CRC Report No. 665

INTERNAL DIESEL INJECTOR DEPOSITS

CRC Project No. DP-04-13a

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COORDINATING RESEARCH COUNCIL, INC.

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Prepared by the Deposit Analysis Panel
of the
CRC Diesel Performance Group

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CRC Performance Committee
of the
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Internal Diesel Injector Deposits

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Summary

Internal diesel injector deposits (IDID) in high pressure common rail fuel injection systems are a relatively new problem facing original equipment manufacturers (OEMs) across the globe and in both light duty diesel and heavy duty diesel applications. These deposits can slow the response, or cause sticking, of moving internal injector parts, resulting in a loss of control of injection event timing and/or the amount of fuel delivered. Due to the complex injection sequences utilized in common rail engines, the effects of IDID on performance range widely but can include poor operability, reduced fuel economy and power, increased emissions, failure to start and, in extreme cases engine failure.

An evaluation of the open literature and information presented to CRC by EMA and participating companies, through March 2013, leads to the following conclusions:

- Modern common rail fuel injection systems can be less tolerant of deposits due to tighter tolerances, lower mass parts and sophisticated injection strategies. At the same time, these fuel systems provide a more reactive environment due to higher operating pressures and temperatures.
- The inherent role of ultra-low sulphur diesel (ULSD) or of biodiesel, if any, is unclear but there is some correlation between the introduction of ULSD and/or use of biodiesel and the incidence of IDID.
- Metal carboxylate soap is the most common type of IDID in North America and globally, though other chemical types (e.g. amide lacquers) have been identified.
- A preponderance of industry data indicates that salts of Na or Ca with dodecenyl succinic acid (DDSA) and hexadecenyl succinic acid (HDSA) are highly likely to contribute to IDID.
- Controlled dynamometer engine tests have demonstrated the ability to replicate field problem symptoms in less than 100 hr using fuel dosed with Na/Ca and DDSA/HDSA, creating carboxylate soap deposits that match those in the field.
- Deposit control additive-based keep clean and clean up performance has been demonstrated in the field and in dyno engines.
- Investigations on sources of “lacquer” type deposits with confirmatory engine test data has focused on interactions of PIBSI with fatty acids, and the impact of low molecular weight PIBSI as deposit sources , but with less clear consensus than the Na soap deposits.

Recommendation

- Minimize access of Na/Ca to engines via housekeeping throughout the fuel (mineral and biodiesel) manufacture, distribution and storage system.

Introduction

Internal diesel injector deposits (IDID) in high pressure common rail fuel injection systems are a relatively new problem facing original equipment manufacturers (OEMs) across the globe and in both light duty diesel and heavy duty diesel applications. These deposits can slow the response, or cause sticking, of moving internal injector parts, resulting in a loss of control of injection event timing and/or the amount of fuel delivered. Due to the complex injection sequences utilized in common rail engines, the effects of IDID on performance range widely but can include poor operability, reduced fuel economy and power, increased emissions, failure to start and, in extreme cases engine failure.

Though the absolute number of injector failures in the field is difficult to estimate, the attention and interest to the problem is widespread. The number of engine and fuel system manufacturers who have reported on the problem is lengthy (1-8). Incidences have been reported on all continents but Antarctica (4, 6-8). Industry working groups have been formed. In the U.S., the heavy duty Truck and Engine Manufacturers Association (EMA) has engaged the Coordinating Research Council to investigate. Likewise in Europe, carmakers from European Automakers Association (ACEA) have identified IDID as an issue of concern and engaged the Coordinating European Council (CEC). This group has initiated action to develop an engine test to discriminate causes and solutions to both soap and lacquer type deposits. A CEN (European Committee for Standardization) ad-hoc task force on injector sticking was formed under TC19/WG24 specifically to address sodium-based IDID. Specific recommendations were made to remove Na-based pipeline corrosion inhibitors from use. In 2013 the group’s focus has shifted to amide-lacquer deposits.

Deposits internal to the injector are not new. The original direct injection deposit test, the Cummins L10, was based on rating needle deposits (9). However, there are significant differences between high pressure common rail (HPCR) and prior generation fuel injection equipment that contribute to the emergence of IDID as a significant performance issue.

