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MULTI-CYLINDER ENGINE TESTING OF FUELS CONTAINING METHYLCYCLOPENTADIENYL MANGANESE TRICARBONYL (MMT)

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ABSTRACT

Methylcyclopentadienyl Manganese Tricarbonyl (MMT) was once commonly used in gasoline fuel as an alternative to Tetraethyl Lead (TEL) and, in the late 1970's and early 1980's, was widely promoted as octane booster. However, due to increasing awareness of the public heath concerns relating to use of metallic additives in road transport fuel its use has been steadily reducing. Today MMT only appears in certain (mostly developing) market fuels, namely Africa, Pakistan and some parts of Asia and South America and there is very little recently published material relating to the operational effects of MMT in modern engines in the literature. It is known however that these effects can be significant. In the USA, for example, MMT usage was completely phased out not only due to the aforementioned health concerns but also due to operational problems with deposits and contamination/poisoning of engine after-treatment, namely oxygen sensors and catalysts. MMT containing fuels are also thought to promote low speed pre-ignition (LSPI) more readily than fuels that do not contain MMT – although there are additional factors that must be considered, for example the underlying fuel quality in the markets of interest. This paper presents the results of experiments to characterise the behaviour of fuels known to promote LSPI, with the addition of MMT in quantities based on dedicated in-market sampling. The frequency and magnitude of LSPI events of fuels containing MMT was found to be significant and led to engine damage as a result of mechanical stress and significant increases in fuel-in-oil dilution.

Keywords: MMT, LSPI, Abnormal combustion. Fuel additives, Alkaline metal oxides

NOMENCLATURE

Roman letters

MMT	Methylcyclopentadienyl Manganese Tricarbonyl
LSPI	Low Speed Pre-Ignition
SI	Spark Ignited
CAF	Coryton Advanced Fuels
DI	Direct Injection
TC	Turbo Charged
TLC	Top Land Crevice
RON	Research Octane Number
AKI	Anti-Knock Index
PMAX	Maximum Cylinder Pressure
KV100	Kinematic Viscosity [cST] at 100 °C
MFB02	2% Mass Fraction Burnt
FiO	Fuel in Oil Dilution
ICP	Inductively Coupled Plasma

Greek letters

σ	Standard Deviation

1. INTRODUCTION

The internal combustion engine remains the most widely used form of propulsion in on-highway vehicles. Spark ignited (SI) engines currently dominate the new vehicle market due to their affordability and compatibility with hybridisation. Here, vehicle manufacturers typically favour down-sized and pressure charged engines as they provide high power density whilst simultaneously meeting emissions targets and delivering CO_2 reductions. One barrier that remains to further increases in performance and reliability from these engines is abnormal combustion, the causes of which have been extensively investigated by researchers in recent years [1–4]. The main focus of these earlier works was on the influence of oil detergent additives, with some observations that certain fuel properties or additive compounds could promote LSPI. Concurrently, vehicle owners had observed engine failures in several African markets, most notably Senegal. Examination of failed engine parts from these vehicles showed signs of abnormal combustion and damage patterns similar to those seen in Fig. 1, strongly suggesting that the failures were due to LSPI and mega-knock. Subsequent analysis of Senegalese fuels highlighted high concentrations of Methylcyclopentadienyl Manganese Tricarbonyl (MMT), which is also

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found in fuels across most of Africa, as well as some countries in South America and Asia – with Pakistan having the highest recorded concentrations at 140 ppm, Fig. 2 [5]. The current work seeks to explore the effect of the addition of MMT to fuels with properties known to provoke LSPI in order to determine if the addition of metal oxides has a promoting effect similar to that of oil detergents.



(a) Piston second land damage



(b) Spark plug insulator, centre and ground electrode damage

FIGURE 1: Typical LSPI and Mega Knock Damage

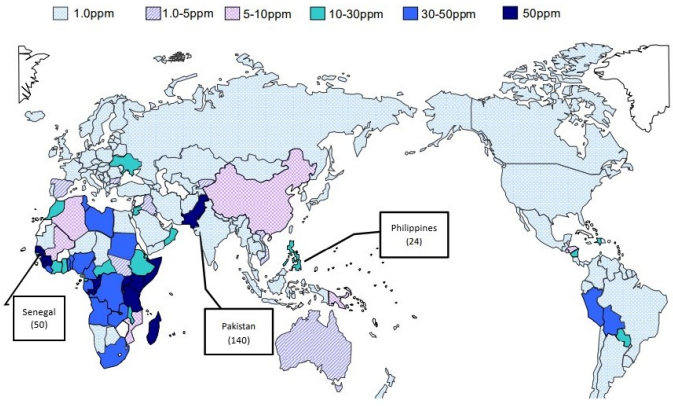

FIGURE 2: Fuel Survey Data, Manganese Concentration by Geographical Location, numbers in brackets indicate the maximum values recorded in the indicated markets - Adapted From [5]

