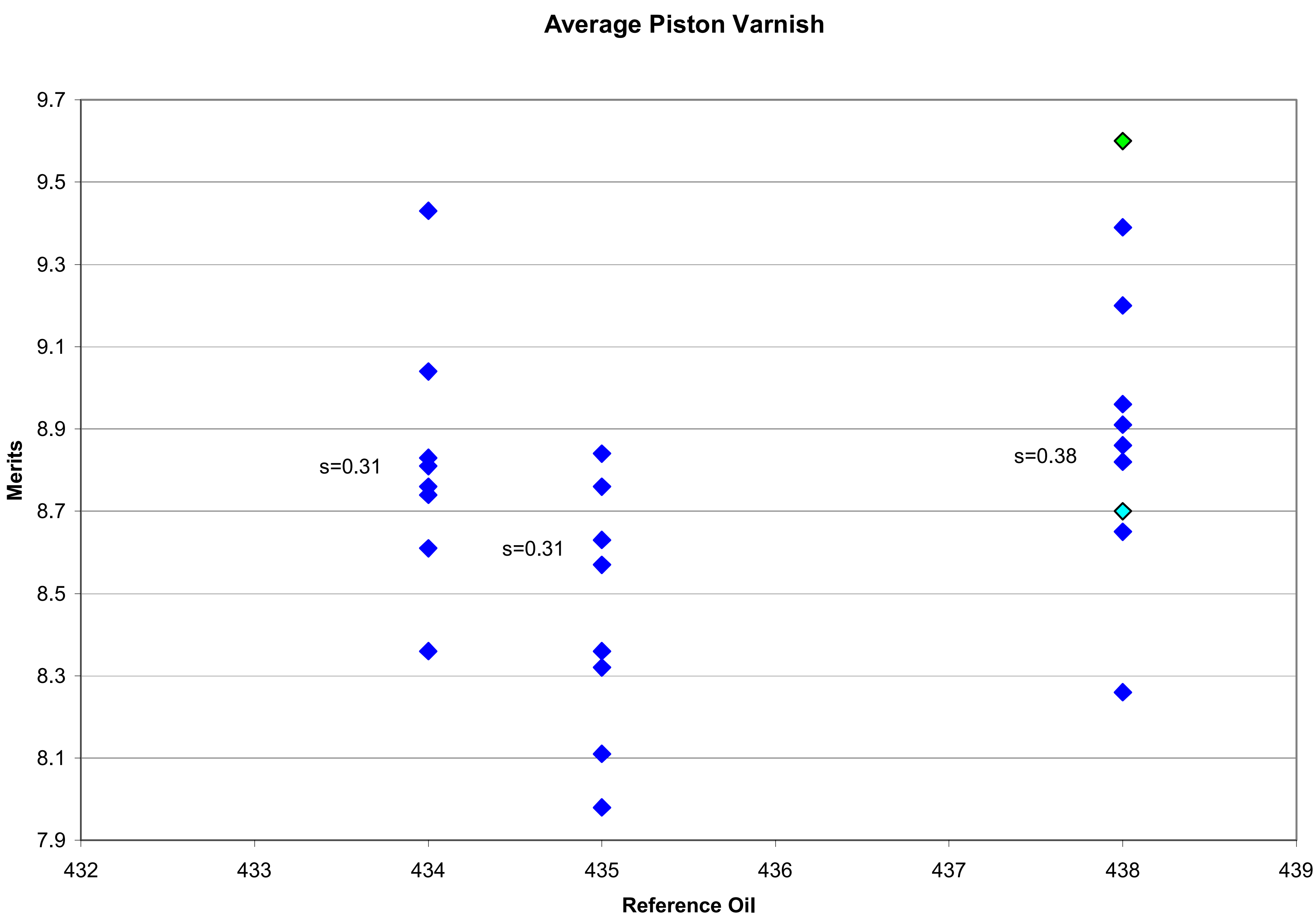
Average Piston Varnish (APV)

- Root Mean Squared Error=0.320346 (20 df)
- No Statistical Evidence that the Labs Differ
- Statistical Evidence that the Oils Differ

Average Piston Varnish (APV)

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	434	435	438		
Oil 434		0.072	0.929	8.82	8.59 to 9.06
Oil 435	0.072		0.034	8.45	8.21 to 8.68
Oil 438	0.929	0.034		8.88	8.64 to 9.12

