CRC Project DP-04-13b

Internal Injector Deposits;
A Scoping Study to Evaluate the Delphi Test Rig

August 2013

COORDINATING RESEARCH COUNCIL, INC.
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CRC Diesel Performance Group
Deposit Panel

Internal Injector Deposits; A Scoping Study to Evaluate the Delphi Test Rig

CRC Project Report DP-04-13b

August 2013

Report Outline

- **Program Objective and Scope**
- **Summary of Delphi Internal Injector Deposit (IID) Test method**
- **Summary of CRC Test Methodology and Fuel test Matrix**
- **Analysis of Results**
  - Quick comparison of all tests
  - CARB (California) diesel
  - South American Diesel
  - UK No Deposit (UKNoDep) Diesel
  - UK Deposit Forming Diesel (UKDep) Diesel
  - Effects of Low Molecular Weight PIBSI
- **Summary, Conclusions, and Next Step**

- Appendix 1: Fuel Analysis and Blending
- Appendix 2: Photographs of Specimens from All Tests Performed
- Appendix 3: Deposit Thickness Measurement

Bench/Rig Investigation Sub Panel Members

- Hind Abi-Akar
- Joan Axelrod
- Rodica Baranescu
- Rick Chapman
- Richard George
- Alex Kulinowski
- Paul Lacey
- Shailesh Lopes
- Manuch Nikanjam (leader)
- Jim Rutherford

Acknowledgement

- Delphi and Paul Lacey devoted valuable test facilities, resources, and expertise to carry out this program at no cost to CRC.

Abstract

- Shortly after the formation of a Deposit Panel within the CRC Diesel Performance Group, the EMA approached the Panel and requested initiation of an urgent effort to evaluate the causes of a new internal injector deposit problem. The panel diverted attention to this issue and formed three sub-panels:
  - Data Analysis
  - Bench / Rig Investigation
  - Engine Investigation
- The Bench / Rig Investigation sub panel identified two proposed rigs, evaluated them, and concluded that, neither, in their present state, could discriminate among deposit forming and not deposit forming fuels.
- Subsequently, another rig developed by Delphi was proposed for evaluation. This report provides a detailed description of the new rig and the results obtained from a scoping study using four fuels in a statistically designed matrix. Given that this is a scoping study, the effects of detergents additives (other than LMW PIBSI), biodiesel, and impurities were not included.

Objective

- Identify or develop a laboratory bench top or test rig for evaluating fuel's tendency to cause internal injector deposits as well as additive's effectiveness to avoid such deposit formations.

Scope

- This phase was a limited scoping and screening program using Delphi's in-house rig to determine if fuels which are expected to cause internal injector deposits can be differentiated from those that are not expected to form such deposits.

Not Included in This Study

- Detailed study of:
  - Impurities
  - Additives
  - Biodiesel

Delphi Internal Injector Deposit (IID) Test Stand

- **Designed to Accelerate Formation of Internal Injector Deposits**
  - Simulates severe engine operating conditions
- **High Pressure Common Rail System**
  - Mounted on electric motor driven test stand
  - Injected fuel is not recirculated
- **Operating Conditions Simulate "Thermal Soak Back"**
  - **Continuous replication of transient shut down condition**
  - High injection pressure and temperature, low injected volume
    - Maximum fuel stress with minimum fuel flow.
- **Not Specific to any FIE Design or Brand**
  - Current tests use older generation injector design
  - Not suitable for evaluation of FIE design or construction
    - Artificially accelerated test condition
- **Combustion Temperature Replicated Using Electrical Heaters**
  - Environment similar to an operating engine

Operating Conditions for IID Test Stand

Detailed Description of Test Methodology in Attached document*

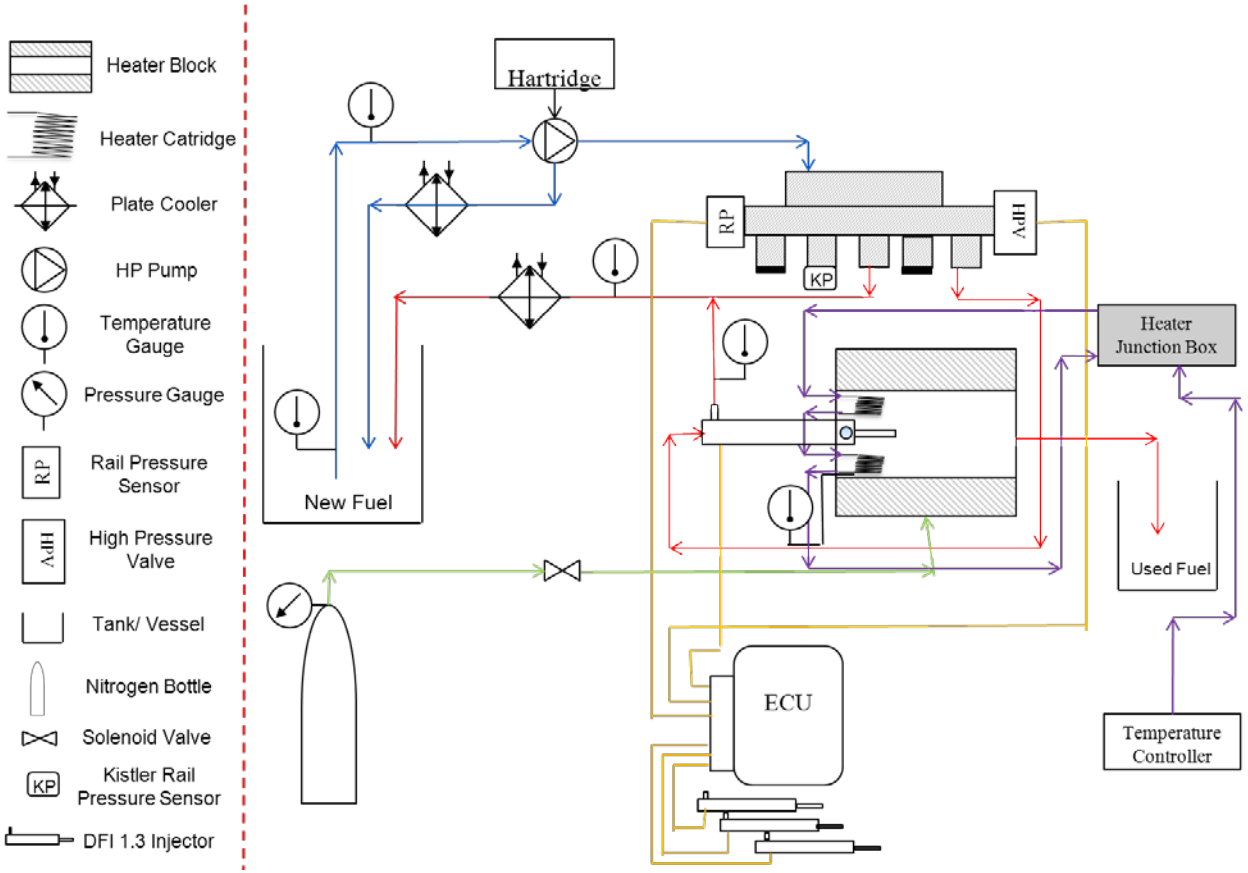
Test Conditions		
Typical Test Duration	7**	hrs
Heater Set Point	200	°C
Pump Speed	1750	Rpm
Rail Pressure	1800	bar
Injection Pulse Length	Calibrated to give 5g/min fuel delivery at the start of test	
Injection Frequency	12.5	Hz
Injected Fuel Flow Rate	5	g/min

* Delphi Test Methodology for Internal Injector Deposit (IID) Apparatus, P. Lacey, 25 Feb 2013
**Additional tests also performed at 14, 21 and 28 hours

Schematic Diagram of IID Test Stand

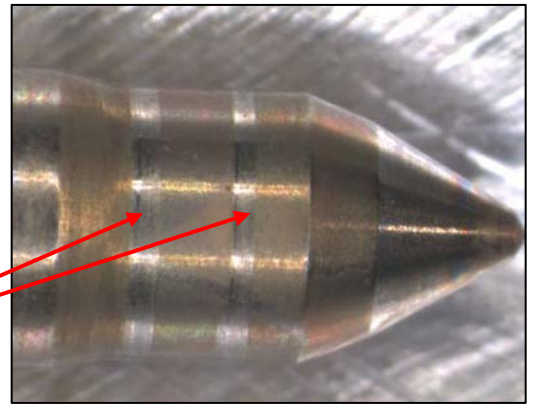
Note: Injected fuel is not returned to storage tank

Replicates burned fuel in vehicle operation



Post Test Deposit Analysis Techniques

- **Fourier Transform Infrared (FTIR) Performed Directly on Deposits**
  - ATR mono-reflexion mode
  - Germanium crystal
  - Applicable to solid or liquid samples without sample preparation
  - Analysed area = 100x100µm
- **EDX Elemental Analysis Performed on Some Deposits**
- **Deposit Thickness Measurement**
  - Zygo NewView 5000 Optical Interferometer
    - Failed to “see” darker deposits in these tests
  - Talysurf surface profilometer



Tracks cut in deposit
Deposit thickness measured here

CRC Test Program

- **Matrix of 12 Tests**
  - Four fuels tested in duplicate defined by CRC
    - Two Deposit Forming Fuels
    - Two Non Deposit Forming Fuels
  - Plus a blank test with “clean” fuel between each test

- **Deposit Thickness was Measured for Half of the Tests**
  - Deposit chemistry was evaluated for the other half
    - Thickness measure may chemically contaminate specimens

- **12 Additional Tests Performed by Delphi to Indicate the Effects of:**
  - Test duration
  - Higher concentrations of contaminant
  - Effect of LMW PIBSI on deposit chemistry*

*Low molecular weight fraction of PolyIso Butylene Succinimide Deposit Control Additive

Overview of Test Fuels

Matrix of Four Fuels defined by CRC

Additional chemistry containing LMW PIBSI suggested by Delphi

Fuel Designation	Suggested by	Supplier	Comment
CARB (California) Diesel	CRC	Chevron	Not expected to produce deposit
South America Diesel	CRC	Innospec	Believed to produce deposit. Deposit composition unknown.
UKNoDep[1]	CRC	Delphi	Not expected to produce deposit Additive free S10 EU test fuel[1] Free of elemental contamination
UKDep[2]	CRC	Delphi	Should produce metal carboxylate deposit UKNoDep Fuel + NaOH and DDSa Blended Sodium concentration =2.79 mg/kg Recipe provided by Afton Chemical
UKDep + LMW PIBSI	Delphi	Delphi	Should produce metal carboxylate and amide deposit UKNoDep Fuel + NaOH and DDSa + LMW PIBSI

1) UKNoDep is a reference fuel containing only lubricity additive. See Appendix 1 for certificate of analysis
2) See Appendix 1 for blend instructions (results in 5.23mg/L NaOH+ 9 mg/L active DDSa)
3) UKNoDep diesel also used as a rinse test between each fuel above

Blend Instructions for UKDep Fuel

- **Blend Instructions Provided by Afton Chemical**
  - Detailed instructions provided in Appendix 1
  - Uses DDSa and Sodium Hydroxide

- **Increased Contaminant Concentrations Also Evaluated by Delphi**
  - Not part of the CRC matrix
  - Blended in the same manner as the standard treat rate
  - At concentrations defined in table on subsequent slide

- **Afton Recipe Uses Water as a Co-solvent for NaOH**
  - Some issues observed with precipitates during blending
    - Likely to reduce true sodium concentration in test fuel
  - Less issues observed when ethanol used as co-solvent
    - Ethanol used for most tests not defined in CRC matrix

Overview of Chemicals Used for Fuel Treatment

- **DDSa is a Commonly Used Pipeline Corrosion Inhibitor**
  - Strongly linked with IID issues in both Europe and North America
  - Typically Added at 20 mg/L including Carrier Solvent
  - Believed to be approximately 45 wt% active ingredient
  - True treat rate of DDSa was 9 mg/L

- **NaOH Used as the Source of Sodium**
  - 57 wt% sodium

- **LMW PIBSI Low Molecular Weight Fraction of PIBSI**
  - PolyIso Butylene Succinimide Deposit Control Additive
  - Not part of original CRC matrix
  - Believed to be 5.5 wt% active ingredient

All Treat Rates Reflect Concentration of Active Component

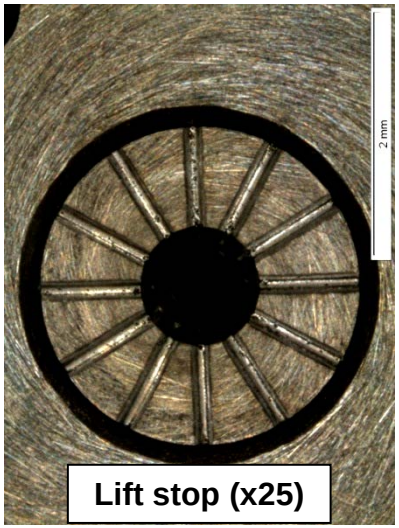
Summary of Complete Test Matrix Performed

Test	Fuel	Time, hr	Action	Injector No
CRC Defined Test Matrix				
1	UKNoDep	7	Needle and control valve sent for FTIR	E120 175 HEF
2	CARB	7	Measure Deposit Thickness	E120 8163 GEF
3	UKNoDep	7	No Action	E120 3408 HEF
4	UKDep (Water used as co-solvent for NaOH)	7	Measure Deposit Thickness	E120 3351 HEF
5	UKNoDep	7	No action	C120 385 FEF
6	South American	7	Measure Deposit Thickness	E120 601 HEF
7	UKNoDep	7	No Action	N/A
8	South American	7	Needle and control valve sent for FTIR	E120 4940 HEF
9	UKNoDep	7	No Action	E120 2707 HEF
10	UKDep (Water used as co-solvent for NaOH)	7	Needle and control valve sent for FTIR	E120 193 HEF
11	UKNoDep	7	No Action	E120 6130 HEF
12	CARB	7	Needle and control valve sent for FTIR	E120 3455 HEF
	Supplemental Tests Performed by Delphi In Addition to CRC Defined Matrix			
13	UKDep Water used as co-solvent for NaOH)	14	Deposit thickness measurement	E120 1729 HEF
14	UKDep (Ethanol used as co-solvent for NaOH)	28	Measure Deposit Thickness	C120 385 FEF
15	UKNoDep	7	No Action	E120 6107 GEF
16	UKNoDep+ 13.2 mg/L NaOH + 225mg/L DDSa (Ethanol or water?)	7	Needle and control valve sent for FTIR	E120 807 HEF
17	UKDep (Ethanol used as co-solvent for NaOH)	14	Deposit thickness measurement	E120 95 HEF
18	South American	28	Deposit thickness measurement	E120 2059 HEF
19	UKNoDep	28	Photo Only	E120 6595 GEF
20	UKNoDep	7	None	
21	UKNoDep +3.3 mg/L NaOH+56 mg/L DDSa +27.5ml/L LMW PIBSI	7	Measure deposit thickness	E1203636 HEF
22	UKNoDep +6.6 mg/L NaOH+112 mg/L DDSa +55 mg/l LMW PIBSI	7	Measure deposit thickness	E1203652 HEF
23	UKNoDep +13.2mg/L NaOH+ 225 mg/L DDSa +110ml/L LMW PIBSI	7	Control valve sent for FTIR Deposit thickness measure on needle	E1203481 HEF