Potential Hypotheses

A link between fuel quality, MMT concentration , and LSPI has been proposed by Nomura et al [6]. Two mechanisms are proposed;

- The first is related to the fact that fuels using MMT as an octane booster have low base volatility as they tend to be in lower fuel quality.
- The second is that manganese oxide in scattered droplets supplies the ignition energy to ignite the droplet, which is a mixture of fuel and lubricating oil. Within this hypothesis, two chemical ignition mechanisms are proposed related to exothermic heat release from further oxidation of manganese oxides.

Little test data is available in the public domain from modern engines running on fuels containing MMT. Ths paper seeks to address this gap by reporting on an experiments designed to evaluate the effect of MMT addition to specially selected fuels on the performance of a multi-cylinder engine in combination with clean and used lubricating oils.

TABLE 1: Engine Specification

Combustion System	Spray Guided, Direct Injection
Valve-train	Double Over-head Cam, Variable Int & Exh Phasing, Continuously Variable timing and Lift
Aspiration	Turbo-charged
Cyl Displacement [cm^3]	499.2
Compression Ratio	10.5:1
Bore [mm]	83.00
Stroke [mm]	92.00
Oil Type	0W 20

2. MATERIALS AND METHODS

A high specification multi-cylinder engine test bed with an AVL Gigabit Indimaster combustion analyser and the ability to run barrelled fuels was used to undertake the engine testing. The engine was a production 4-Cylinder gasoline engine and the calibration was practically as per the production release, with the exception of minor modifications to extend the knock control retard authority (determined by the first 'aggressive' test fuel). The engine specification can be found it Table 1.

Properties of MMT

Table 2 presents some the chemical and physical properties of MMT. Of note is the boiling point, when compared to the fuel distillation curves in Figure 4 it can be seen that this corresponds with the final boiling point of the 'heaviest back-end' distillation fuel at 235.5 °C. This could mean that MMT is likely to accumulate with fuel 'heavy-ends' in the Top Land Crevice (TLC) volume noted in previous works as a potential source for the droplet triggered LSPI mechanism [7].

Fuels Matrix

Table 3 shows the test fuel matrix used to evaluate the effect MMT on LSPI promoting fuels. A 95 RON E10 (10% ethanol)

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TABLE 2: Chemical Properties of MMT

Methylcyclopentadienyl Manganese Tricarbonyl	
Chemical Formula	$C_9H_7MnO_3$
Molar Mass (g/mol)	218.09
Appearance	Pale yellow to dark orange
Odour	Faint, pleasant
Melting Point (°C / °F / K)	-1 / 30 / 272
Boiling Point (°C / °F / K)	232–233 / 450–451 / 505–506
Solubility in Water (ppm)	Low
Solubility	Hydrocarbons (gasoline), ether, alcohol
Vapour Pressure (mmHg at 100 °C)	7

reference fuel, based on EN228, was used for general engine combustion system and calibration. A high octane (102 RON) E0 (0% ethanol) reference fuel was used to characterise the baseline LSPI level and to periodically assess the health of the engine between the LSPI testing. For proprietary reasons, the Author's are unable to publish distillation curves or further compositional data on this fuel and hence no results are presented for this fuel. Varying concentrations of MMT were observed in fuel samples collected in Senegal, 30 ppm was taken as representative of an average forecourt fuel. With reference to Fig. 2, this concentration doesn't represent the highest levels of MMT recorded in Senegal, but was nevertheless deemed acceptable to allow for fair characterisation of abnormal combustion. In addition to in-market fuel sampling in Africa, this study also considers a representative fuel of 'poor' quality from the Chinese market which is known to promote LSPI. This fuel is characterised by low RON ($\leq$ 92), elevated low, mid and back-end evaporation temperatures, and a high aromatic content.