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	Lab A	Lab G			
Lab A		0.332		8.78	8.59 to 8.97
Lab G	0.332			8.65	8.46 to 8.84



Average Piston Varnish
Merits
9.7
9.5
9.3
9.1
8.9
8.7
8.5
8.3
8.1
7.9
s=0.31
s=0.31
s=0.38
432
433
434
435
436
437
438
439
Reference Oil

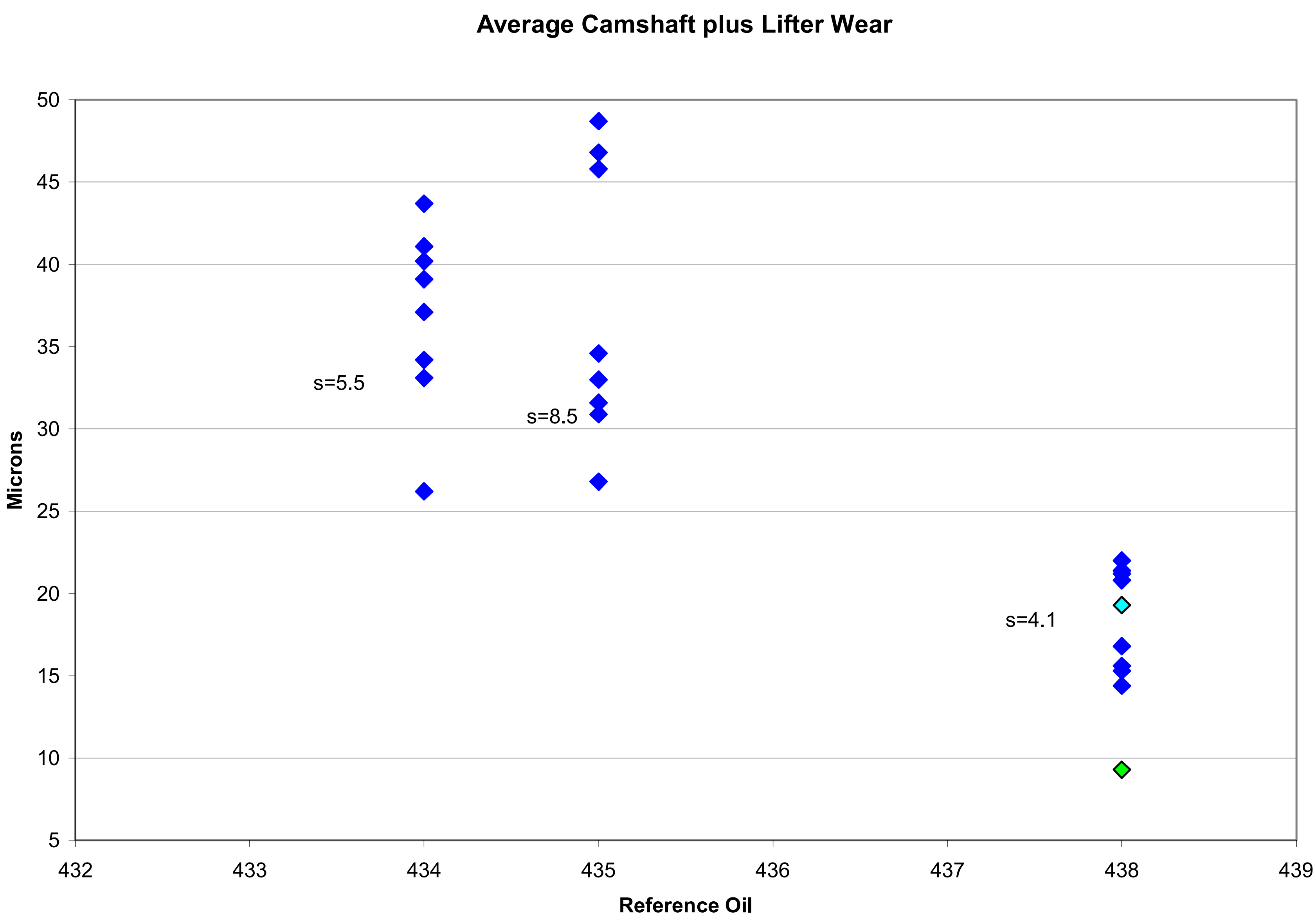
Average Camshaft plus Lifter Wear (ACLW)