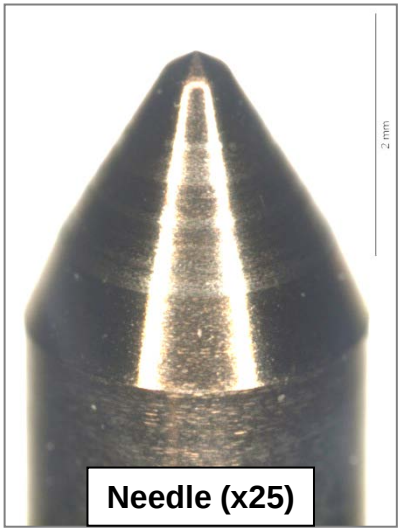
Preliminary Comparison of Deposits From CRC Defined Test Matrix

CARB	South American	UKNoDep	UKDep
 Test 2	 Test 6	 Test 1	 Test 4
 Test 12	 Test 8	 Test 3	 Test 10

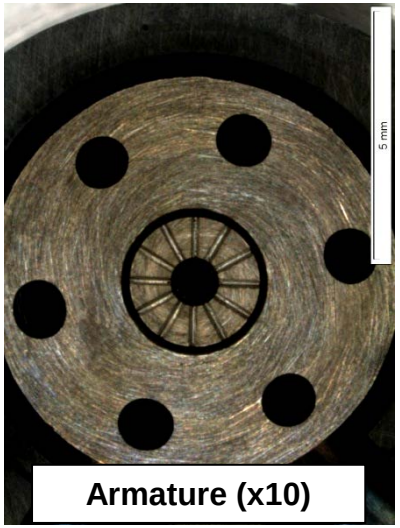
As Expected the "**CARB**" Fuel Produced Virtually No Deposit (Test 12)



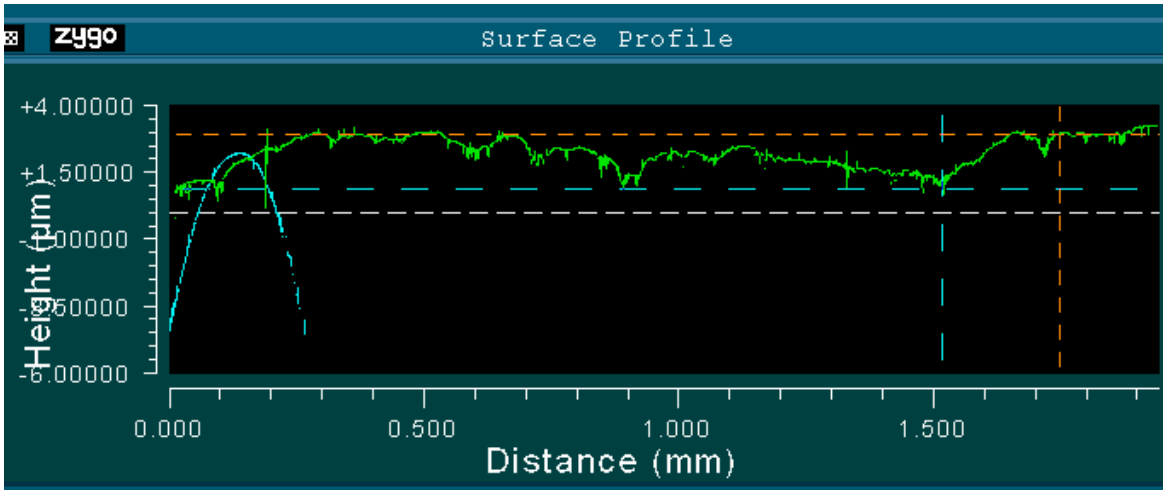
Lift stop (x25)



Needle (x25)



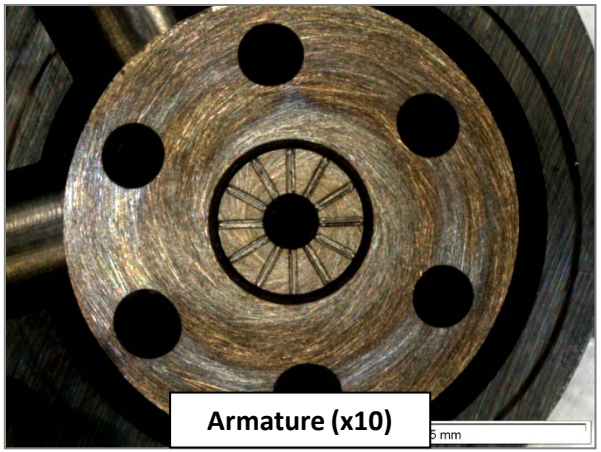
Armature (x10)

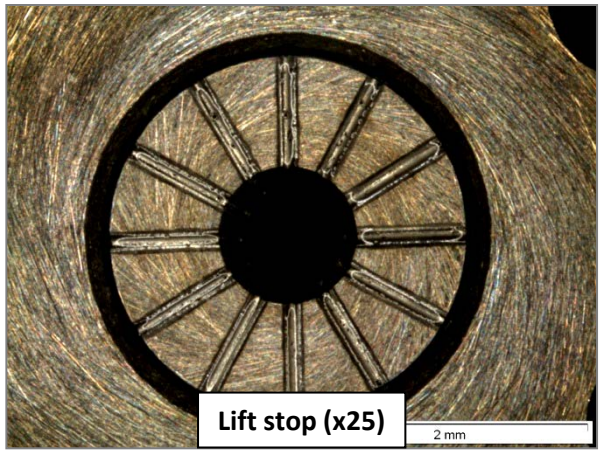


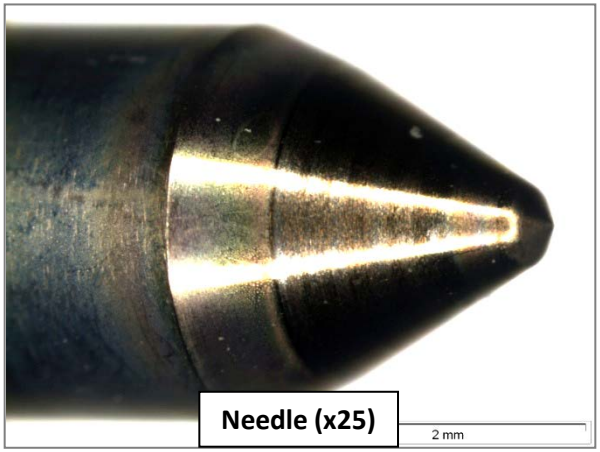
(E120 3455 HEF)

“**South American**” Fuel Produced Slight Deposit (Test 8)

Too Thin for Accurate Measurement Resutl following 7 Hour Test





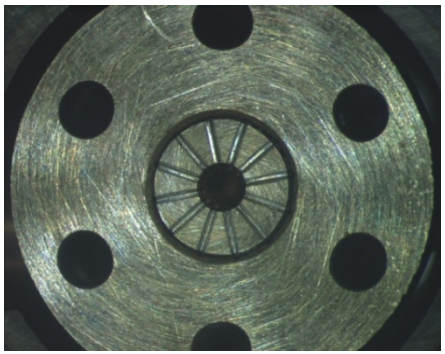
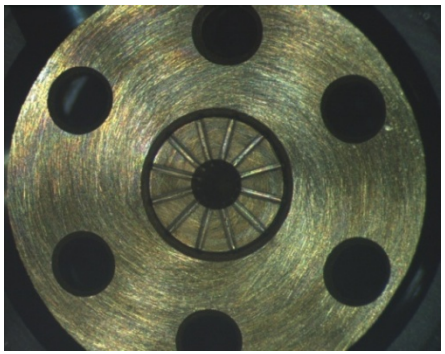
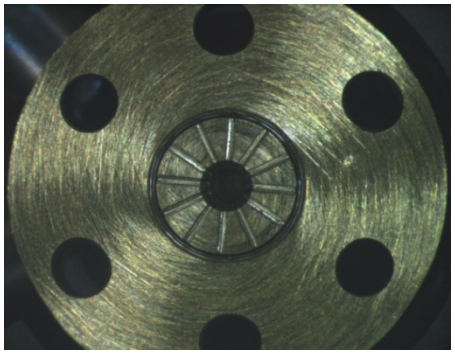


(E120 4940 HEF)

Increased Test Duration Produces More Deposit With **South American** Fuel

Visible Deposit Thicker Than Observed in 28 Hour Test With UKNoDep Diesel

However, deposit thickness remains too thin to measure

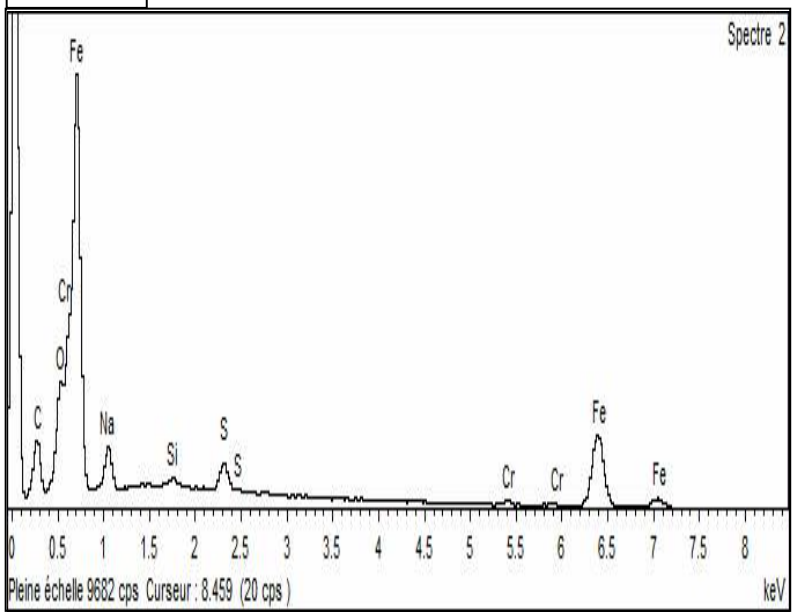
	South American Diesel		UKNoDep
	7 Hour Test (Test 7)	28 Hour Test (Test 18)	28 Hour Test (Test 19)
Needle	 (E120 601 HEF)	 (E120 2059 HEF)	 (E120 6595 GEF)
Armature			

IID Chemistry From **South American** Fuel Unlike Commonly Reported Field Deposits (Test 8)

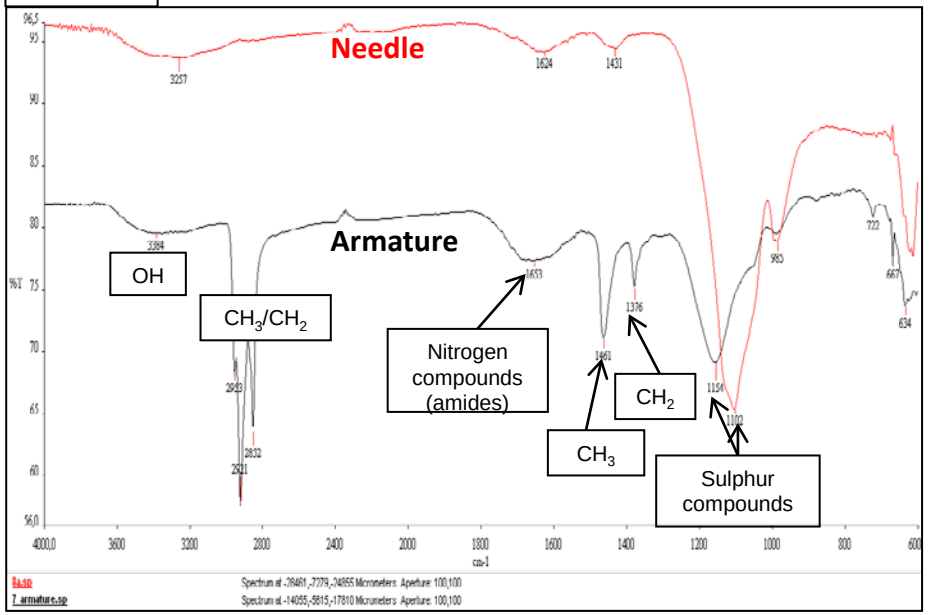
Very Little Deposit and What is there has Unusual Chemistry

Sulphur compounds with small contribution of amides

EDX

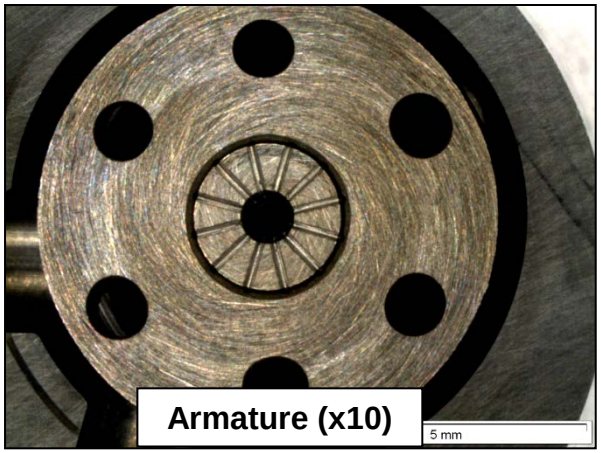


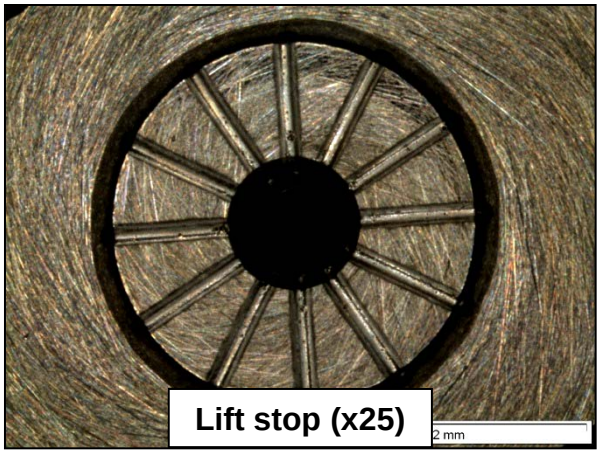
FTIR

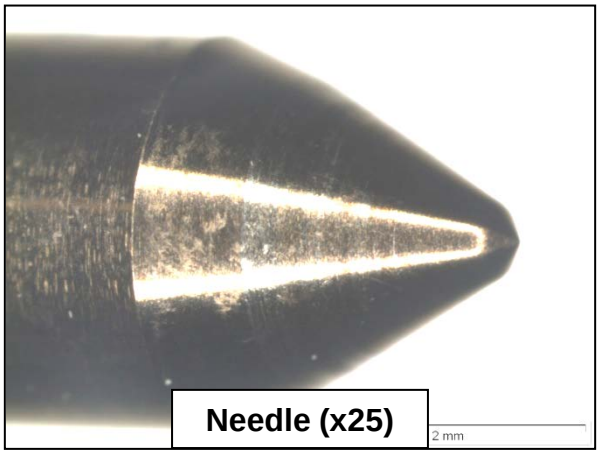


(E120 4940 HEF)