First, HPCR systems are much more extreme environments, placing new demands on the fuel. HPCR systems operate at significantly higher pressures than prior generation technology. Higher injection pressures are driven by the need for improved fuel spray and injection control to meet emissions regulation and fuel consumption challenges. Since the introduction of the common rail system in the late-1990’s the operating pressures and temperatures have increased dramatically. Starting at around 1000 bar, the operating pressures today exceed 1800 bar and are anticipated to reach close to 3,000 bar in the future. Due to the heat generated during pressurization of the fuel at the high pressure pump and expansion during pressure let-down within the injector, fuel temperature can exceed 150C (3,6). The likelihood of chemical reactions with negative consequences occurring increases as the temperature and pressure increase. The thermodynamic and hydrodynamic regimes experienced under these extreme conditions are also far different than occurs in lower pressure systems, such that the behavior of sparingly soluble materials may be affected (6). In most systems a portion of the pressurized fuel is recycled back to the fuel tank, increasing its residence time in the fuel system and allowing further degradation.

Second, HPCR systems are more susceptible to the impact of deposits than prior generation systems. Because of elevated pressure operation, the tolerances between moving parts of HPCR systems is today measured in single digit microns. Even deposits of a few microns thickness can fill small gaps and cause moving parts like injector needles, command pistons and metering valves to bind or stick. The moving parts within injectors are an order of magnitude smaller and lighter than their unit injector predecessors (Figure 1), and thus carry lower inertia to resist the binding effects of deposits. Finally, HPCR systems use sophisticated injection strategies that can involve six or more discrete injections during a single combustion cycle. Delay or reduction in fuel volume during any one of these injections can have performance repercussions (2).

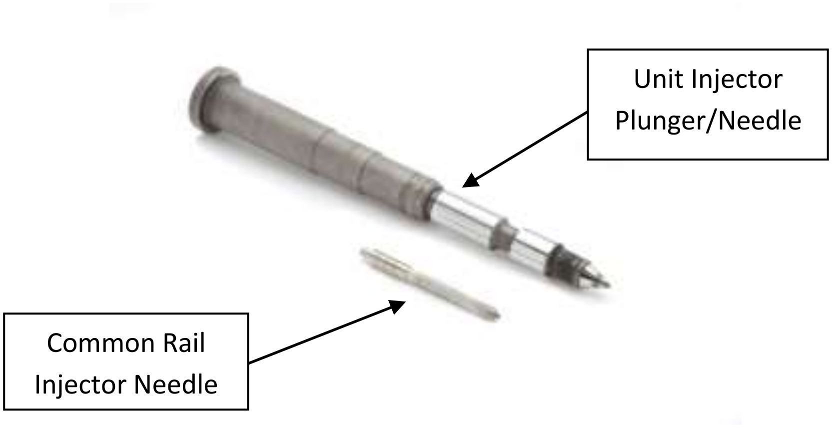

Figure 1 – Comparison of Unit Injector Plunger/Needle and Common Rail Injector Needle.

Several OEMs have pointed to a correlation between the introduction of ultra-low sulfur diesel (ULSD) and the occurrence of IDID (3, 4, 5, 27). Blizard (3) reported the incidence of IDID in the US correlated to the introduction of ULSD and Schmidt (4) reported that IDID problems became more prevalent in John Deere Tier 3 engines after the introduction of ULSD. Wilson (5) reported problems attributed to IDID at one fleet initiated directly after the introduction of ULSD. Whitacre (27) reported that problems attributed to IDID at an off-road fleet were alleviated when fuel supply was switched from ULSD to LSD. Hypothesis around this relationship are primarily secondary in nature: ULSD typically has lower aromatics than LSD, which may lead to lower solubility for sparingly soluble components; ULSD may have lower levels of natural acidic polar molecules to tie up Na or other metals in soluble form; reports of increased corrosion in ULSD distribution systems and difficulty in meeting pipeline NACE ratings may have resulted in increased use of corrosion inhibitors that may exacerbate the problem. Also, some data suggests biodiesel may contribute one or more of the necessary reactants (29, 30, 36). Cardenas Almena (30) and Lacey (29,36) both commented that FAME could contribute Na-ions, though their conclusions about the organic (acidic) contribution of FAME to IDID based on negative engine (30) and positive bench (36) rig results were contradictory.