- Analyzed on Natural Log Scale
- Root Mean Squared Error=0.193585 (20 df)
- No Statistical Evidence that the Labs Differ
- Strong Statistical Evidence that the Oils Differ

Average Camshaft plus Lifter Wear (ACLW)

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	434	435	438		
Oil 434		1.000	0.000	36.44	31.59 to 42.03
Oil 435	1.000		0.000	36.45	31.60 to 42.05
Oil 438	0.000	0.000		18.19	15.77 to 20.98

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	Lab A	Lab G			
Lab A		0.673		29.40	26.17 to 33.04
Lab G	0.673			28.42	25.30 to 31.94


Average Camshaft plus Lifter Wear
Microns
50
45
40
35
30
25
20
15
10
5
s=5.5
s=8.5
s=4.1
432
433
434
435
436
437
438
439
Reference Oil

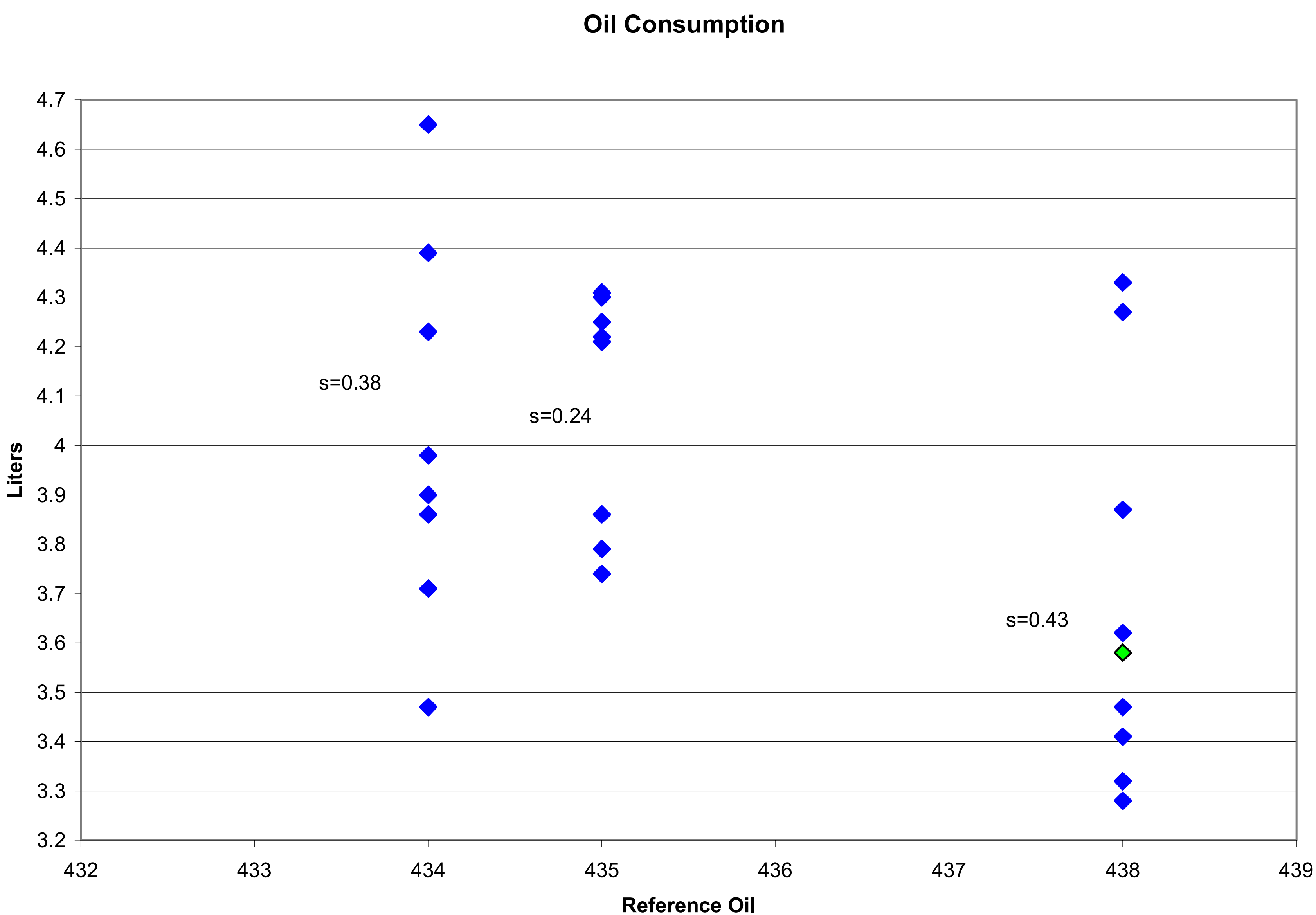
Oil Consumption (OC)