TABLE 3: Test Fuel Matrix

Name	MMT (ppm)	Description
95RON E10	0	EN228-based reference fuel, 10% ethanol
102RON E0	0	High-RON reference fuel, 0% ethanol
China LSPI	0	CAF Chinese market representative fuel
High LSPI	0	CAF LSPI-promoting reference fuel for LSPI testing
Africa LSPI MMT	30	CAF synthesized fuel based on Senegal market sampling
High LSPI MMT	55	CAF High-LSPI fuel with added MMT
China LSPI MMT	55	CAF China LSPI-promoting fuel with added MMT

Coryton Advanced Fuels (CAF) provide a number of LSPI promoting fuels, synthesised based on their detailed knowledge of fuels, market sampling, and referred to as 'China LSPI' and 'High LSPI'. China LSPI, is a CAF reference fuel based on in-market sampling which represents worst case characteristics for LSPI testing. It has the highest final boiling point of all of the fuels at 200 °C. A version of this fuel, based on worst case in-market sampling (55 ppm MMT), was included to understand the difference made to a fuel known to excite LSPI ('China LSPI MMT'). High LSPI is a 96 RON reference fuel created by CAF to promote LSPI, as a result of it's high mid and back-end distillation characteristics. Similarly 55 ppm MMT was also added to LSPI High fuel to make a comparable fuel at the equivalent MMT treat rate as the China LSPI MMT fuel.

Octane Rating

While it is known that octane number itself does not increase the propensity for LSPI, it is known to impact the severity of mega-knock if it occurs [8] [9]. Figure 3, details the variation of RON, MON and AKI for the fuels in the test matrix.

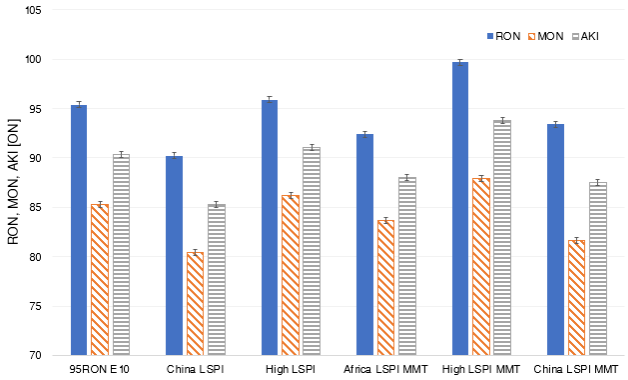

FIGURE 3: Octane Rating – RON ASTM D2699, MON ASTM D2700, uncertainty for the same test laboratory ± 0.3 RON, MON and AKI.

The reference fuel and the High LSPI fuel had similar RON (95 - 96), MON (85 - 86) and hence AKI (90 - 91) values. With the addition of MMT the High LSPI fuel the RON was increased to 99.7. The Africa LSPI MMT had a relatively low RON (92), MON (84) and AKI (88). China LSPI had the lowest RON, MON and AKI at (90, 80 and 85) respectively. The addition of MMT had the effect of increasing ON by approximately 2 - 3 RON.

Evaporation Behaviour

It has been demonstrated that the evaporation behaviour of a fuel has a strong link to the LSPI tendency, [10]. Fuels most likely to excite LSPI tend to have higher mid- and back-end evaporation temperatures. The worst fuels also tend to have high final boiling points 200 - 220 °C. As maximum engine sump temperatures don't generally exceed 150 °C, and are more typically between 90 - 120°C, as much as 25% of the mid and heavier ends of the fuel could be present in the lubricating oil depending on operating conditions [11]. As shown in Fig. 4 the evaporation temperatures of the market and market + MMT fuels are high throughout the curve, something that is generally indicative of poor base fuel

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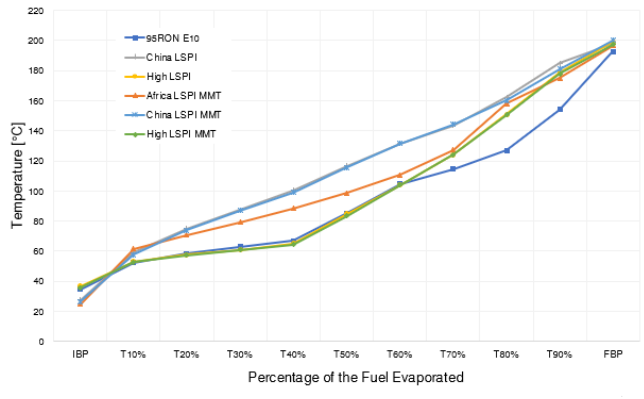

FIGURE 4: Fuel evaporation curves, ASTM D86, uncertainty for the same test laboratory, at T50% ± 2 °C.

quality. The high LSPI fuel, having been deliberately designed by CAF is consistent with an EN228 or typical European fuel until T65%, thereafter it has been chemically engineered to behave like a market fuel with a high back-end evaporation temperature.