As Expected the **UKNoDep Diesel** Produced Virtually No Deposit (Test 1 Shown)



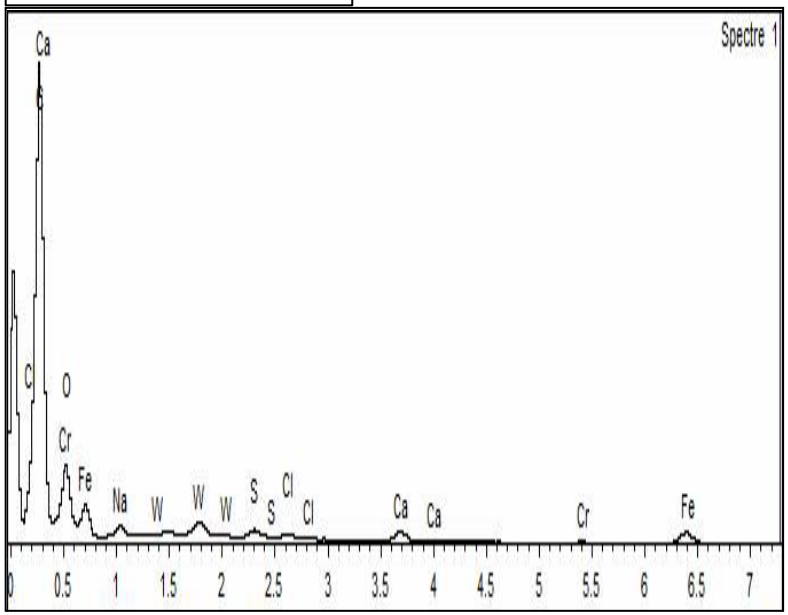




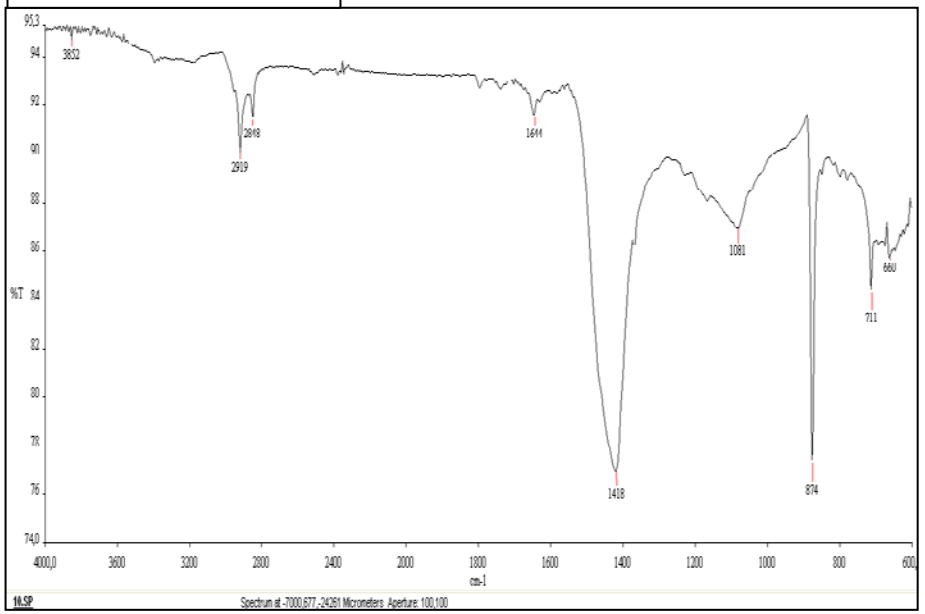
(E120 175 HEF)

Slight Deposit from the **UKNoDep Diesel** was an Undefined Organic Compound (Test 1 Shown)

EDX on Needle



FTIR on Needle

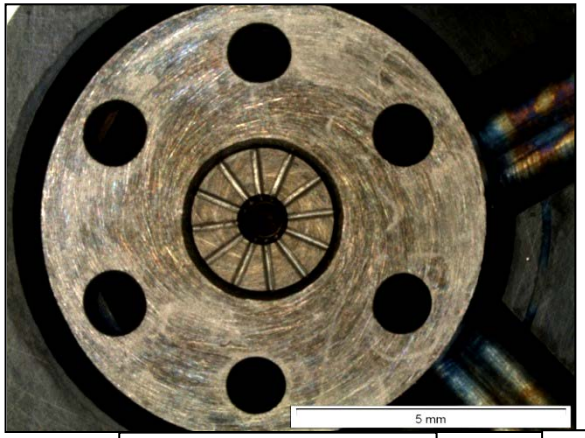


(E120 175 HEF)

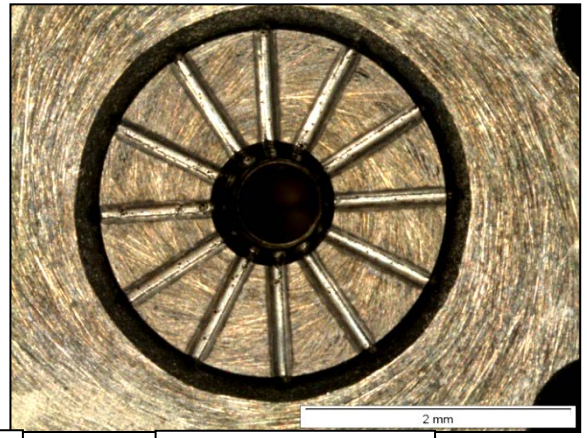
UKDep Fuel Produced Some Deposit During 7 Hour Test (Test 10)

Test No 10 = UKNoDep + 5.23mg/L NaOH + 9mg/L DDSa Corrosion Inhibitor

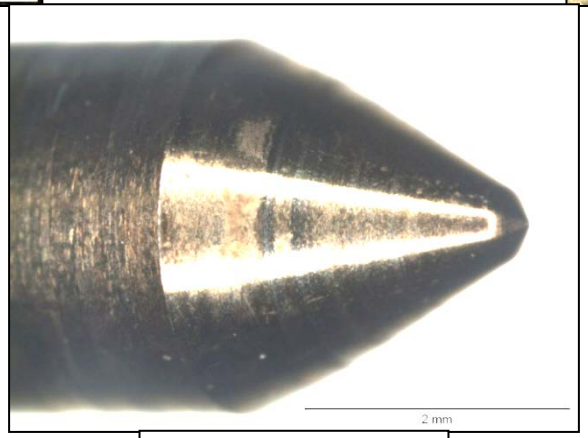
Equivalent to 2.9 mg/L sodium concentration



Armature (x10)



Lift stop (x25)



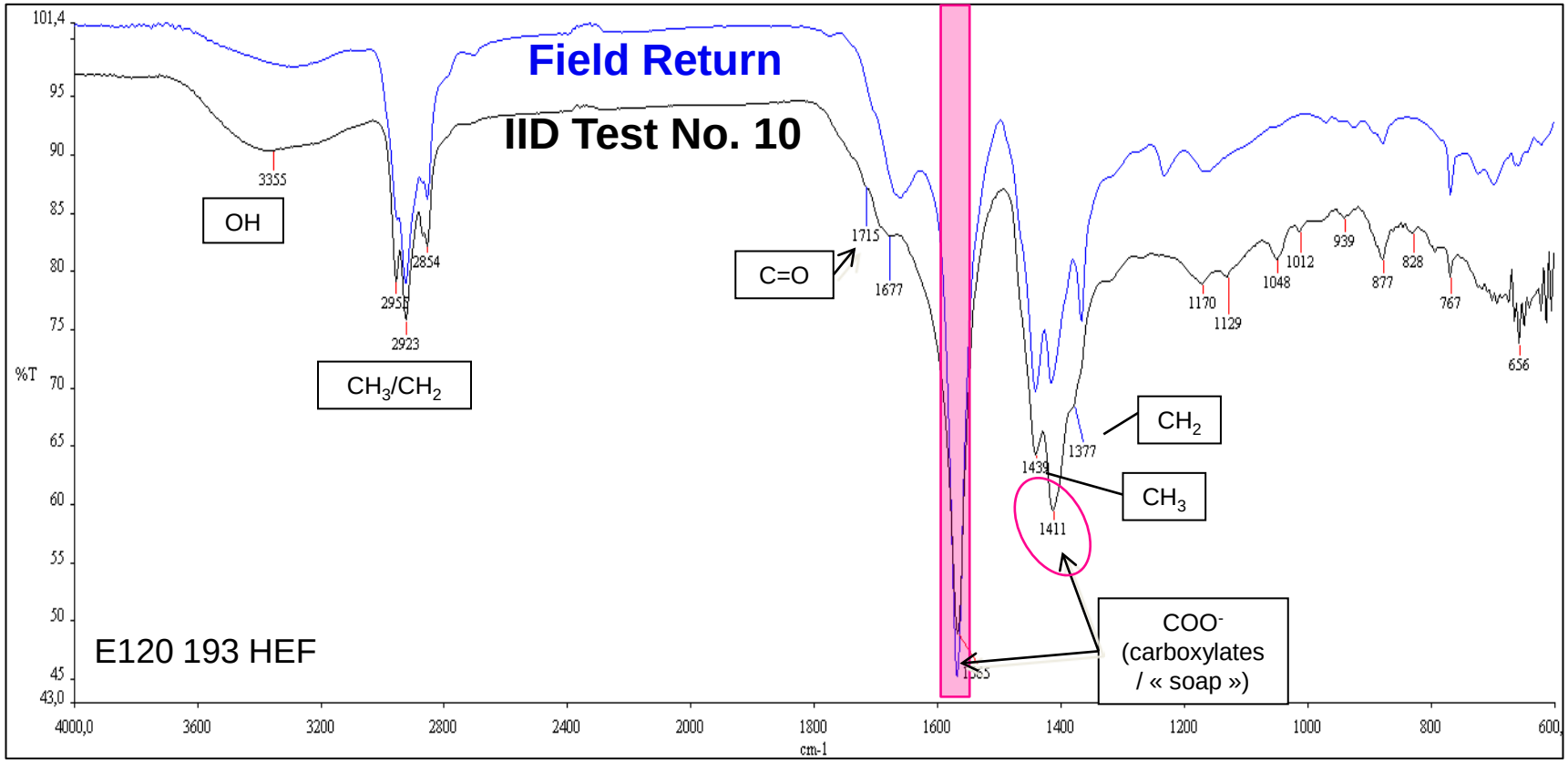
Needle (x25)

(E120 193 HEF)

UKDep Fuel
Forms Metal Carboxylate IID Chemistry (Test 10)

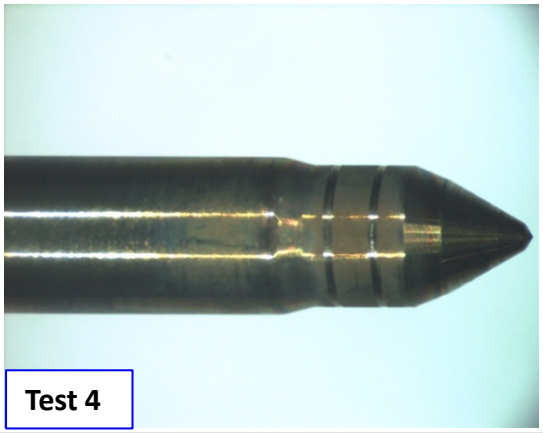
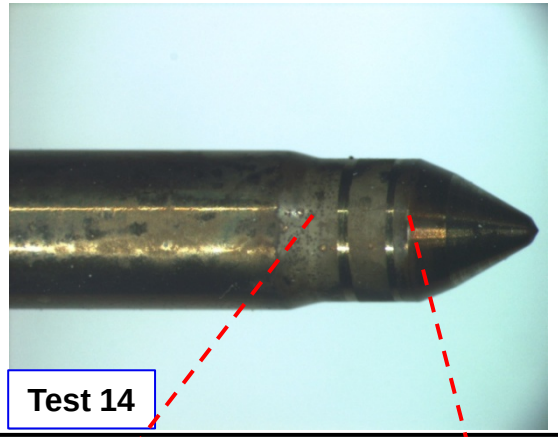
Effectively Recreates Commonly Reported European Field Issue

Does <u>not</u> Recreate Amide Deposit Observed on Some Field Returns

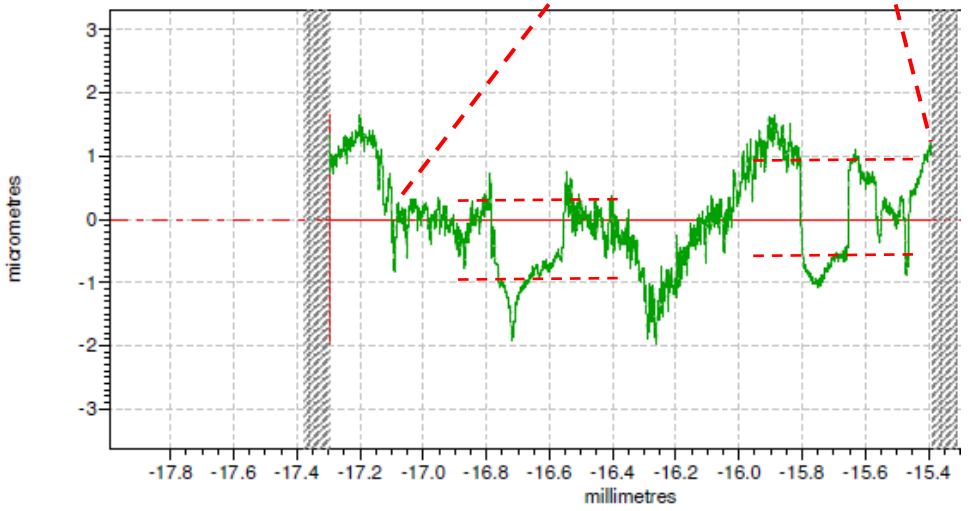


Deposit Thickness Increases With Longer Test Duration

UKDep = UKNoDep + 5.23mg/L NaOH + 9mg/L DDSa Corrosion Inhibitor)