- Weak Evidence of a Stand Effect (Stand not Fit in Final Model)
- Root Mean Squared Error=0.3282 (20 df)
- Statistical Evidence that the Labs Differ
- Some Statistical Evidence that the Oils Differ

Oil Consumption (OC)

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	434	435	438		
Oil 434		0.926	0.138	4.02	3.78 to 4.27
Oil 435	0.926		0.069	4.09	3.84 to 4.33
Oil 438	0.138	0.069		3.70	3.45 to 3.94

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	Lab A	Lab G			
Lab A		0.045		3.79	3.59 to 3.99
Lab G	0.045			4.08	3.88 to 4.28


Oil Consumption
Liters
4.7
4.6
4.5
4.4
4.3
4.2
4.1
4
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
s=0.38
s=0.24
s=0.43
432
433
434
435
436
437
438
439
Reference Oil

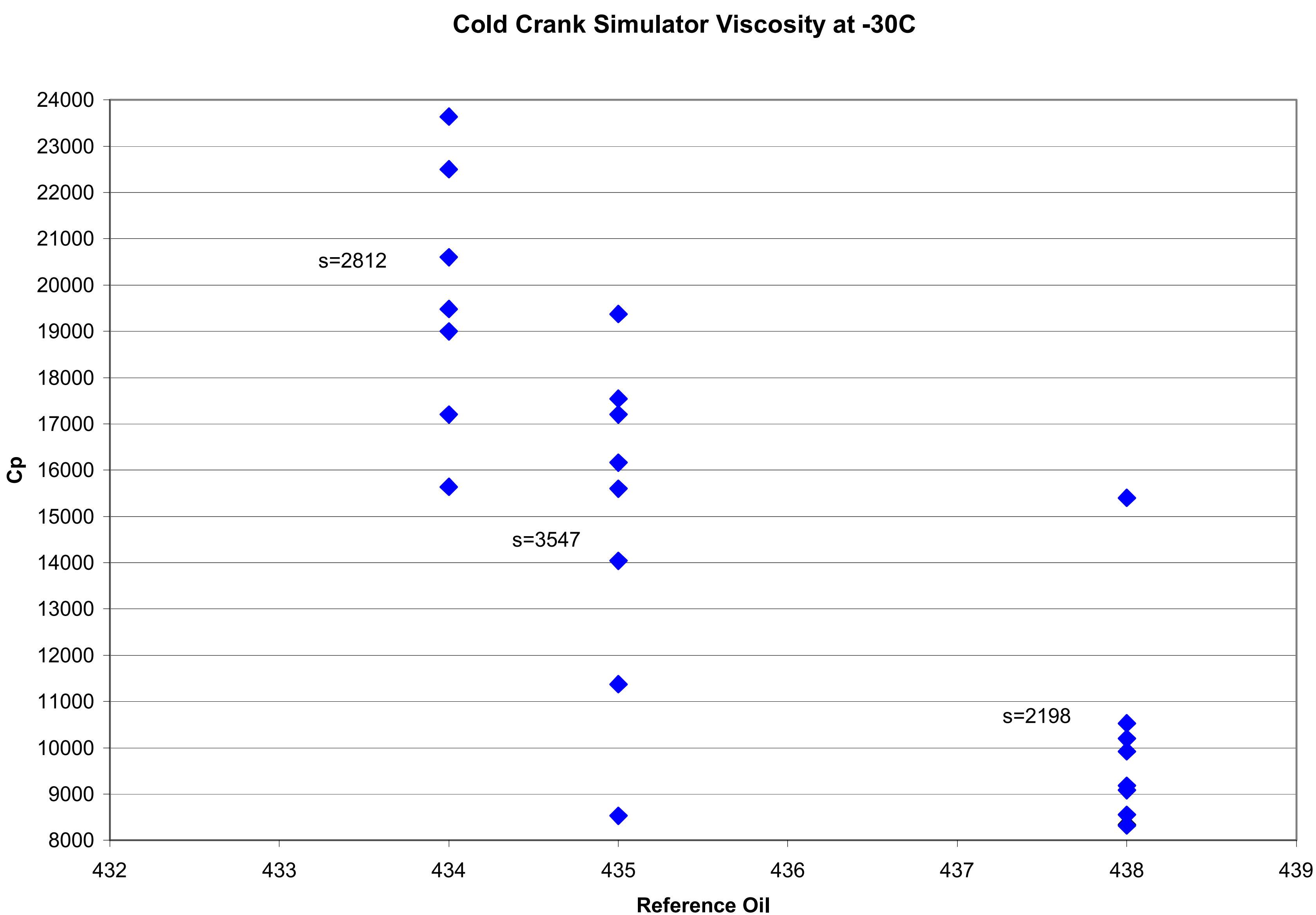
Cold Crank Simulator Viscosity (CCS)