As the problem has come to light, many stakeholders have since investigated the source of IDID (1-8, 10-24, 26-40). In an investigation into a problem such as this, a feedback loop process description can be employed. First, analysis of available facts is used to form a hypothesis. That hypothesis is then tested in simulations of the problem to gain a measure of validation. The loop is closed when validation is achieved via observation of real world situations consistent with the analysis. Within the context of the IDID problem, field experience provides the source of information and materials to be used in the analysis. Problem injectors are disassembled and studied; their deposits and fuel samples obtained from the field are analyzed. Investigators then typically seek supporting validation via bench or engine test simulations of the problem under controlled condition. Optimally, however, the loop is closed by validation of the hypothesis via real world conditions in the field.

This study consolidates industry investigations of the problem with respect to deposit and fuel analysis, causal theories derived from this data and bench, engine and field data supporting or disputing those theories. A summary is presented in which the theories are ranked according to the body of available evidence.

ANALYSIS AND HYPOTHESIS

The characteristic deposit types have been classed as “carboxylate soap,” “amide lacquer,” “inorganic salt” and “carbonaceous,” described further below.

Deposits with a characteristic sodium or calcium ion and carboxylate function are commonly referred to as “waxy” or “ soapy” deposits, based on their physical appearance and consistency. This type of deposit makes up the vast majority of those seen in North American heavy duty incidences and is also commonly cited of those observed in Europe. Investigators using SEM / EDX analysis identified the presence of metals sodium and/or calcium in at least a subset of deposits analyzed in nearly every work (1-8, 10-13, 15-24). FTIR spectroscopy, mass spectroscopy (MS), MS/MS and LC/MS and other methods have been used to establish functional molecular information and, again, in most cases have confirmed the presence of a carboxylate functional group (1-8, 10-13, 15-24). From these analyses and observations, investigators started to develop hypotheses for the formation of IDIDs. With respect to carboxylate soap deposits, most suggest that this type of IDID could be formed when sodium or calcium contaminants from refinery salt dryers or caustic washes, biodiesel, water ingress, motor oil or sodium nitrite pipeline corrosion inhibitor, reacted with a suitable acid source in situ, forming a sparingly soluble carboxylate salt. Through methods that are capable of identifying specific organic species, e.g. ESI MS, MS/MS and LC/MS, investigators have further characterized components from the deposits. Other techniques have been developed to improve understanding of compositional differences by depth via ToF-SIMS (31, 25).

Many have identified the presence of dodecenyl succinic acid (DDSA) and/or hexadecenyl succinic acid (HDSA), which are common pipeline corrosion inhibitors. Some studies identified these components as the dominant or only organic species present (5, 7, 9, 11, 16, 18, 19, 20), while others observed they were one of several components that might be found in combination with sodium and calcium in the deposit (6, 8, 15, 17, 21). Other species identified included natural mono fatty acids (1, 6, 15, 17), dimer acids (15), polyisobutylene-based succinic acid (PIBSA)(21) and sulfonate (15).

Figure 2. Typical “Carboxylate Soap” Type Deposit Appearance

Inorganic salts, on the other hand, are deduced via SEM/EDS where Na/Ca and chlorine or sulphur/oxygen are the only elements detected in significant quantity (1, 36, 39). In some cases it is possible to see distinct crystals on the metal surface.

On other occasions where no metals were observed and FTIR confirmed the presence of an amide functional group, often also indicated the presence of polyisobutylene (pib) polymer, the deposits are dubbed “lacquer” for their visual appearance or “amide” for the functional marker (1, 8, 11, 20-22). Amide lacquer deposits have been observed in a significant number of affected European injectors (8, 22, 37). Ulmann (1,37) demonstrated that the amide lacquer type were formed from the interaction of polyisobutylene succinimide (PIBSI) type detergents and mono acid lubricity agents in lab tests. Galante-Fox (22) and Quigley (20) have questioned this hypothesis and focused on low molecular weight PIBSI species, that may be formed during in the PIBSI manufacturing process, are the deposit source (also 16, 21, 38).

Figure 3. Typical “Lacquer” Type Deposit Appearance

The root cause of “carbonaceous” deposits is somewhat more difficult to define. These deposits are hypothesized to be the product of thermal degradation of diesel fuel, biodiesel, additives or of existing deposits (1, 13, 14, 37) potentially accelerated by reactive compounds such as the cetane improver 2-ethyl hexyl nitrate (8).