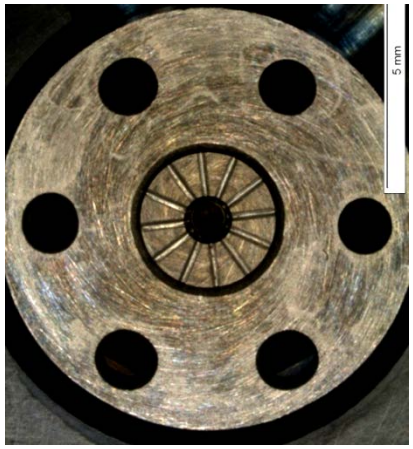
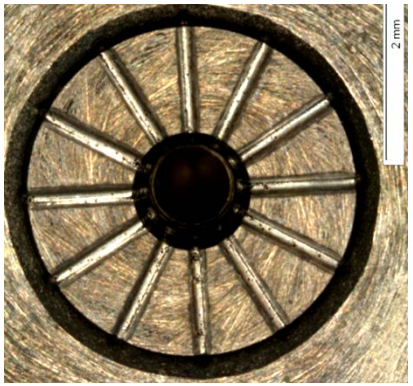
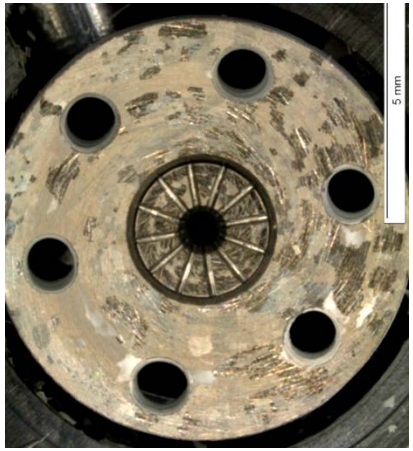
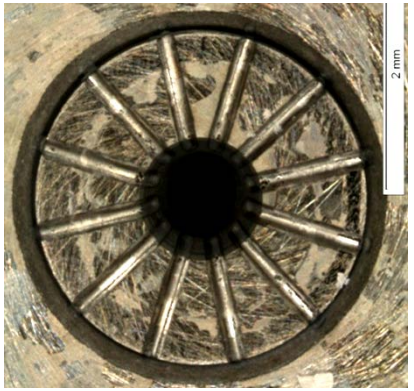
7 Hours	14 Hours	28 Hours
 Test 4	 Test 17	 Test 14

Talysurf Depth = 1.35 micron



(See slide 54 for description of deposit thickness measurement)

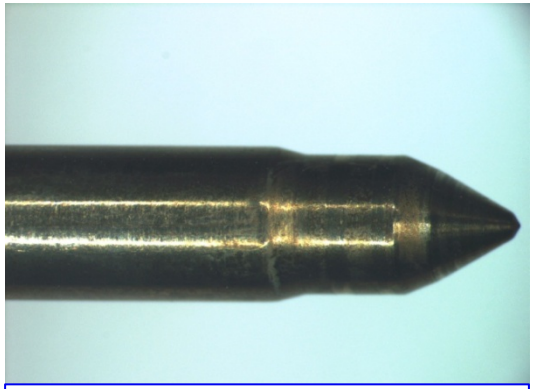
Significantly More Deposit Created When Contaminant Concentration is Increased

	Armature	Lift Stop	Needle
UKDep Diesel (Test 10) **UKNoDep+5.23mg/L NaOH +9mg/L DDSa** (E120 193 HEF)			
Increased Additive Conc. (Test 16) **UKNoDep+13.2mg/L NaOH + 225mg/L DDSa** **(**E120 807 HEF)			

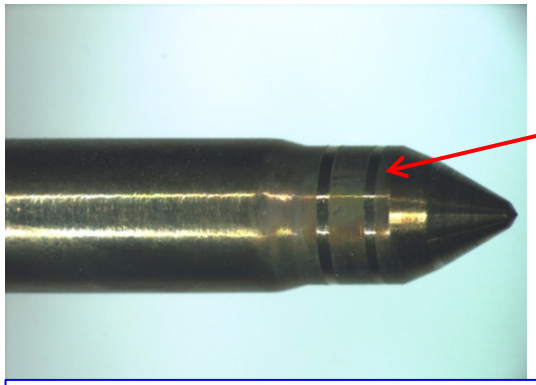
Co-Solvent Used to Dissolve NaOH May Have an Effect on Deposits

- **Co-Solvent Required to Facilitate Solution of NaOH in Diesel**
  - CRC instruction requires water be used as co-solvent
- **Poor Solution was Obtained with Water in these Tests**
  - Solid white precipitate dropped to bottom of beakers
    - Did not dissolve even with extended mixing and heating
    - Reduces material available for deposit formation
- **No Such Issues Observed when Ethanol Used as Co-Solvent**
  - Plus increased deposits observed with water

Most Tests Outside of CRC 12 Fuel Matrix Use Ethanol as Co-Solvent



Test 13: UKDep NaOH in Water

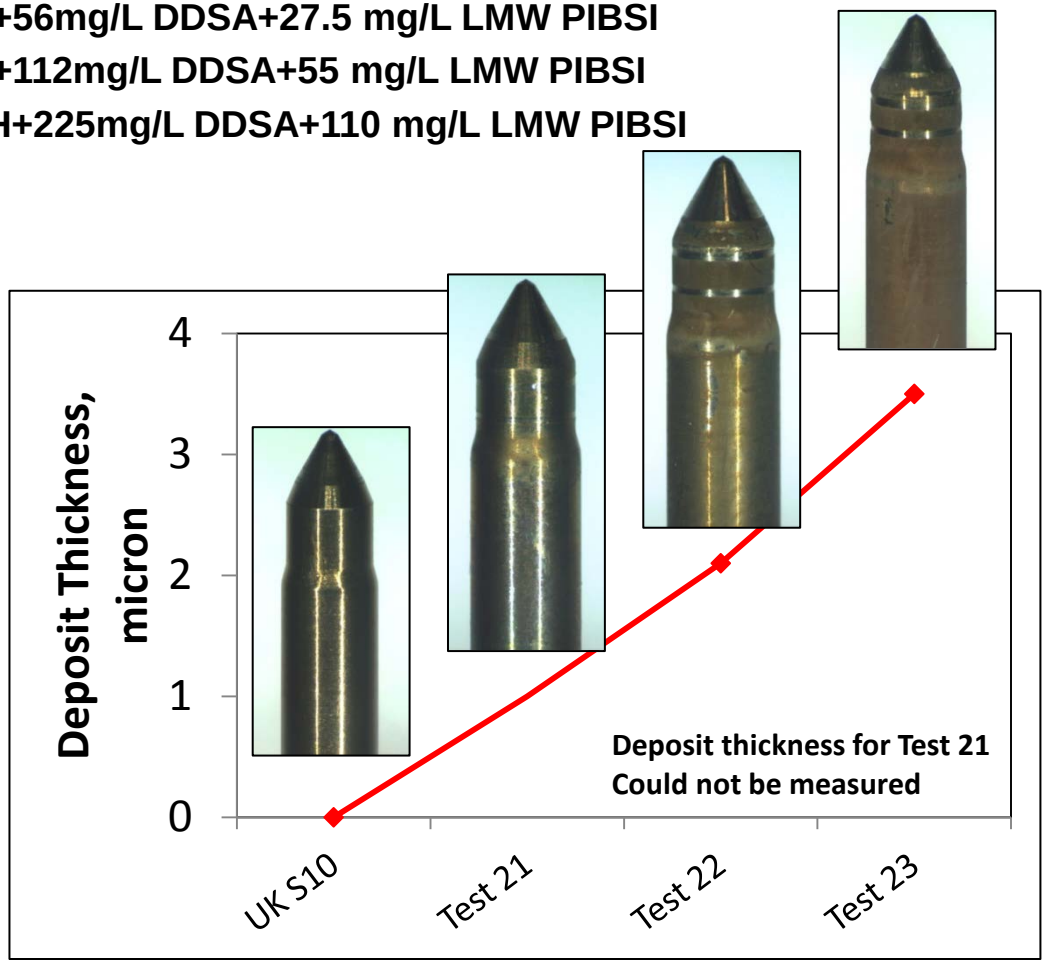


Test 17: UKDep NaOH in Ethanol

A Test Matrix Was Also Performed with LMW PIBSI + Na + DDSa

Test 20 = UKNoDep Diesel
Test 21 = UKNoDep +3.3 mg/L NaOH+56mg/L DDSA+27.5 mg/L LMW PIBSI
Test 22 = UKNoDep +6.6 mg/L NaOH+112mg/L DDSA+55 mg/L LMW PIBSI
Test 23 = UKNoDep +13.2 mg/L NaOH+225mg/L DDSA+110 mg/L LMW PIBSI

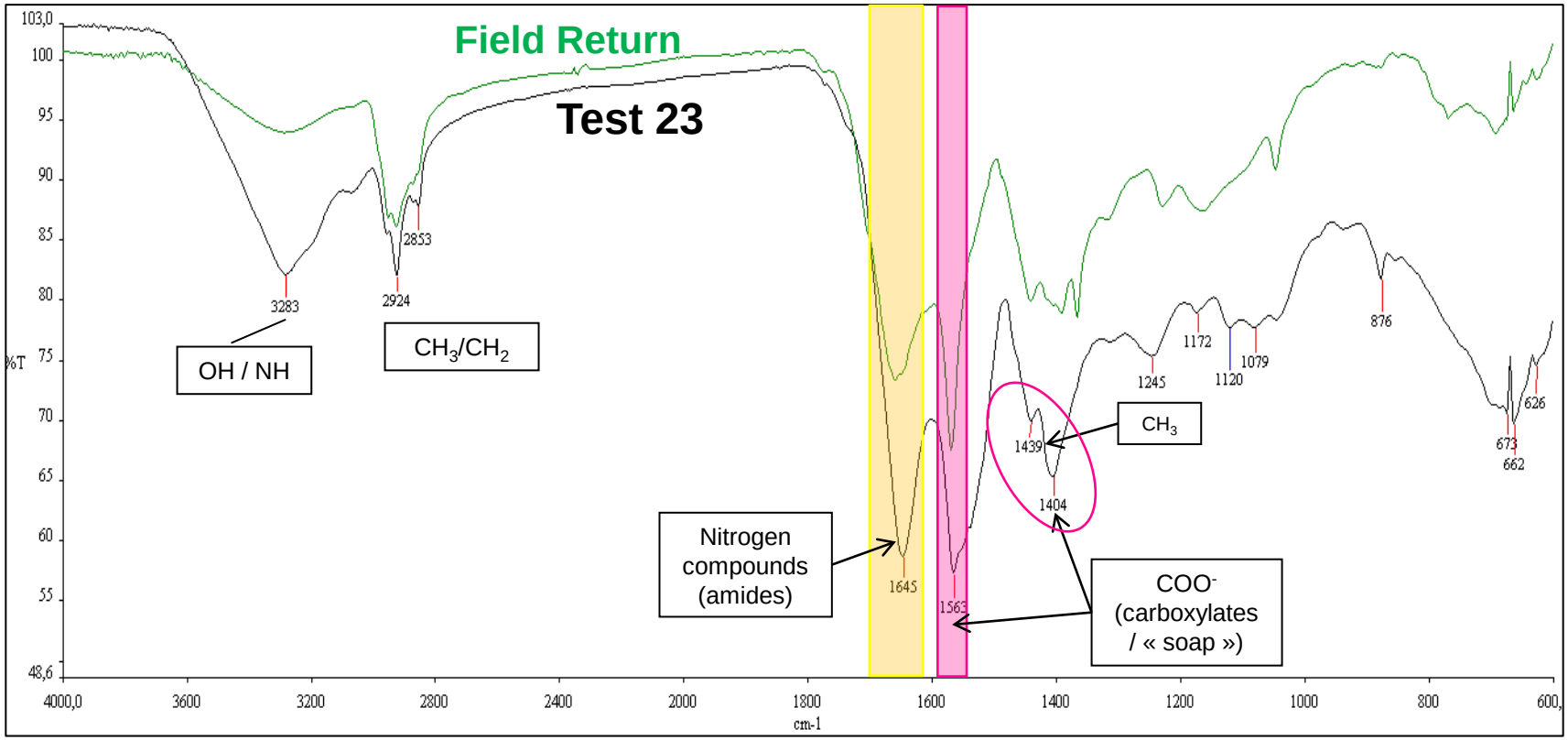
Again Deposit Thickness Increases with Concentration



NaOH + DDSa + LMW PIBSI Reproduces Field Returns That Show Metal Carboxylate & Amide Deposits

Carboxylate and Amide Deposit Widely Reported in the Literature

Frequently reported combination in EU



(E120 3481 HEF) **Test 23 = UKNoDep + 13.2 mg/L NaOH + 225 mg/L DDSA + 110 mg/L LMW PIBSI**

Summary of Results Obtained with Each Fuel

- **Good IID Separation Obtained Between "Good" and "Bad" Fuels**
- **Test Forms Appropriate Internal Injector Deposit Chemistries**

Test Fuel	Deposit Expected	Deposit Observed	Deposit Chemistry
UKNoDep	No	None	No Deposit
CARB Diesel	No	None	No Deposit
South American	Yes(?)	Very little	Not similar to any common field issue
UKDep[1]	Yes	Yes	Duplicates sodium carboxylate field issues
UKDep + LMW PIBSI	Yes	Yes	Duplicates sodium carboxylate and amide field issues

1) Note Ethanol may be a preferable co-solvent to water for NaOH

Summary and Conclusions

- **CRC Fuels Matrix Evaluated in Delphi IID Test Stand**
  - Additional tests with LMW PIBSI performed by Delphi
- **IID Test Methodology Sensitive to Fuel Composition**
  - No Deposit with California (CARB) Fuel
  - Very slight deposit with South American Fuel
  - No deposit with pure hydrocarbon (UKNoDep)
  - Strong deposit with UKDep fuel
  - Strong Deposit with UKDep + LMW PIBSI
- **Treated Fuels Produced "Expected" Deposit Chemistry**
  - UKDep (NaOH +DDSa) → Sodium carboxylate salt
  - UKDep + LMW PIBSI → Sodium carboxylate salt and amide deposit
- **Longer Test Duration Required at Low Contaminant Concentrations**
  - Sufficient deposit for FTIR following 7 hours
  - Extended 28 hour test produces measurable deposit thickness