- Only 23 Out of 24 Matrix Results Available
- Root Mean Squared Error=3001.769 (19 df)
- No Statistical Evidence that the Labs Differ
- Strong Statistical Evidence that the Oils Differ

Cold Crank Simulator Viscosity (CCS)

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	434	435	438		
Oil 434		0.014	0.000	19,716	17,485 to 22,192
Oil 435	0.014		0.011	14,976	12,789 to 17,164
Oil 438	0.000	0.011		10,148	7960 to 12,335

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	Lab A	Lab G			
Lab A		0.944		14,992	13,178 to 16,805
Lab G	0.944			14,902	13,000 to 16,804


Cold Crank Simulator Viscosity at -30C
Cp
24000
23000
22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
432
433
434
435
436
437
438
439
Reference Oil
s=2812
s=3547
s=2198

MRV Viscosity (MRV)

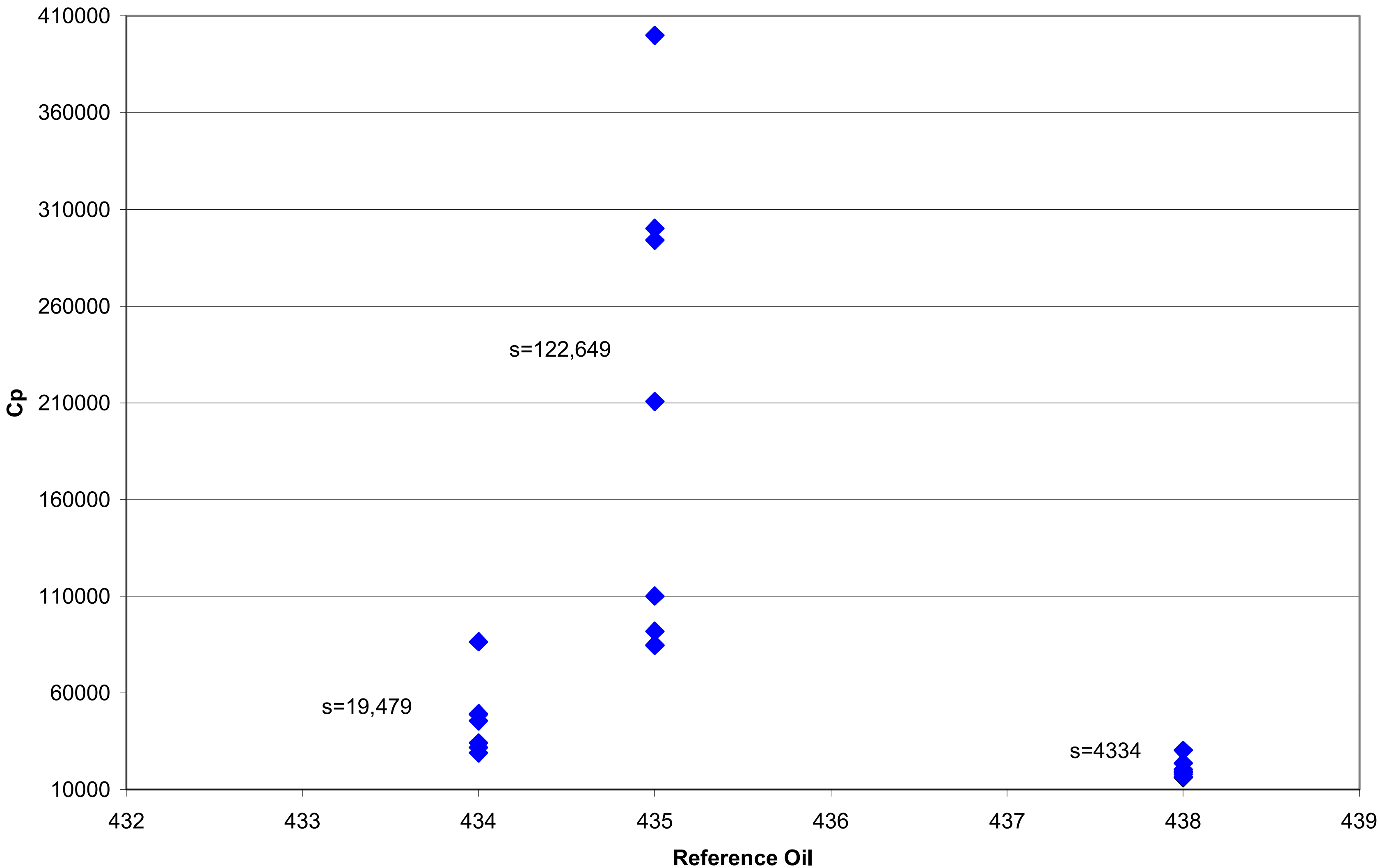
- Only 23 Out of 24 Matrix Results Available
- Analyzed on Inverse Square Root Scale
- Weak Evidence of a Stand Effect (Stand not Fit in Final Model)
- Root Mean Squared Error=0.000695 (19 df)
- Statistical Evidence that the Labs Differ
- Strong Statistical Evidence that the Oils Differ
- Analysis Performed with and without Oil 435, but Transformation Holds, Oil Discrimination Holds, and Standard Deviation in Transformed Units Remains Approximately the Same (Note, However, that there is only Some Evidence of Lab Effects)