Fuels Test Sequence

The test sequence is shown in Table 4. To evaluate the effect of used oil in addition to MMT, tests were conduced with new (clean) and used oil. Following the initial engine performance testing and baseline to confirm that the installation was successful, testing then moved to the 'safe' 102 RON fuel. The engines 'health' was regularly assessed using the 102 RON fuel to ensure that the affects of accumulated damage were not impacting the test results. An additional objective of running periodic reference tests, was to utilise the potential cleaning effect via removal of deposits, with the objective of returning the combustion chamber surfaces to their original condition.

3. ENGINE HARDWARE AND LUBRICANTS

The following section details hardware considerations also evaluated in the engine testing.

Engine Hardware

In addition to the hypotheses proposed in the introduction, a question was raised as to whether MMT might in fact trigger LSPI by damaging or inhibiting the correct operation of engine components such as injectors, spark plugs or lambda sensors as opposed to the particle or metallic catalytic pathways suggested previously. Accordingly, in order to fairly consider this potentially cumulative effect all injectors, spark plugs and lambda sensors were replaced each time the LSPI promoting fuel type was changed.

Lubricants

A 140 kg of used 0W 20 oil was collected from vehicles, consolidated into a single batch, and chemically analysed. Table 5 shows an analytical comparison of new vs used oil. The KV100 kinematic viscosity was reduced by 1 cSt from new to used oil. Inductively Coupled Plasma (ICP) analysis found that there were increases in typical wear metals for an ICE in terms of aluminium, copper and iron in the used oil. Of note was the 59

TABLE 4: Fuel Test Sequence

Name	Oil Type	Description
102RON E0	Clean Oil	Baseline
102RON E0	Clean Oil	Baseline (repeat)
95RON E10	Clean Oil	Baseline
95RON E10	Used Oil	Baseline
China LSPI	Clean Oil	
China LSPI	Used Oil	
102RON E0	Clean Oil	Re-baseline and health check
China LSPI MMT	Clean Oil	
High LSPI	Clean Oil	
High LSPI	Used Oil	
102RON E0	Clean Oil	Re-baseline and health check
High LSPI MMT	Clean Oil	
102RON E0	Clean Oil	Re-baseline and health check
Africa LSPI	Clean Oil	
Africa LSPI	Used Oil	
Africa LSPI MMT	Clean Oil	
102RON E0	Clean Oil	Re-baseline and health check

ppm manganese present in the used samples. UK fuel is blended to EN228, and as such there should be no manganese present. The engine manufacturer was consulted to identify the source of manganese and it was found to be present in the crankshaft and timing chain material, hence in the oil due to component wear. A small reduction of calcium was observed (1801 - 1781 ppm), indicating the detergent levels are still high and able to counter acid formation. Minor oxidation and some nitration had occurred, but this is typical for used oil. Overall, the consolidated mixture of used oil was determined to be in good condition and suitable for testing.

4. LSPI TEST AND CLASSIFICATION

The need to investigate LSPI in a controlled and time efficient manner has led to the development of dedicated test cycles. Well known examples in the literature include; the Toyota LSPI test [12] and the GM Dexos test [13]. Each test has its own advantages and disadvantages, for example the Dexos test is the most transient. In contrast the Toyota test is steady state and spends most of the test at peak torque. The current work uses a new test cycle, shown in Fig. 5, specifically designed to evaluate both LSPI mechanisms (oil droplet and deposit initiated LSPI) in a single test.

No consensus exists regarding LSPI classification criteria to date in industry or academia, however there are common themes between those in the literature. When making a relative comparison of factors influencing LSPI behaviour, a set of deterministic criteria must be applied. However a level of manual inspection is

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TABLE 5: Comparison of new vs. used SAE 0W 20 oil. KV (ASTM D445) uncertainty ± 3%. Elemental analysis ICP (ASTM D5185) uncertainty ± 10%. FiO GC MS (ASTM D2887) uncertainty ± 5%. Nitration: new oil (ASTM D5762), used oil (ASTM D5763). Oxidation: new oil (ASTM D7528), used oil (ASTM D7414). Overall uncertainty for FTIR MS ± 10%.