Next Steps

- Results from this limited scoping study suggests that fuels can be discriminated
- A more comprehensive program is required to confirm the capability and also to establish correlation with actual diesel engines
- A cooperative funded program between Delphi and the EMA members is proposed for the next step
- If meaningful correlation can be established, the test rig can be set up at a U.S. research facility for future evaluation of fuels and additives
- A subsequent study at that time can determine the effects of other parameters such as biodiesel, additives, and impurities

Appendix 1

Fuels and Fuel Analysis

Blend Instructions for UKDep Fuel

Blend Instructions Received from Afton Chemical

– Received from Alex Kulinowski, 4 Dec 2012

Steps

1) Blend up a 17.3% solution of NaOH (caustic soda) in water.
   (So add 17.3 grams of NaOH to 82.7 grams of water)
2) Take 1 L of UKNoDep Diesel fuel.
3) To the 1L of fuel add 140 mg DDSa
   (i.e. this will be 20 mg/L in the finished 7L, as instructed below)
4) Place 0.210 grams of the water/NaOH solution into the 1L of fuel
   (this is equivalent to 0.0363 g NaOH or 0.0208 g of Na)
5) Sonicate the 1L for 10 minutes
6) Pour the 1 L into the 6 L to come up with a total test volume of 7L
   (final Na Conc = 0.0208/7 = 2.971 mg/L of Na or 5.18 mg/L of NaOH)

Final sodium blend concentration = 2.971 mg/L

Active DDSa concentration 9 mg/L (@ 45% active)

Other Additive Ratios Blended in the Same Manner

– At concentrations defined in table on subsequent slide
– Above recipe demands water as a cosolvent

Properties for CARB & South American Diesel by BP

Test	Method	units	CARB	SA
Sulfur	D7039 (XRF)	ppm-w	8	69
Biodiesel Content	Infracal (IR)	%	0	0
Stability, 140°C	D7545 (Petro Oxy)	minutes	83.1	135.1
Stability, 155°C	D7545 (Petro Oxy)	minutes	29.1	43.5
Water Separation	D7261 (MSEP)			
ICP	D7455 / D7578	ppm	-	-
Al			<0.162	<0.162
Ba			<0.042	<0.042
Ca			<0.034	<0.034
Cd			<0.015	<0.015
Co			<0.034	<0.034
Cr			<0.021	<0.021
Cu			<0.040	<0.040
Fe			<0.021	<0.021
K			<0.025	<0.025
Li			<0.035	<0.035
Mg			<0.047	<0.047
Mn			<0.032	<0.032
Mo			<0.006	<0.006
Na			<0.029	<0.029
Ni			<0.033	<0.033
Pb			<0.061	<0.061
Ti			<0.005	<0.005
V			<0.027	<0.027
Zn			<0.011	<0.011
Zr			<0.001	<0.001
API Gravity	D4052	°API	37.7	36.2
Specific Gravity @60°F			0.8365	0.8440
Cloud Point	D5773 (Phase Tech)	°C	-12	-2
		°F	10	28
Pour Point	D5949 (Phase Tech)	°C	-18	-3
		°F	0	27
Distillation	D86	°F	-	-
IBP			343	283
10%			412	417
50%			513	530
90%			618	650
FBP			663	699

Properties for UKNoDep Diesel by Delphi

(Specialty Diesel Containing Only Lubricity Additive)

Test	Method	Unit	Limit		Result
			Min	Max	
Appearance	Visual		Report		C&B
Cetane Number	ASTM D613		52	54	52.1
Cetane Index	EN ISO 4264		Report		51.6
Marking			Report		Dyed
Density @ 15°C	EN ISO 12185	kg/L	0.833	0.837	0.8353
Polycyclic Aromatics	EN 12916	% m/m	3	6	3.1
Total Aromatics	EN 12916	% m/m	Report		24.4
Total Aromatics	ASTM D1319	% v/v	Report		22.1
Olefins	ASTM D1319	% v/v	Report		0.8
Saturates	ASTM D1319	% v/v	Report		77.1
Sulfur	EN 20846	mg/kg	6	10	10.0
Flash Point	EN ISO 2719	ºC	55	-	65.5
Carbon Residue (10% Dis. Res)	EN ISO 10370	% m/m	-	0.2	<0.01
Ash	ASTM D482	% m/m	-	0.01	<0.001
Water and Sediment	ASTM D2709	% m/m	-	0.02	0
Copper Corrosion (3h at 50ºC)	ASTM D130	rating	1	-	1a
Fatty Acid Methyl Ester (FAME) Content	EN 14078	% v/v	Report		<0.1
Oxidation Stability	ASTM D2274	g/m^3	-	25	1
Lubricity, Corrected Wear Scar Diameter (WSD 1.4) at 60ºC	EN ISO 12156-1	µm	-	400	226
Viscosity at 40ºC	ASTM D445	mm^2/s	2.3	3.3	2.553
Strong Acid Number	ASTM D974	mgKOH/g	-	0.02	0
Gross Calorific Value	IP 12	MJ/kg	Report		45.56
Net Calorific Value	IP 12	MJ/kg	Report		42.75
Carbon Content	ASTM D5291	% m/m	Report		86.75
Hydrogen Content	ASTM D5291	% m/m	Report		13.25
Oxygen Content	Calculation	% m/m	Report		0
H/C	Calculation		Report		1.820
CFPP	EN 116	ºC	-	-15	-31
Cloud Point	ASTM D2500	ºC	-	-5	-11

Properties for UKNoDep Diesel by Delphi (Cont.)

Test	Method	Unit	Limit		Result
			Min	Max	
Distillation					
% v/v Evaporated at 250ºC	EN ISO 3405	% v/v	Report		38.7
% v/v Evaporated at 350ºC	EN ISO 3405	% v/v	Report		95.8
IBP	ASTM D86	ºC	Report		172.6
10% Volume Evaporated	ASTM D86	ºC	Report		205.8
20% Volume Evaporated	ASTM D86	ºC	Report		220.9
30% Volume Evaporated	ASTM D86	ºC	Report		236.1
40% Volume Evaporated	ASTM D86	ºC	Report		252.1
50% Volume Evaporated	ASTM D86	ºC	245	280	265.5
60% Volume Evaporated	ASTM D86	ºC	Report		278.4
70% Volume Evaporated	ASTM D86	ºC	Report		291.0
80% Volume Evaporated	ASTM D86	ºC	Report		306.4
90% Volume Evaporated	ASTM D86	ºC	Report		327.9
95% Volume Evaporated	ASTM D86	ºC	345	350	345.1
FBP	ASTM D86	ºC	-	370	356.7
Residue	ASTM D86	% v/v	Report		1.4

Element	Mg/kg
Sodium	<0.1
Zinc	<0.1
Copper	<0.1
Silicon	<0.1
Phosphorous	<0.1

Properties for CARB & South American Diesel by ExxonMobil

Property	Unit	CARB	South America
Sufur content(D5453)	mg/kg	9.9	66.4
FAME Content (EN14078)	%(V/V)	<0.05	<0.05
Oxidation Stability (D2274)			
Adhernt Insolubles	mg/100ml	<0.10	<0.10
Total Insolubes	mg/100ml	<0.10	0.1
Filterable Insolubes	mg/100ml	<0.10	0.1
Water Deparation (D7261)	DSEP	0	0
ICP for Metals (D5708_MOD)			
Cobalt	mg/kg	<0.100	<0.100
Barium	mg/kg	<0.100	<0.100
Boron	mg/kg	<0.100	<0.100
Cadmium	mg/kg	<0.100	<0.100
Calcium	mg/kg	0.805	0.927
Chromium	mg/kg	<0.100	<0.100
Copper	mg/kg	0.265	0.100
Iron	mg/kg	0.292	0.220
Lead	mg/kg	<0.100	<0.100
Magnesium	mg/kg	0.126	0.136
Manganese	mg/kg	<0.100	<0.100
Molybdenum	mg/kg	<0.100	<0.100
Nickel	mg/kg	<0.100	0.106
Potassium	mg/kg	0.331	0.288
Sodium	mg/kg	0.683	0.665
Tin	mg/kg	<0.100	<0.100
Selenium	mg/kg	<0.100	<0.100
Vanadium	mg/kg	<0.100	<0.100
Zinc	mg/kg	<0.100	<0.100
Cloud Pont (D5773)	°C	-12	-2
Pour Point (D5949)	°C	-18	-9
API Gravity at 60 F (D4052)	API	37.5	35.9
Distillation (D86)			
IBP	°F	351.9	309.4
10%	°F	417.6	424
50%	°F	515.6	531.1
90%	°F	619.4	651
FBP	°F	664.3	708.8

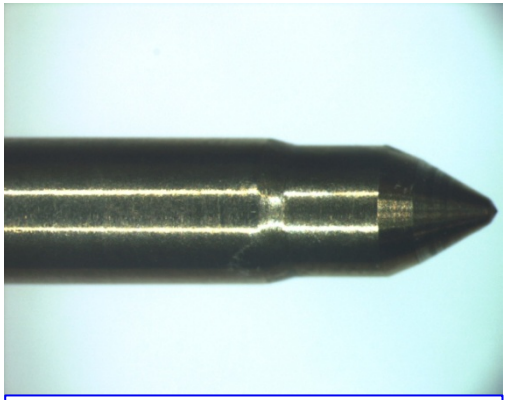
Properties for CARB & South American Diesel by Innospec

Property	Result	Result2
Sulfur D7039	7.5 mg/kg	74.8 mg/kg
Biodiesel Content (IOSP Internal Method)	0%	0%
Oxidation Stability D7545 @ 140ºC	85 min. 13 sec.	122 min. 19 sec.
Oxidation Stability D7545 @ 155ºC	27 min. 49 sec.	25 min. 36 sec.
Water separation D7261	DSEP Rating 82	DSEP Rating 86
ICP for Metals	ppm	ppm
Iron	<1	<1
Chromium	<1	<1
Nickel	<1	<1
Aluminum	<1	<1
Copper	<1	<1
Lead	<1	<1
Tin	<1	<1
Cadmium	<1	<1
Silver	<1	<1
Vanadium	<1	<1
Silicon	<1	<1
Sodium	<1	<1
Potassium	<1	<1
Titanium	<1	<1
Molybdenum	<1	<1
Antimony	<1	<1
Manganese	<1	<1
Lithium	<1	<1
Boron	<1	<1
Magnesium	<1	<1
Calcium	<1	<1
Barium	<1	<1
Phosphorous	<1	3
Zinc	<1	<1
Cloud Point D5773	-12ºC / 10.4ºF	-2ºC / 28.4ºF
Pour Point D5950	-18ºC / -0.4ºF	-9ºC / 15.8ºF
API Gravity D4052	37.78º	36.03º
Distillation D2887 (D86	ºF	ºF
IBP	351.0	205.5
10%	415.9	385.7
50%	515.7	543.4
90%	620.4	691.0
FBP	677.8	827.1

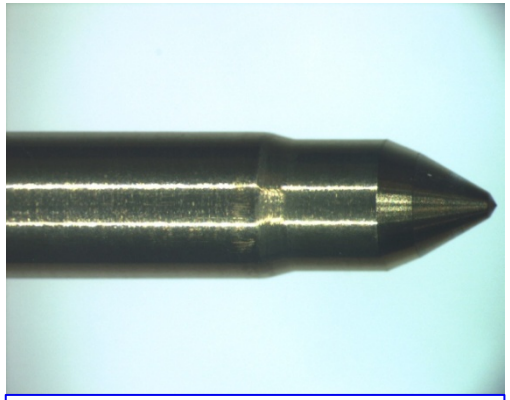
Appendix 2

Photographs of All Tests

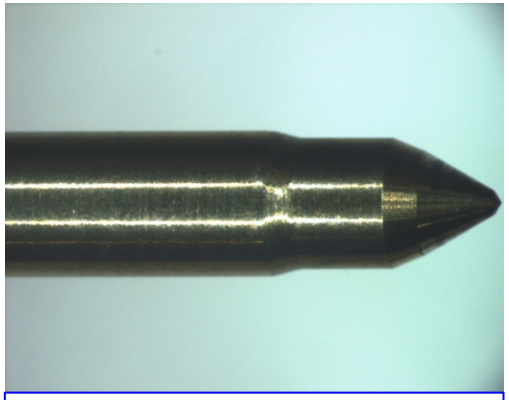
View of Injector Needles
From CRC Defined Test Matrix