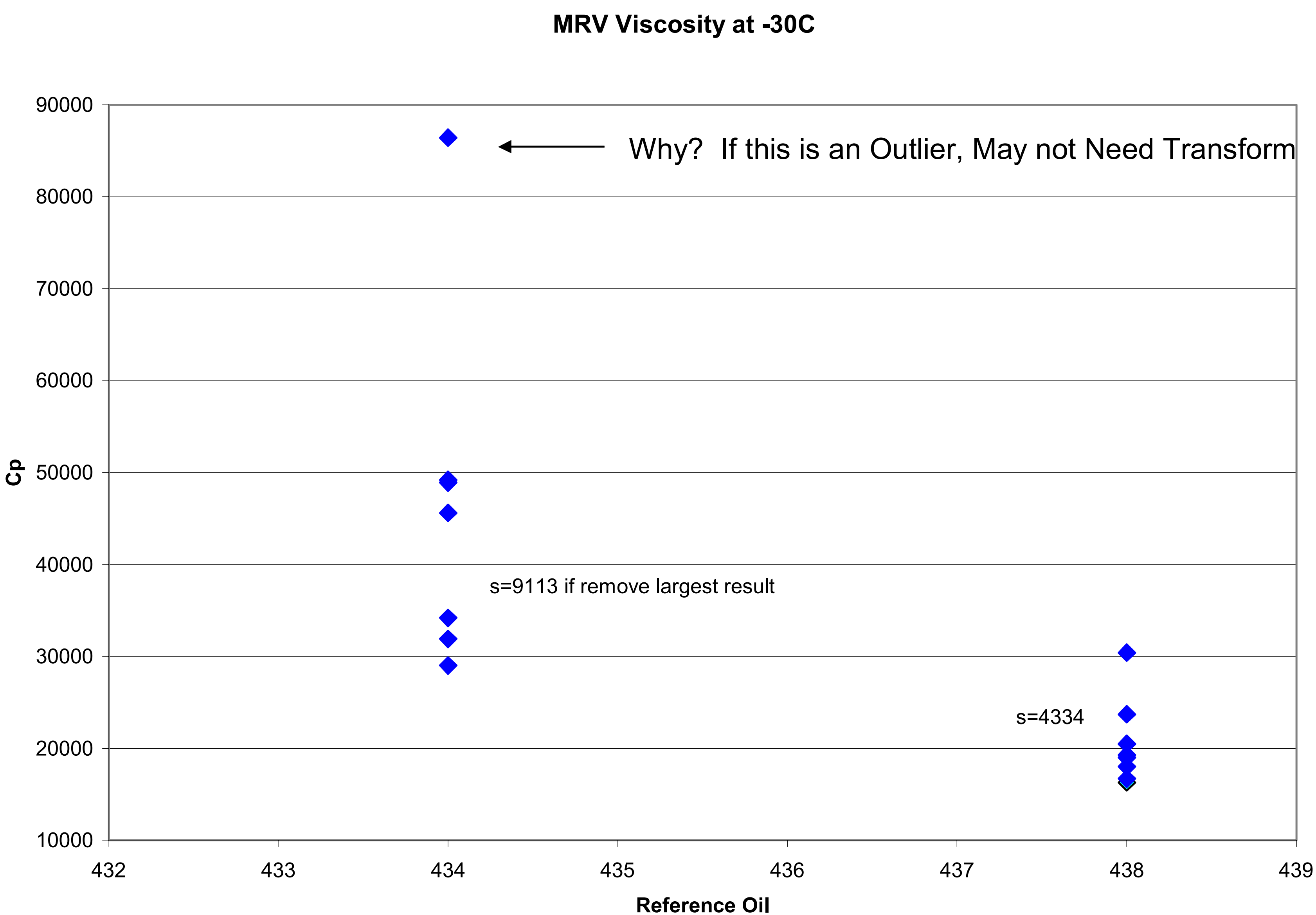
MRV Viscosity (MRV)