In some cases the deposit contained more than one of the aforementioned features, and thus appeared to be a combination of deposit types. In select other cases, analysis or hypothesis are put forth that do not fit neatly into the above categories, such as the detection of winter operability flow improver in deposits (6).

TESTING AND VALIDATION

While the analysis of deposit can provide interesting and valuable insights into the possible mechanism for formation, it is important to note that the range of IDIDs found are complex in nature and virtually impossible to fully analyze and dissect to such an extent to provide a full mass balance. The presence of metals can be detected by SEM / EDX and FTIR can provide some knowledge of the function groups present. Mass spectrometry has also been used to speciate individual compounds, but it should be noted that only components soluble in typical solvents are extracted and analyzed. A large portion of the deposit that is not readily dissolved, remain unassigned by mass spec.

Furthermore, it is important to differentiate between materials that cause IDID to form and other fuel surfactants that naturally adsorb on to the deposits when it has already formed. The distinction is particularly relevant when discussing the role of additives such as PIBSI (polyisobutylene succinimide) deposit control additives. These components by their nature and function will attract to deposit so separating presence from cause cannot be decided by analysis alone. Thus the analysis of IDID is used to form hypotheses, but these hypotheses must be further validated. To date there is no industry standard test for IDID. As stated earlier, bench and engine tests are the usual next step utilized after forming of hypotheses to support validation.

Ulmann (1) used laboratory bench glassware testing to show an interaction between certain types of PIBSI detergents and monoacid lubricity agent and between sodium and the same monoacid. Other studies (16,20-22) have shown different results , supporting different pathways to these internal deposits.. Painsi (40) evaluated diesel fuel samples with and without additives, varying pressure and thermal, oxidative and shear stresses. Lacey (8) used a bench rig simulator composed essentially of a complete, non-fired fuel injection system.

At the other end of the spectrum are engine test methodologies based on parameters from the field. Arters and Baranescu utilized developmental engines on dynamometer stands that were experiencing IDID to test solutions (10). Wilson (5) and Blizard (7) of Cummins have developed dynamometer tests to both create the problem under controlled conditions and test solutions based on engines and cycles that exhibited problems in the field. Wilson developed a “click tester” that measures the motive force required by the injector solenoid to initiate movement of the afflicted metering valve. This method is particularly compelling in that it directly measures the performance at the point in the injector where it has been determined field problems initiate, and it can be completed in situ without disassembly of the injector. Lubrizol (16, 20, 21) developed a test on a John Deere engine that also exhibits incidence of IDID in the field. This test relies on several confirmatory measurements, including engine power, exhaust temperature profile, motive force of disassembly of the needle and command piston from the injector (post-test) and inspection of the disassembled injector parts. Schwab (12) and Galante-Fox (22) of Afton first successfully adapted the CEC F-98-08 nozzle coking test procedure in the Peugeot DW10B engine to investigate sodium soap type IDID. They rely primarily on exhaust temperature measurement, post-cold start at idle, to identify non-performing injectors. Others have since also utilized the DW10B engine (30, 33, 34) or cycle (28). Ullman (37) used a bespoke cycle and three different engines (PSA, VW, BMW). Performance was monitored but no detriment due to the deposits generated was observed. All of these tests use predominantly high load, steady state cycles to accumulate engine-on hours, and all employ a cold soak period. Maximizing fuel temperature has been noted as an important parameter (16, 20, 30). Under steady state conditions, high load generates the highest internal fuel temperatures and injector tip temperature. Transient high temperatures can also occur when the engine shifts from high to low load. The role of the soak period is not generally considered to be one of reaction or deposit formation, but in creating the viscous deposit ”stickiness” and internal tolerance conditions between injector parts (due to thermal expansion/contraction) to create sticking upon start up. Total test duration to failure can be as little as eight hours in severe deposit-forming conditions (5, 12) and as long as 1000 hours to demonstrate robust no harms (5).