Description	Unit	New Oil	Used Oil
Kinematic Viscosity at 100°C	cSt	7.6	6.6
Kinematic Viscosity at 40°C	cSt	34.1	29.1
Viscosity Index		199	195
Aluminium	ppm	<1	6
Iron	ppm	<1	50
Copper	ppm	<1	24
Manganese	ppm	<1	56
Calcium	ppm	1801	1781
Magnesium	ppm	<1	6
Silicon	ppm	3	17
Fuel Dilution	%	0	3.75
Nitration (1630 FTIR Peak)	Abs/cm	0	13
Oxidation (1720 FTIR Peak)	Abs/cm	0	4

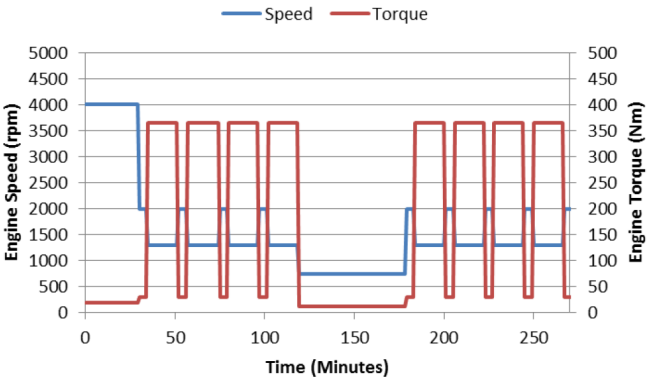

FIGURE 5: LSPI Test cycle

also recommended to minimise the likelihood of 'miss detection' of LSPI events. If differing classification criteria were applied to the same data it is reasonable to expect that the absolute number of LSPI 'events' identified could be different. Where a test generates large numbers of LSPI events a small difference, due to differing classification methods, is not critical to the overall outcome of the experiment. In the case where the results of two tests, with different noise factors, generated the results: 0 and 1 events, this would not be valid as it would not be possible to interpret the results with sufficient statistical confidence. Accordingly, where possible, four repeat tests were conducted for each fuel with the number of LSPI events per fuel reported in this work being the average value across all four tests. Note however, that in some cases, where the LSPI observed was so severe it was not possible to run four repeat tests, either the engine or the in-cylinder pressure sensors were damaged. These cases will be specifically identified in the text.

The classification method chosen for the current work was a modified version of that defined by Leach *et al.* [10], shown graphically in Fig. 6. Note that two of Leach's three criteria for classifying an LSPI event (the MFB2 and PMAX criteria) are based on a 3σ difference to a moving average (of the 100 preceding cycles). However, high levels of variability were experienced with the fuels tested in this work due to the extremely advanced combustion phasing resulting in significant LSPI mis-detection when applying Leach's original criteria. As a result, Leach's 3σ LSPI 'trigger' criteria were relaxed to 4σ for this study.

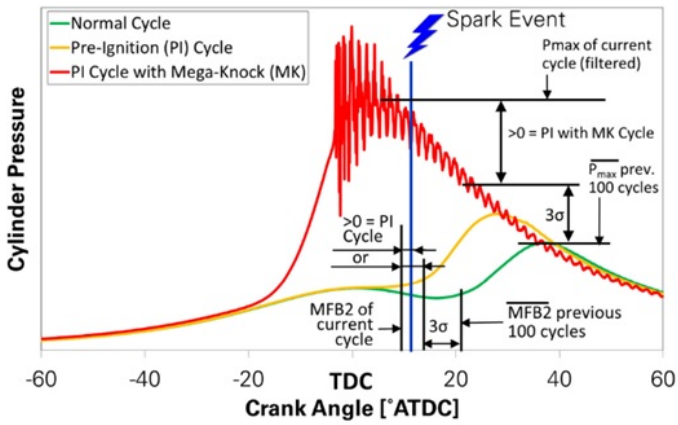

FIGURE 6: Abnormal combustion triggers and LSPI classification [10]

Interpretation of the LSPI Result Plots

The combustion analyser was operated in a continuous buffer mode whilst running the LSPI test. Any cycles that exceeded some or all of the modified Leach 'trigger' criteria, shown below, were automatically identified and saved for later post-processing by a bespoke MATLAB routine.