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	434	435	438		
Oil 434		0.000	0.000	43,187	34,768 to 55,104
Oil 435	0.000		0.000	150,581	104,665 to 234,964
Oil 438	0.000	0.000		20,519	17,802 to 23,918

p-values in Hypothesis Test of No Difference				Mean	95% Confidence Interval for the Mean
	Lab A	Lab G			
Lab A		0.044		38,402	32,783 to 45,599
Lab G	0.044			49,891	41,362 to 61,390

MRV Viscosity at -30C




MRV Viscosity at -30C
Why? If this is an Outlier, May not Need Transform
s=9113 if remove largest result
s=4334
Cp
90000
80000
70000
60000
50000
40000
30000
20000
10000
432
433
434
435
436
437
438
439
Reference Oil

Summary of Means and Standard Deviations by Oil

	VIS		LN(VIS)		NVIS	
	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev
Oil 434	129.34	61.35	4.7729	0.4447	106.40	24.42
Oil 435	214.49	54.33	5.3416	0.2433	176.79	25.47
Oil 438	110.15	20.57	4.6870	0.1832	122.97	17.89
Model Std Dev		48.78		0.2919		23.34
ACC Ep		**0.62**		**0.77**		**1.29**

Summary of Means and Standard Deviations by Oil

	APV		WPD		NWPD	
	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev
Oil 434	8.82	0.31	4.34	0.88	4.38	0.70
Oil 435	8.45	0.31	3.38	0.43	3.62	0.32
Oil 438	8.88	0.34	3.28	0.44	3.50	0.35
Model Std Dev		0.32		0.60		0.47
ACC Ep		**0.94**		**0.50**		**0.65**

Summary of Means and Standard Deviations by Oil

	ACLW		LN(ACLW)		OC	
	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev
Oil 434	36.84	5.55	3.5956	0.1624	4.02	0.38
Oil 435	37.28	8.47	3.5961	0.2247	4.09	0.24
Oil 438	18.44	3.20	2.9009	0.1767	3.70	0.42
Model Std Dev		6.13		0.1936		0.33
ACC Ep		**1.63**		**1.87**		**1.53**

Summary of Means and Standard Deviations by Oil

	CCS		MRV		1/SQRT(MRV)	
	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev
Oil 434	19,723	2812	46,457 (39,800)	19,479 (9113)	0.00485629	0.00084309
Oil 435	14,976	3547	197,025	122,649	0.00257696	0.00079675
Oil 438	10,148	2260	21,013	4318	0.00698055	0.00062174
Model Std Dev		3002	434,438 only	7131		0.000695
ACC Ep		**0.27**		**1.12**		**~1**