The strongest case for validation based on the literature is that supporting the interaction of sodium or calcium and DDSA and HDSA. There are many controlled rig and engine test procedures that validate the propensity of these constituents to form IDID (5, 7, 8, 12, 16, 20, 22, 28, 30, 33, 34). These engine tests are similar, but do offer some variation. Cummins and Afton pre-mix the sodium and acid, while Lubrizol blends the components separately into the fuel in a small fuel recycle/makeup tank within the engine test cell. Afton has demonstrated the effect using both NaCl and NaOH as the source of sodium (12), Repsol doses both NaOH and NaCl (28) and Infineum and Innospec use a more soluble source of Na (33, 34). While all have demonstrated the effect at a sodium concentration of 3 ppm (and near-stoichiometric levels of DDSA), Blizard (7), Barbour (38) and Chapman (34) have also demonstrated the effect at a lower levels (0.5-1 ppm), noting the rate of deposition appears to scale with concentration. Each has matched deposit analysis with those of field injector deposits.

Alternatively, other engine test results do not support the interaction of mono fatty acids with sodium to form soap type IDID (12, 16, 20-22). Total (30) have recently presented results in agreement that no deposits are formed in B0 dosed with Na and fatty acid, but the addition of a fatty acid to a fouling B7/Na blend did increase the level of deposit.

Less engine data is available for other hypothesized types of Na/Ca soap contributors. Dimer acid (16) and higher molecular weight succinic acid (21) at high concentrations (500 ppm) were found not to form IDID when dosed in fuel with NaOH, while low molecular weight PIBSA (polyisobutylene succinic acid) at high concentrations (on the order of 25% of a 500 ppm PIBSA dose) were found to form deposits (22). The addition of calcium sulfonates to fuel, whether alone or in combination with DDSA, also failed to generate sulfonate or succinic acid deposits (16). Certain antioxidants commonly used in biodiesel were shown to significantly increase the level of deposit in a fouling European B7/multifunctional diesel fuel additive (30).

The testing of amide lacquer is progressing but is still at less of a consensus position. Arters (16) ran several engine tests to evaluate the hypothesis of Ulmann (1), dosing the fuel with a PIBSI that matched the criteria for reactivity with mono fatty acid and dimer acid, and even pre-reacting the PIBSI and mono acid at elevated temperatures for extended periods, but was unable to form any deposits. Arters also first investigated the potential of low molecular weight PIBSI to form deposits based on the observation that such low molecular weight species were noticeable in deposit analysis of tests using effective deposit control additives, and would explain the presence of pib in such deposits. However, that testing, incorporating low molecular weight deposit control additive into the high molecular weight parent deposit control additive, did not produce deposits, indicating that an effective deposit control additive (DCA) would negate the impact of potentially harmful low molecular weight species. In follow up testing, a neat C12-16 molecular weight PIBSI was tested in combination with mono fatty acid and did not form deposits. However, Galante-Fox (22) reported that very high treat rates (112 ppm) of pure low molecular weight (<350 MW) PISBI can rapidly cause injector sticking, producing deposits consistent with those from the field with respect to SEM and EDX analysis. Barbour (38) followed up again and evaluated the structural characteristics of different low MW PIBSIs in engine tests, characterized as varying the number of amine groups and type of pendant amine, and concluded that the head group amine was a critical parameter in the formation of deposits. Depending on the head group, results ranged from no harm to causation to prevention of deposits. The presence of a monoacidic lubricity component was found to have no negative impact on the results. In contrast, Ullmann (37) revisited the laboratory findings of their original paper (1) that PIB succinimide deposit control additives interacting with carboxylic acids, such as mono-acidic based lubricity improvers, are the cause of lacquer type internal deposits, presenting results in lab glassware, bench rig and fired engine tests. Deposit testing was carried out on different PSA, VW and BMW engines. Two different PIBSI samples were used in combination with mono-acidic lubricity improver at 10-fold overdose treat rates. In the fired engines, one PIBSI gave little or no deposit while the other PIBSI showed significant levels of internal deposits, allowing differentiation of PIBSI effects. Both PIBSIs reacted with monoacid in glassware experiments, and thus it was concluded that chemical reactivity is not a sufficient predictor of deposit formation in engines. Intriguingly, Arondel (30) developed data indicating that multifaceted interactions play a key role in the amount of IDID formed, based on a visual rating system of the injector needle tip and cylinder deposits generated in a specially designed DW10B engine test. They examined combinations of different FAMEs, acidic lubricity additive, “detergents” and multifunctional additive packages that may contain PIBSI, cetane improver and antioxidants. While significant progress has been made, there is still a lack of consensus on the particulars and real-world scenarios of amide lacquer formation.