Test 1: UKNoDep Diesel

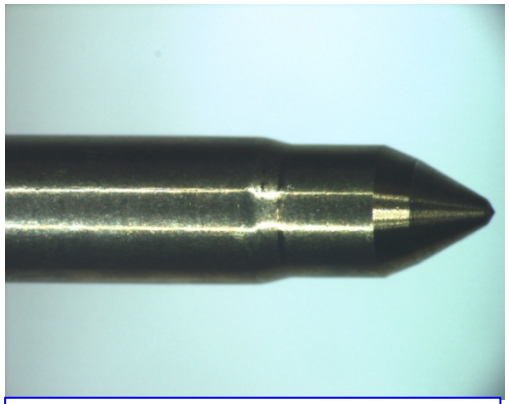


Test 2: CARB Diesel

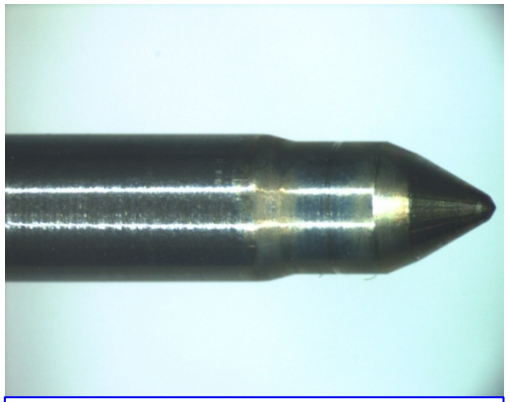


Test 3: UKNoDep Diesel

Test 4: UKDep Diesel

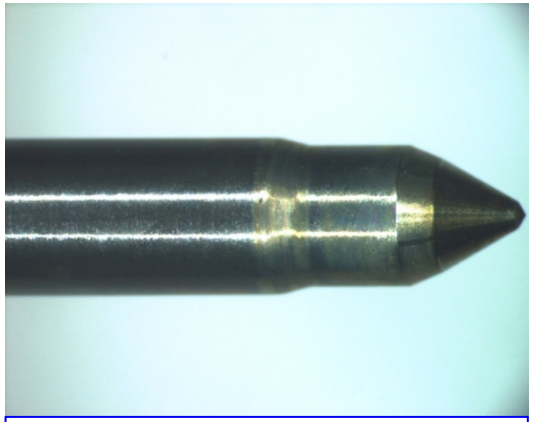
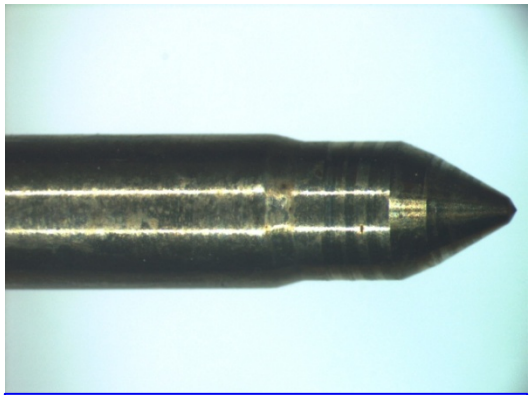
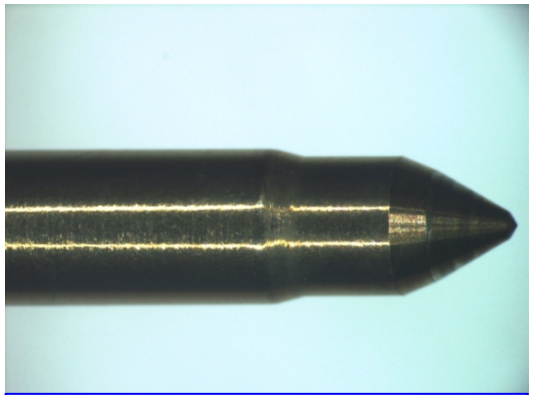
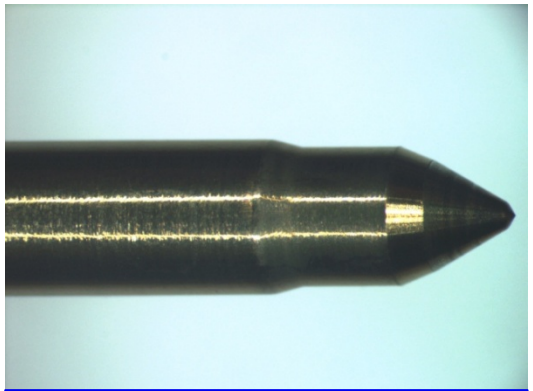


Test 5: UKNoDep Diesel

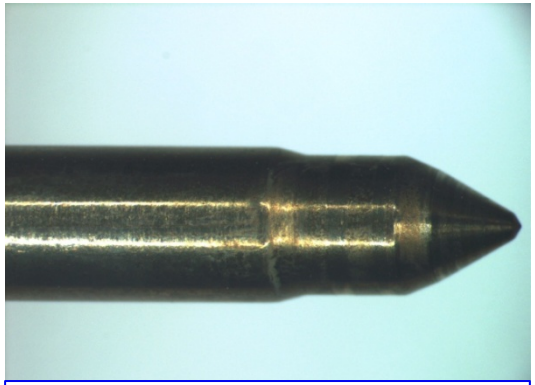


Test 6: South American Fuel

View of Injector Needles from CRC Defined Test Matrix (Continued)

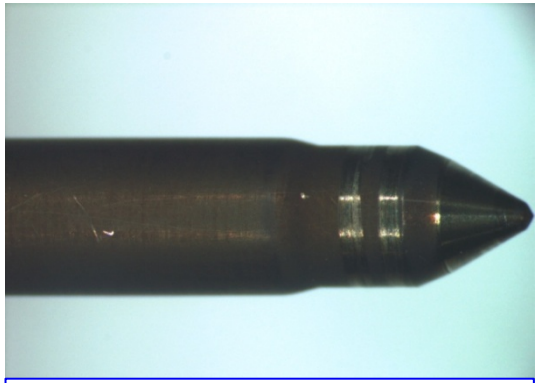
Test 7: (Rinse Test) Performed but Halted Early due to Technical Issues	 **Test 8: South American Fuel**	Test 9: (Rinse Test) Performed but Halted Early due to Technical Issues
 **Test 10: UKDep Diesel**	 **Test 11: UKNoDep Diesel**	 **Test 12: CARB Fuel**

View of Needles from Supplemental Test Matrix



Test 13: UKDep (2 day test)
(NaOH in Water)

Test 14: UKDep
(4 day test)



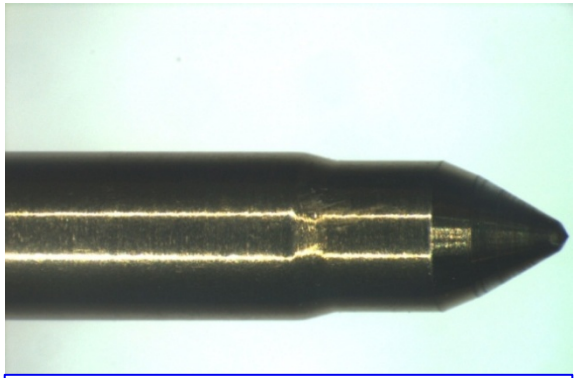
Test 16: UKNoDep+Increased Add. Conc (13.2mg/L NaOH(in ethanol)+ 225mg/LDDSA)

Test 17: UKDep (2 day test)
(NaOH in Ethanol)

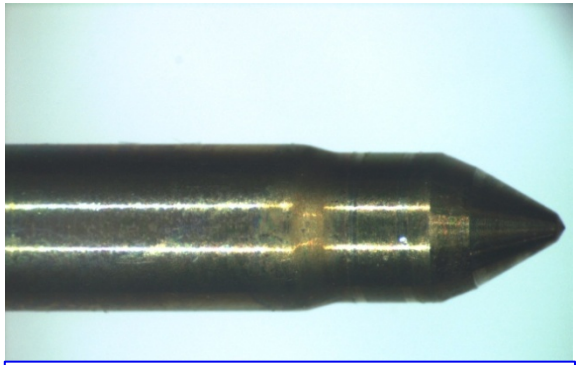
Test 18: South American Fuel
(4 day test)

Test 19: UKNoDep
(4 day test)

View of Injector Needles from Supplemental Test Matrix (Continued)



Test 20: UKNoDep Diesel

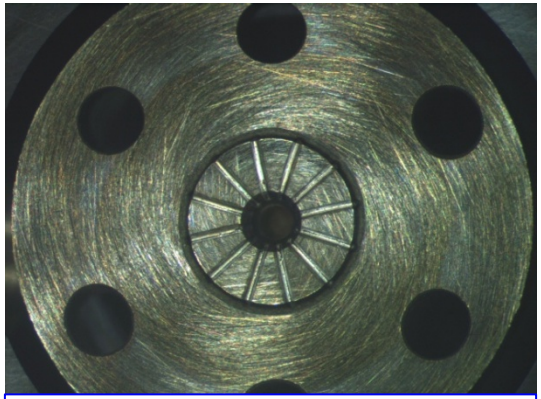


Test 21: UKNoDep +3.3 mg/L NaOH+ 56mg/L DDSA+27.5 mg/L LMW PIBSI

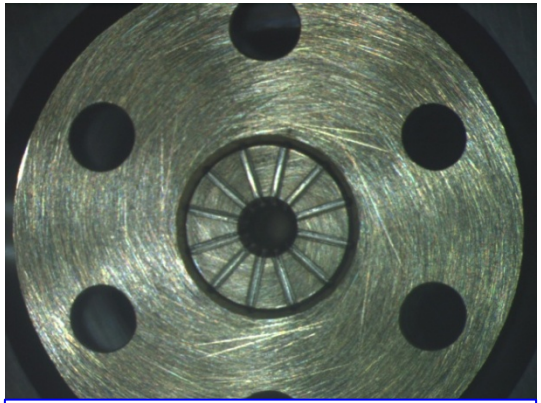
Test 22: UKNoDep+6.6 mg/L NaOH+ 112mg/L DDSA+55mg/L LMW PIBSI

Test 23: UKNoDep+13.2 mg/L NaOH+ 225mg/L DDSA+110mg/L LMW PIBSI

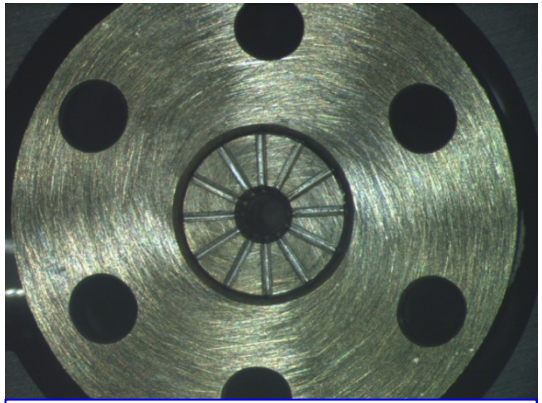
View of Armature with Fuels from CRC Defined Test Matrix



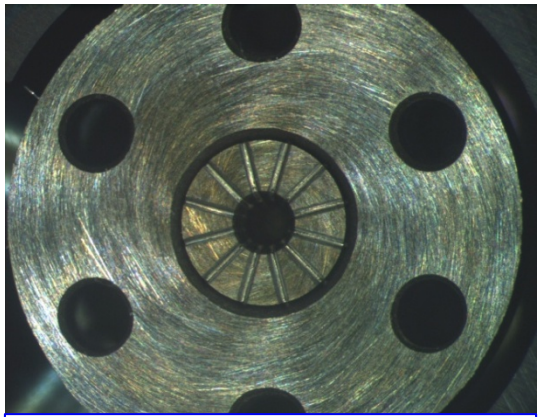
Test 1: UKNoDep Diesel



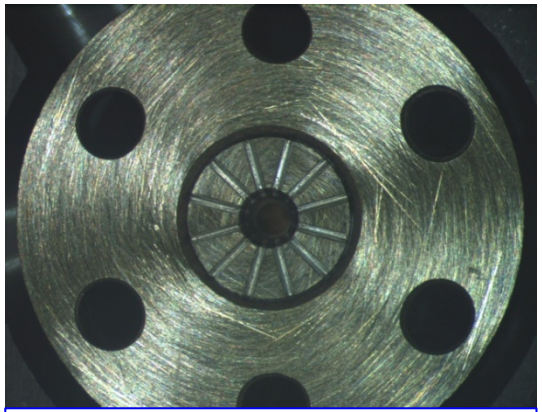
Test 2: CARB Fuel



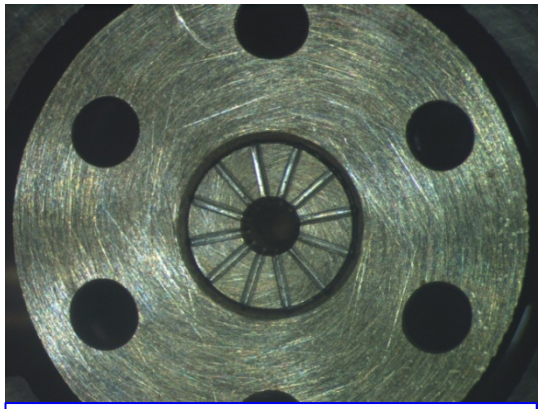
Test 3: UKNoDep Diesel



Test 4: UKDep Diesel
(5.23mg/L NaOH(in water)+ 9 mg/L DDSA)

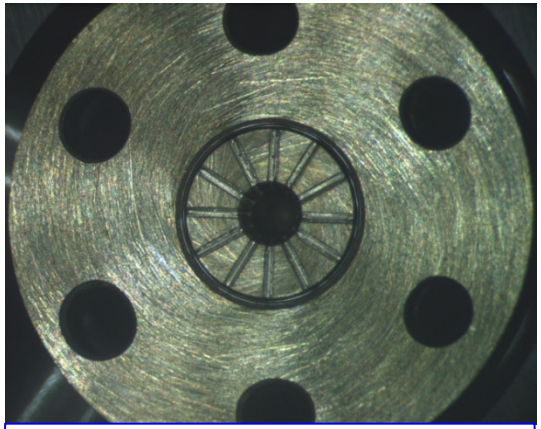
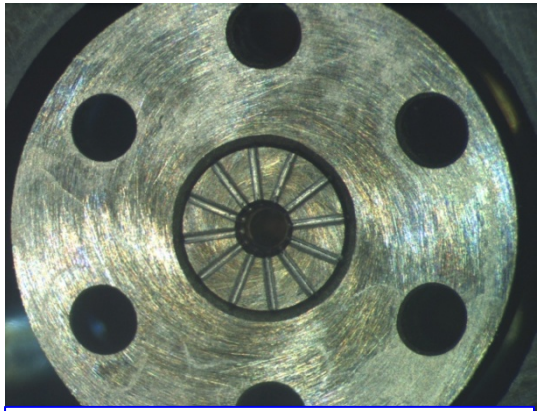
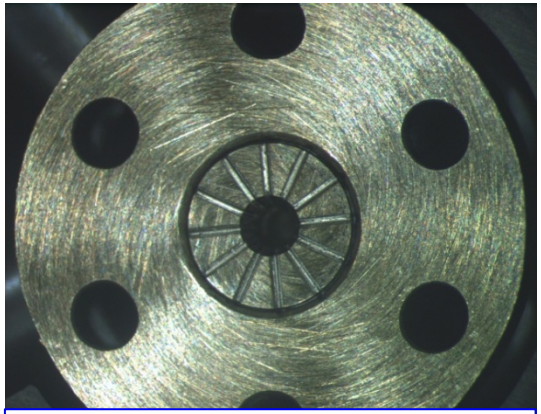
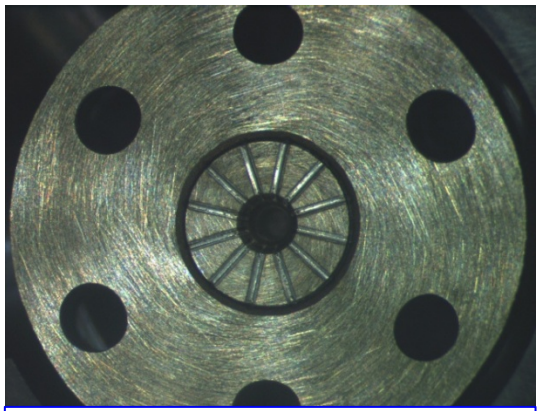