Whether discussing the soap or lacquer types of deposits, it is clear that very specific head-tail chemical compositions are required to achieve the precise balance of polarity for surfaces and (in)solubility at the conditions of injector operation to cause deposits to form. Galante-Fox (35) has proposed a solubility model to explain the results while Lacey (29) has described micellular interactions as a key contributor. Painsi (40) indicated that while thermal and oxidative stresses influence deposit formation, pressure and shear stresses do not. While some success has been achieved in identifying relatively simple explanations, there is evidence that the problem may be more complex. The work of Arondel (30) indicates that complex, multicomponent interactions may play a role and simple one- and two-component models may be insufficient to describe IDID effects fully.

Of course, investigations were also looking for deposit control solutions. Most evaluations tested DCA on top of a sodium/DDSA-dosed fuel to establish deposit control performance (“keep clean”) compared to that of a test run without the DCA. In other variations, the DCA is tested in a sodium/DDSA-dosed fuel after deposits are built up to demonstrate reductions in deposits (“clean up”). While some DCAs were found to be ineffective (21, 29, 30, 32, 36), others cited demonstrations of effective deposit control additives in controlled engine tests (5, 7, 12, 17, 21, 23, 30, 32, 33, 34) and/or in the field (4, 5, 7, 10, 32, 39).

Appendix 1 summarizes the theories of IDID cause and tabulates the supporting (and disputing) evidence based on deposit and fuel analysis, bench and engine testing and field experience.

CONCLUSIONS (IDID CAUSES)

1. A preponderance of industry data indicates that salts of Na or Ca with dodecenyl succinic acid (DDSA) and hexadecenyl succinic acid (HDSA) are highly likely to contribute to “soap” type IDID.
2. Other proposed combinations with Na/Ca, such as alternative succinic acids, fatty acids or dimerized acids, have contradictory data as of the time of this report and no consensus conclusion can be reached.
3. Investigations on sources of “amide lacquer” type deposits with confirmatory engine test data has focused on interactions of PIBSI with fatty acids, and the impact of low molecular weight PIBSI as deposit sources , but with less clear consensus than the Na soap deposits.
4. IDID is caused by very specific chemical head-tail combinations resulting in a particular balance of polarity and solubility at the conditions of injector operation.
5. A few simple combinations are unlikely to explain all sources of IDID.