Test 5: UKNoDep Diesel

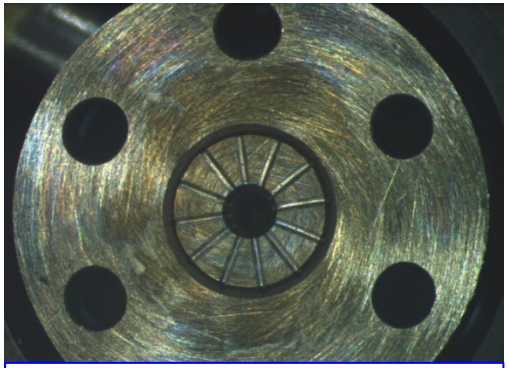


Test 6: South American Fuel

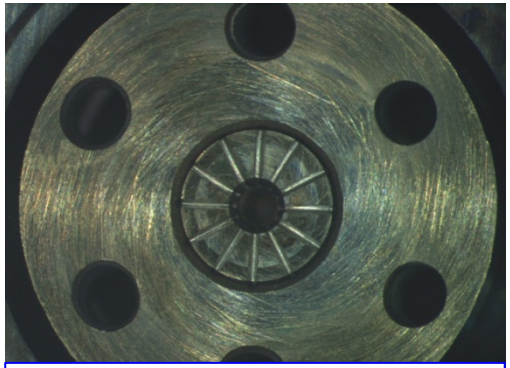
View of Armatures with Fuels from CRC Defined Test Matrix (Continued)

Test 7: (Rinse Test) Halted due to Technical Issues	 **Test 8: South American Fuel**	Test 9: (Rinse Test) Halted Early due to Technical Issues
 **Test 10: UKDep Diesel (5.23mg/L NaOH(in water)+ 9mg/l DDSA)**	 **Test 11: UKNoDep Diesel**	 **Test 12: CARB Fuel**

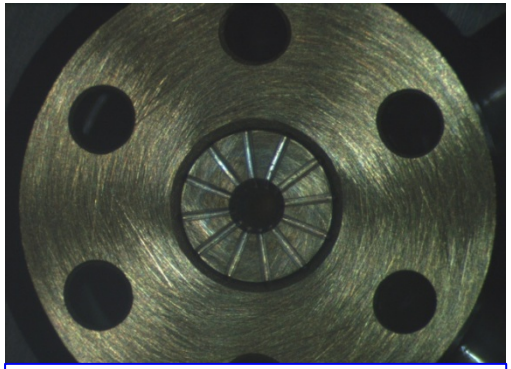
View of Armatures with Fuels from Supplemental Test Matrix



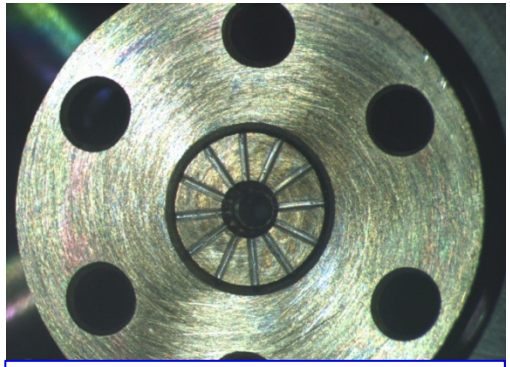
Test 13: UkDep (5.23mg/l NaOH(in water)+ 9mg/L DDSA) (2 day test)



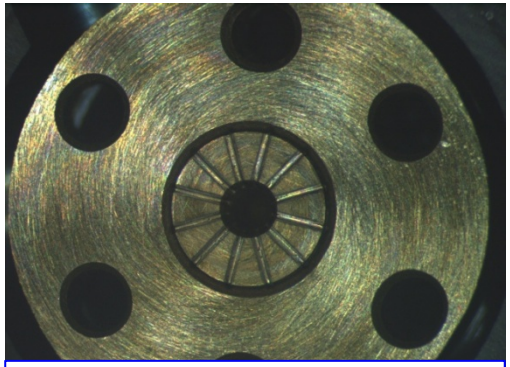
Test 14: UKDep (5.23mg /L NaOH(in ethanol)+ 9mg/L DDSA) (4 day test)



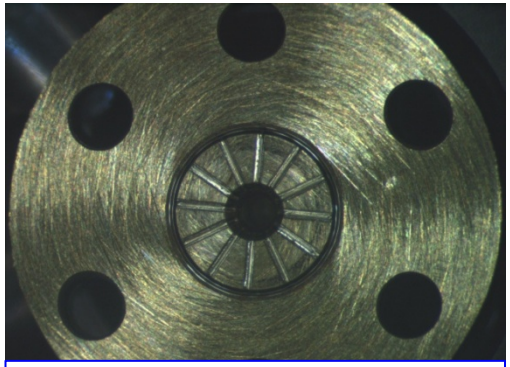
Test 16: UKNoDep+Increased Add. Conc 13.2mg/L NaOH(in ethanol)+ 225 mg/L DDSA



Test 17: UKDep (2 day test) 5.23mg/L NaOH(in ethanol)+ 9 mg/l DDSA

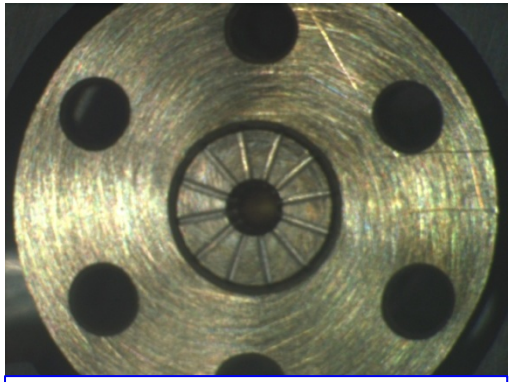


Test 18: South American Fuel (4 day test)

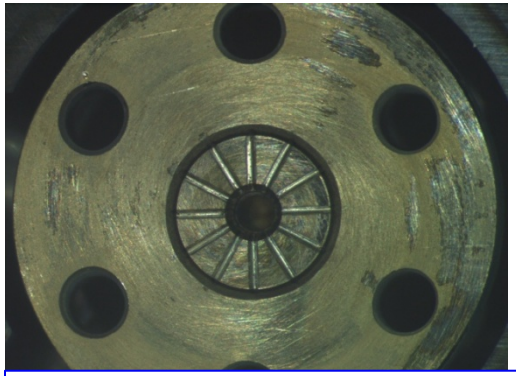


Test 19: UKNoDep Diesel (4 day test)

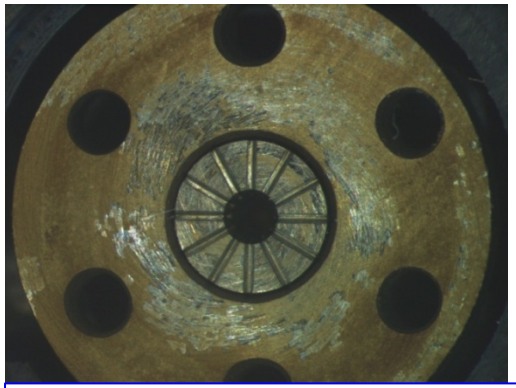
View of Armatures with Fuels from Supplemental Test Matrix (Continued)



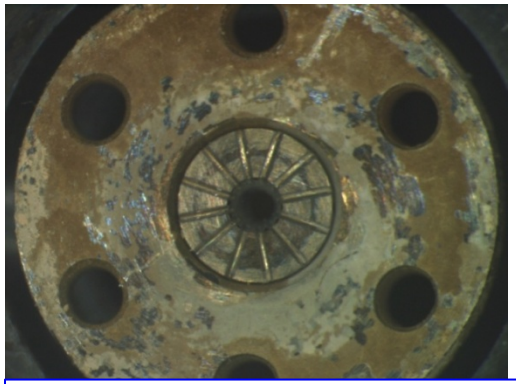
Test 20: UKNoDep Diesel



Test 21: UKNoDep+3.3 mg/L NaOH+ 56mg/L DDSA+27.5 mg/L LMW PIBSI



Test 22: UKNoDep+6.6 mg/L NaOH+ 112mg/L DDSA+55 mg/L LMW PIBSI



Test 23: UKNoDep +13.2 mg/L NaOH+ 225mg/L DDSA+110 mg/L LMW PIBSI

Appendix 3

Deposit Thickness Measurements

Deposit Thickness Measurement

Performed Using Zygo Interferometer and/or Tallysurf

-Zygo not capable of measuring black deposits

Reported Results are the Average of Several Measurements

Measurement Taken from Surface of Deposit to Metal

-Groove cut in deposit to allow measurement

Both the metal Surface and Especially the Deposit have Roughness

-Best fit line visually placed through both surfaces

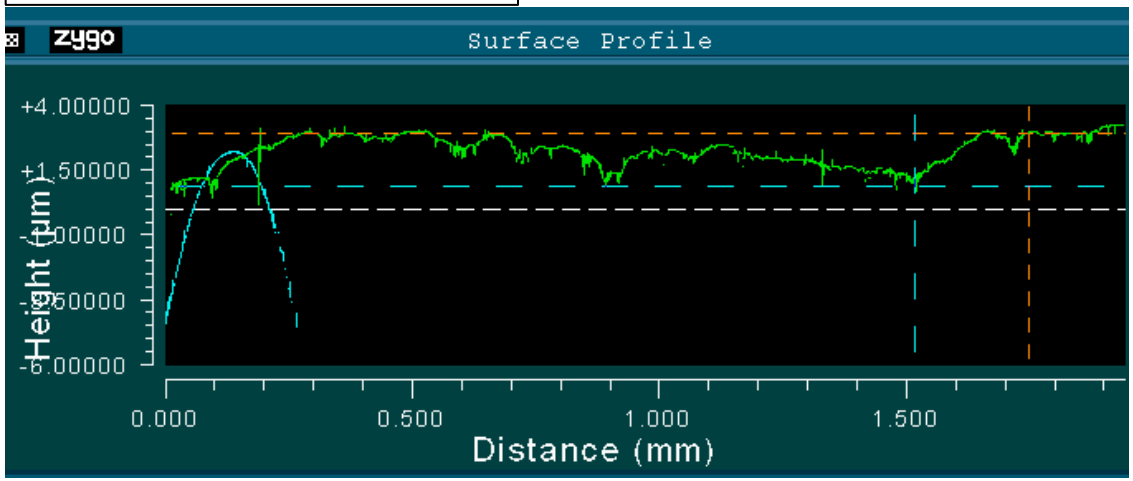
-Distance between lines represents thickness

-This approach used for both Zygo and Tallysurf plots

Note: Dotted Lines on Zygo Profiles are Added by Software and Should be Disregarded

Deposit Thickness Measurement from Test 2
(8 Hr Test with CARB Fuel)

Zygo Interferometer



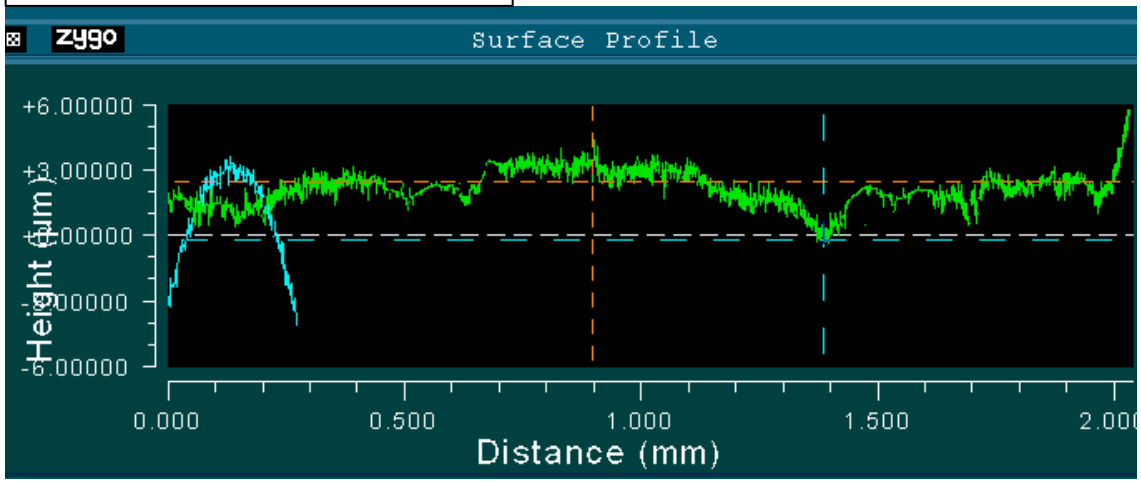
Deposit Thickness Similar to Component Surface Roughness

Talysurf

E120 8163 GEF

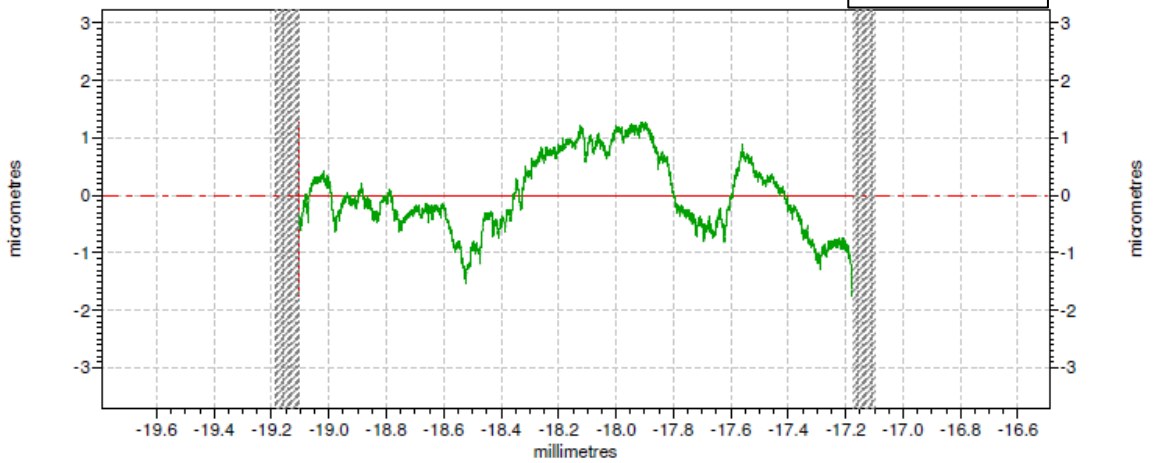
Deposit Thickness Measurement from Test 4
(8 Hr Test with UKDep Diesel)