REFERENCES

1. "Effects of Fuel Impurities and Additive Interactions on the Formation of Internal Diesel Injector Deposits," Jorg Ulmann, Marion Geduldig, Heinz Stutzenberger, Ronaldo Caprotti, Dennis Hess; TAE 7th International Colloquium Fuels, Esslingen, 14-Jan-2009
2. "HPCR Fuel System Background & ULSD Deposit Issues," Charles Klose; SAE Diesel Deposit Technical Workshop, 2-Nov-2009.
3. "HPCR Deposit Issues," Norman Blizard, SAE Diesel Deposit Technical Workshop, 2-Nov-2009.
4. "Off-Road Diesel Fuel System Deposits - Field Experience," Brian Schmidt, SAE FFL220: Internal Deposits in High Pressure Diesel Injection Systems, 26-Oct-2010.
5. "Injector Deposits in High Load Off-Road Applications," Steve Wilson, SAE FFL220: Internal Deposits in High Pressure Diesel Injection Systems, 26-Oct-2010.
6. "Injector Deposit," Akio Tanaka, Satoshi Bunne, Toshihiko Omori, Koichi Yamada, JSAE TWS-3 Injector Deposits Workshop, 31-Aug-2011.
7. "Continuing Studies of Injector Deposit Formation and Field Operational Problems," Norman Blizard, JSAE TWS-3 Injector Deposits Workshop, 31-Aug-2011.
8. "Internal Fuel Injector Deposits," Paul Lacey, Sandro Gail, Jean Marc Kientz, Nebojsa Milovanovch, Christian Gris, SAE 2011-01-1925, 2011.
9. "Cummins L10 Depositing Test to Evaluate Diesel Fuel Quality," Gallant, Cusano, Gray and Strete, SAE 912331, 1991.
10. "Investigations into Internal Diesel Injector Deposits (IDID)," David Arters and Rodica Baranescu, SAE Diesel Deposit Technical Workshop, 2-Nov-2009.
11. "Impact of ULSD and Fuel Additives on HPCR Fuel Systems: Injector Deposits and Other Problems", Rinaldo Caprotti, SAE Diesel Deposit Technical Workshop, 2-Nov-2009.
12. "Internal Injector Deposits in High-Pressure Common Rail Engines," Scott Schwab, Josh Bennett, Steven Dell, Alex Kulinowski, Julie Galante-Fox, Keith Miller, SAE 2010-01-2242, 2010.
13. "Insights into Deposit Formation in High Pressure Diesel Fuel Injection Equipment," Jim Barker, G John Langley, Paul Richards, SAE 2010-01-2243, 2010.
14. "Temperature Programmed Oxidation as a Technique for Understanding Diesel Fuel System Deposits," Jim Barker, B Cheeseman, D Pinch, Paul Richards, SAE 2010-01-1475, 2010.
15. "Deposit Control in Modern Diesel Fuel Injection Systems," Rinaldo Caprotti, Nadia Bhatti, Graham Balfour, SAE 2010-01-2250, 2010.
16. "Investigations into Internal Diesel Injector Deposits," David Arters, SAE FFL220: Internal Deposits in High Pressure Diesel Injection Systems, 26-Oct-2010.
17. "Sticking Deposits in HPCR Injectors," Scott Schwab, Josh Bennett, Steven Dell, Alex Kulinowski, Julie Galante-Fox, Keith Miller, SAE FFL220: Internal Deposits in High Pressure Diesel Injection Systems, 26-Oct-2010.
18. "Internal Deposits in High Pressure Diesel Injection Systems", Tom Livingston, SAE FFL220: Internal Deposits in High Pressure Diesel Injection Systems, 26-Oct-2010.
19. "SAE 2010 Panel on Injector Deposits," Rinaldo Caprotti, SAE FFL220: Internal Deposits in High Pressure Diesel Injection Systems, 26-Oct-2010.
20. "A Study of the Internal Diesel Injector Deposit Phenomenon," Robert Quigley, Robert Barbour, Emma Fahey, David Arters, William Wetsel, James Ray, TAE 8th International Colloquium Fuels, Esslingen, 19-Jan-2011.
21. "Internal Diesel Injector Deposits," David Arters, JSAE TWS-3 Injector Deposits Workshop, 31-Aug-2011.
22. "Diesel Injector Internal Deposits: Causes, Effects and Resolution," Julie Galante-Fox, JSAE TWS-3 Injector Deposits Workshop, 31-Aug-2011.
23. "Internal injector Deposits 2011 Update," Rinaldo Caprotti, JSAE TWS-3 Injector Deposits Workshop, 31-Aug-2011.