Zygo Interferometer



Deposit Thickness Similar to Component Surface Roughness

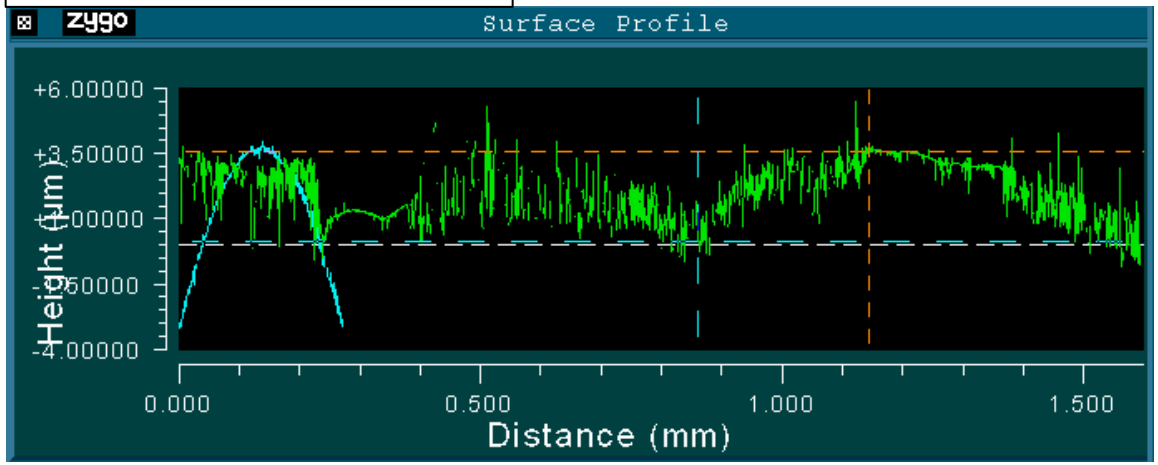
Talysurf



E120 3351 HEF

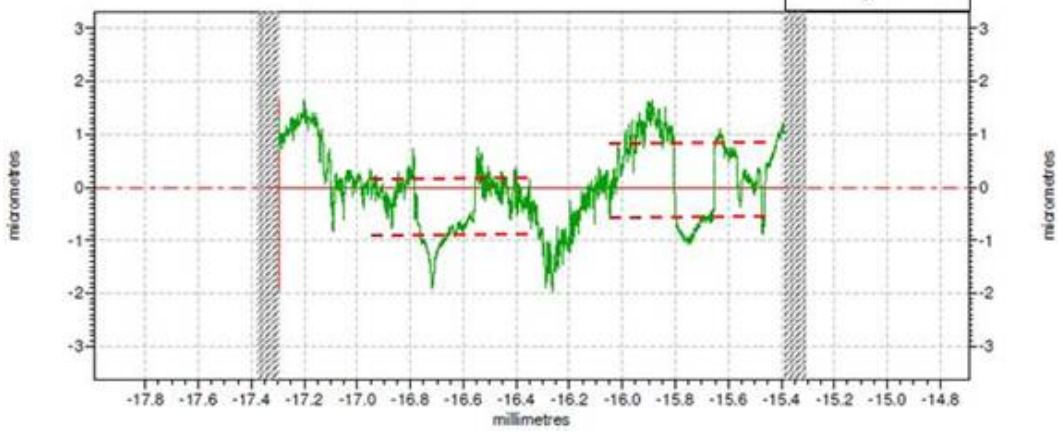
Deposit Thickness Measurement from Test 14
(28 Hour Test with UKDep Diesel)

Zygo Interferometer



Depth = 1.35 micron

Talysurf

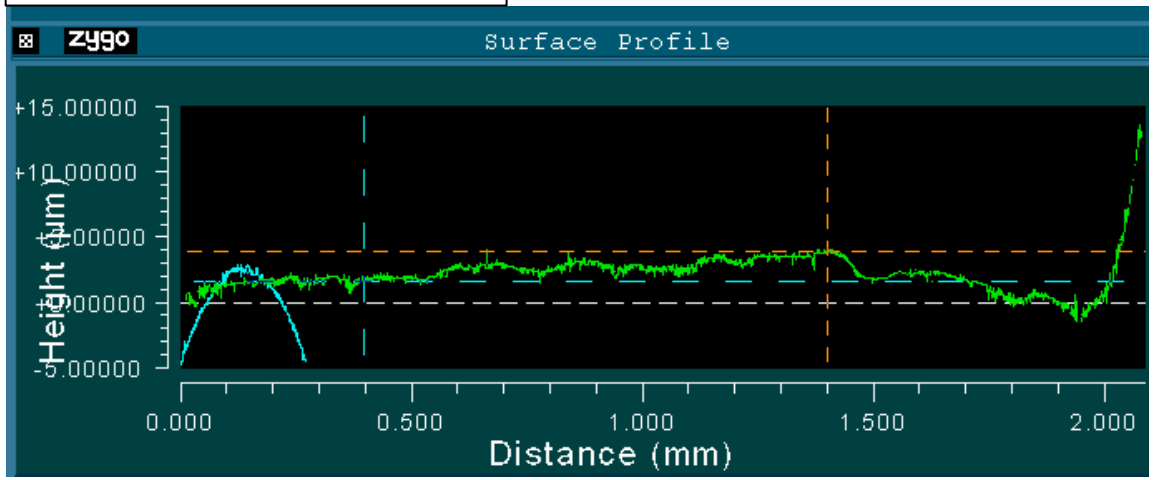


C120 385 FEF

Deposit Thickness Measurement from Test 21

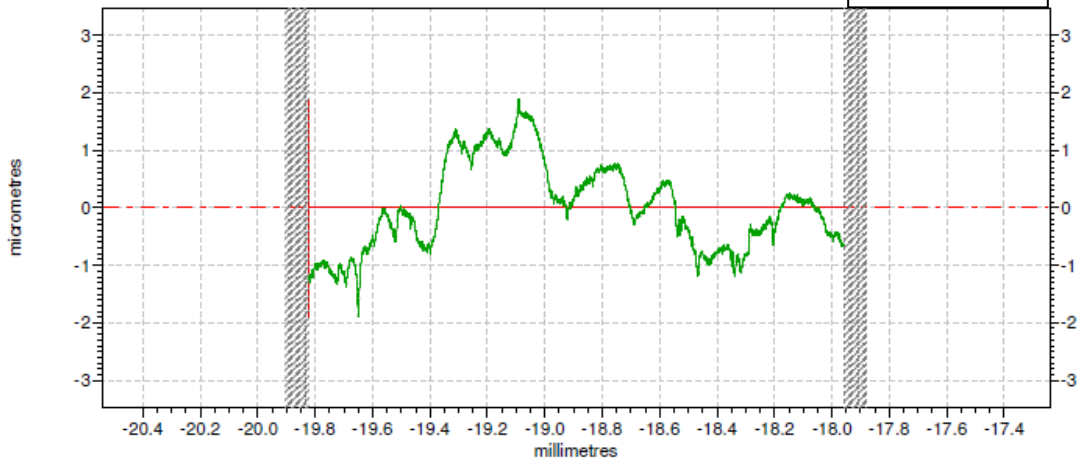
(UKNoDep + 3.3mg/L NaOH + 56 mg/L DDSa + 27.5 mg/L LMW PIBSI)

Zygo Interferometer



No Measurable Deposit

Talysurf



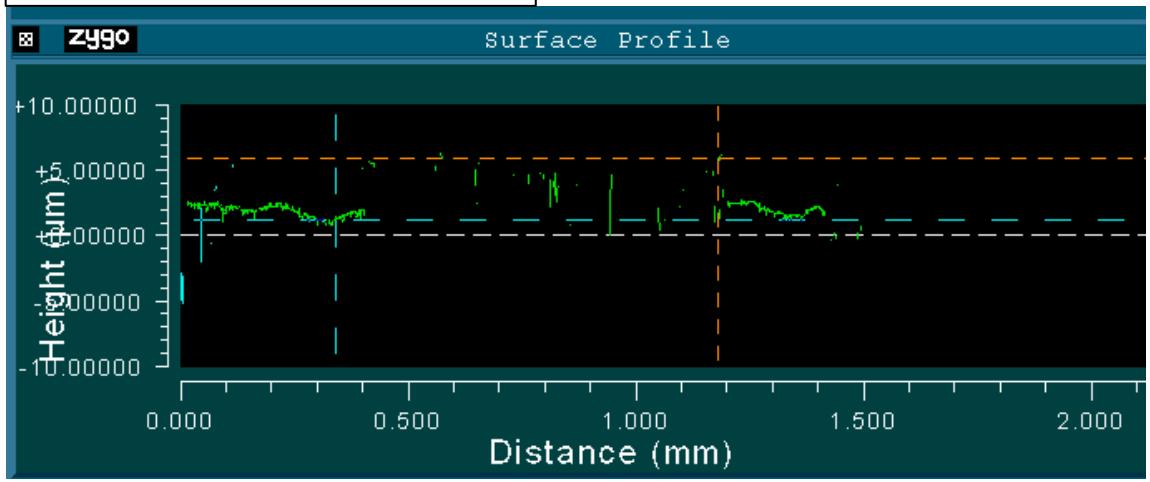
E120 3636 HEF

Deposit Thickness Measurement from Test 22

(UKNoDep + 6.6 mg/L NaOH + 112 mg/L DDSa + 55 mg/L LMW PIBSI)

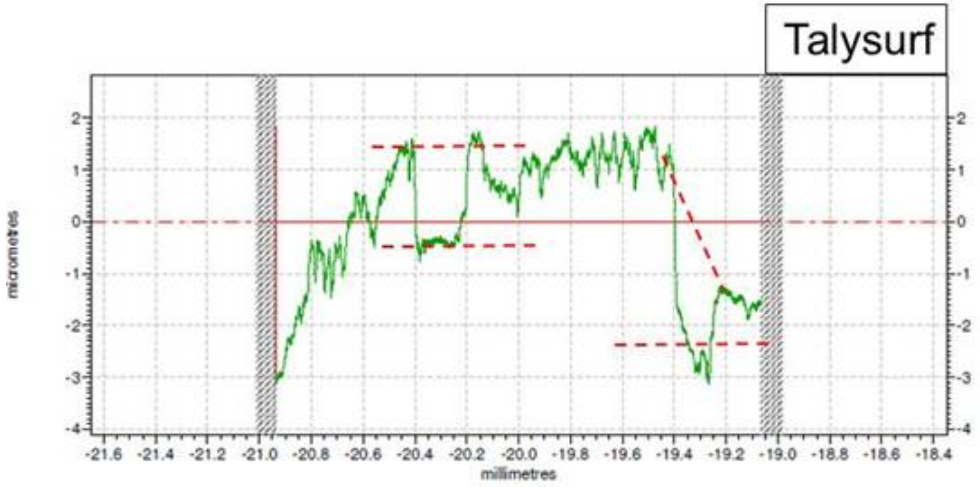
Zygo Interferometer

Poor Reflectance from dark deposit in Zygo



Deposit Thickness ≈2.1µm

Talysurf



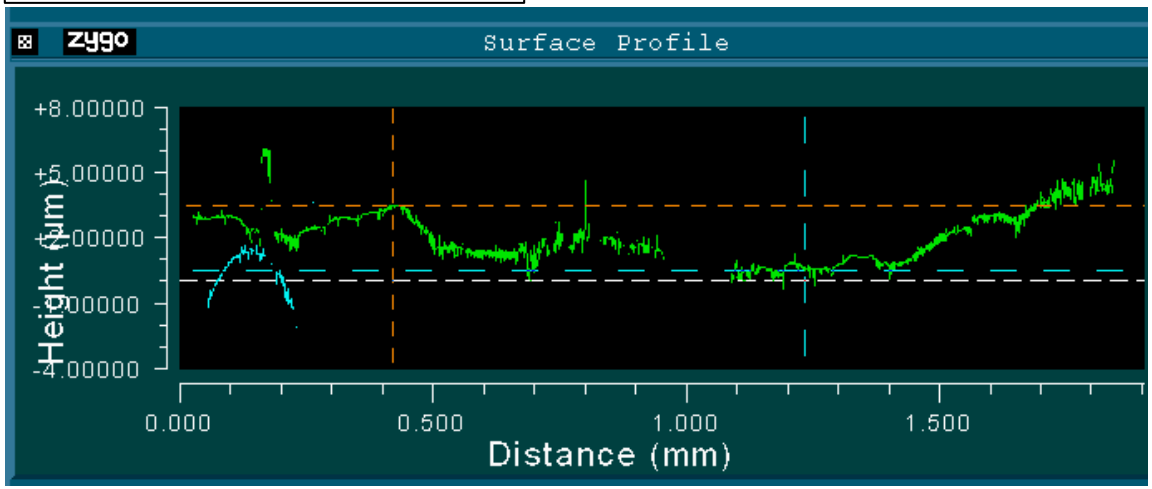
E120 3652 HEF

Deposit Thickness Measurement from Test 23

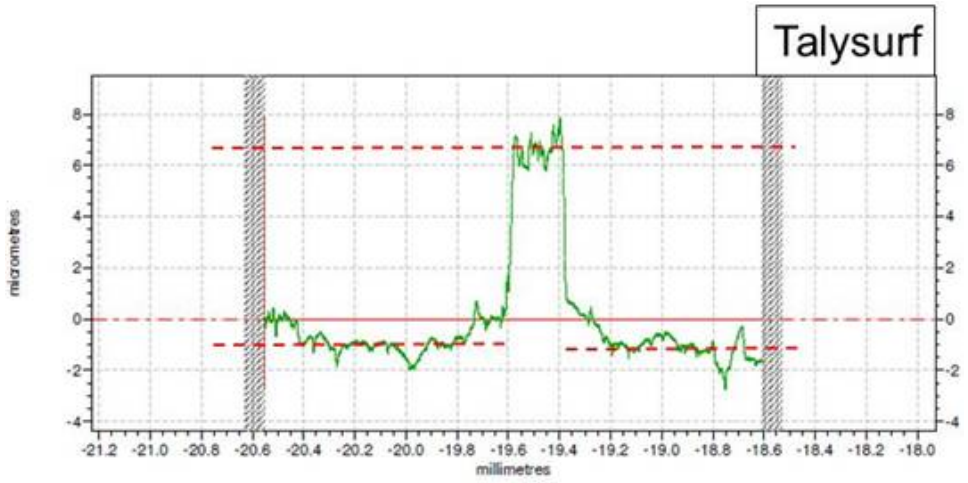
UKNoDep+ 13.2 mg/L NaOH (etOH) + 225 mg/L DDSA + 110 mg/L LMW PIBSI

Zygo Interferometer

Poor Reflectance from dark deposit in Zygo



Deposit Thickness ≈ 3.5µm



E120 3481 HEF