24. “High Pressure Common Rail Injector & Filter Deposits,” Richard Chapman, JSAE TWS-3 Injector Deposits Workshop, 31-Aug-2011.
25. “Diesel Deposits,” Jim Barker, Colin Snape, David Scurr, TAE 9th International Colloquium Fuels, Esslingen, 15-17 Jan-2013.
26. -“Internal Injector Deposits (IID),” Paul Lacey, CRC Deposit Panel Meeting, 31-Jan-2012.
27. “Diesel Injector Deposit Update,” Shawn D. Whitacre, CRC Deposit Panel Meeting, 31-Jan-2012.
28. “Internal Diesel Injector Deposits: Sodium Carboxylates of C12 Succinic Acids and C16 and C18 Fatty Acids,” Maria Dolores Cardenas Almena, Octavio Lucio Esperilla, Francisco Martin Manzanero, Yolanda Murillo Duarte, Luis Carlos Quinitero Toscano, Guillermo Wolff, SAE 2012-01-1689, 2012.
29. “Fuel Quality and Diesel Injector Deposits,” Paul Lacey, Sandro Gail, Jean Marc Kientz, Geoffroy Benoist, Peter Downes, Christian Daveau, SAE 2012-01-1693, 2012.
30. “Fuel Additives for Reduction of Internal Diesel Injectors Deposits (IDID, “lacquering”): A Critical and Priority Route,” Mathieu Arondel, Helene Rodeschini, Manuel Lopes, Bernard Dequenne, SAE 2012-01-1687, 2012.
31. “A Novel Technique for Investigating the Characteristics and History of Deposits Formed Within High Pressure Fuel Injection Equipment,” Jim Barker, Colin Snape, David Scurr, SAE 2012-01-1685, 2012.
32. “Internal Diesel Injector Deposits – Resolving a Sticky Problem,” David Arters, SAE FFL900: The Impact of ULSD, Biodiesel Blends and Fuel Additives on Fuel Systems: Injector Deposits and Other Problems, 18-Sept-2012.
33. “Internal Injector Deposits Update,” Rinaldo Caprotti, SAE FFL900: The Impact of ULSD, Biodiesel Blends and Fuel Additives on Fuel Systems: Injector Deposits and Other Problems, 18-Sept-2012.
34. “Diesel Injector Deposit Studies – Effect of Fuel-borne Contaminants,” Rick Chapman, SAE FFL900: The Impact of ULSD, Biodiesel Blends and Fuel Additives on Fuel Systems: Injector Deposits and Other Problems, 18-Sept-2012.
35. “Insights into the Mechanism of Diesel Internal Injector Deposit Formation,” Julie Galante-Fox, SAE FFL900: The Impact of ULSD, Biodiesel Blends and Fuel Additives on Fuel Systems: Injector Deposits and Other Problems, 18-Sept-2012.
36. “IDID Overview,” Paul Lacey, Sandro Gail, Christian Daveau, SAE FFL900: The Impact of ULSD, Biodiesel Blends and Fuel Additives on Fuel Systems: Injector Deposits and Other Problems, 18-Sept-2012.
37. “Internal Diesel Injector Deposit Formation – Reproduction in Laboratory, System Bench and Engine Tests,” Jorg Ullmann, Heinz Stutzenberger, TAE 9th International Colloquium Fuels, Esslingen, 15-17 Jan-2013.
38. “Finding a Solution to Internal Diesel Injector Deposits,” Robert Barbour, Robert Quigley, Avtar Panesar, James Payne, David Arters, Jim Bush Andrew Stevens, TAE 9th International Colloquium Fuels, Esslingen, 15-17 Jan-2013.
39. “Understanding the Spectrum of Diesel Injector Deposits,” Robert Quigley, Robert Barbour, David Arters, Jim Bush, TAE 9th International Colloquium Fuels, Esslingen, 15-17 Jan-2013.
40. “Understanding Internal Diesel Injector Deposits: New Insights from Laboratory Experiments,” Monika Painsi, Alexander Grafl, TAE 9th International Colloquium Fuels, Esslingen, 15-17 Jan-2013.

Appendix 1. Reference Data Summary

Mechanism	Deposit Analysis - Identified	Bench Testing - Support	Engine Tested - Support	Field Test - Support	Engine Tested – Contraindications
Na/Ca-derived					
• Dodecenyl/hexadecenyl succinic acids	5, 6, 7, 8, 12, 13, 16, 17, 20, 21, 22, 26, 27, 28, 29, 30, 31, 32, 33, 36	26, 29	5, 7, 8, 12, 16, 20, 22, 28, 30, 33, 34	32	-
• Mono fatty acid (additive)	1, 6, 11, 13, 15, 16, 18, 19, 20, 23, 28, 30, 32	1, 11, 15, 18, 37	30, 37	-	12, 16, 20, 21, 22, 28, 30, 32, 38
• Mono fatty acid (FAME)		-	30	-	-
• Low MW PIB succinic acid	22	-	22	-	-
• High MW PIB succinic acid	-	-	-	-	21, 32
• Sulfonate	16, 21, 32	-	-	-	21, 32
• Sulfate/chloride	1, 26, 29, 32, 36	-	39	-	-
• Dimerized fatty acids	1	-	-	-	21, 32
“Lacquer” PIB succinimide derived					
• High MW PIBSI + fatty acid	1, 11, 18	1, 37	37	-	21, 32, 38
• High MW PIBSI + dimer acid	1, 11, 18	1	-	-	21, 32
• Mixed low/high MW PIBSI	16	-	-	-	16, 32
• Low MW PIBSI	22	-	22, 32, 38	-	-
• Unspecified PIBSI + carboxylic acid	-	-	30	-	-
Other					
• Antioxidants (in B7)	-	-	30	-	-
• Cetane improver (2-ethyl hexyl nitrate)	-	26, 29	-	-	30, 32