- Trigger if; $\theta_{MFB2i} < [\sum_{i=-101}^{-1} \frac{MFB2i}{100} - \mathbf{4} \cdot \sigma MFB2i]$
- Trigger if; $\theta_{MFB2i} < \theta_{ign}$
- Trigger if; $\theta_{PMAXi} < [\sum_{i=-101}^{-1} \frac{PMAXi}{100} - \mathbf{4} \cdot \sigma PMAXi]$

The MATLAB post-processing script loads the raw Indimaster .dat file (also know as Ifiles), plots the cylinder pressure from 30 °CA BTDC – 90 °CA ATDC. Filters are then applied to identify which trigger(s) caused the file to be logged. Figure 8 shows an example plot where the post-processing has been applied. The key at the top of the chart indicate which triggers and which cylinder or cylinders have triggered. The least reliable trigger was the location of MFB2. This is notoriously difficult parameter to determine, during heat release analysis, due to the very shallow gradient change at the start of combustion. It can be seen in the examples included in this paper that the location of MFB2 is after the spark despite it being a clear LSPI cycle on manual inspection. Hence the use of all triggers was necessary

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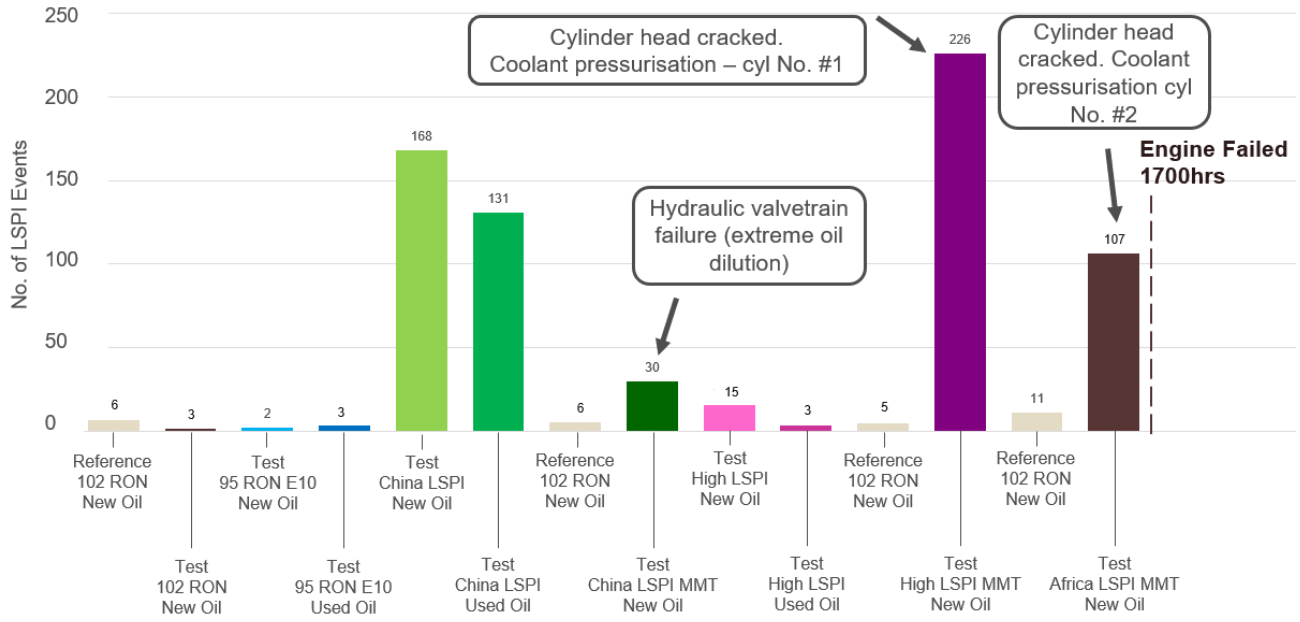

FIGURE 7: Test summary – Number of LSPI events per test (average of 4 tests) using fuel from the fuels matrix

to capture as many cycles as possible without significant quantities of mis-detection. In the example shown in Figure 8, it can be seen that cylinder 2 (black) has experienced pre-ignition and mega-knock. Examining the plot from left to right: the location of MFB2 is just before the ignition timing, given there is a finite ignition delay even for PI cycles, this infers that PI has occurred. The trigger identified by the script was PMAX >120 bar. The start of the pressure rise is inside of the MFB2 moving average window, again further indication of PI. Looking at the cylinders with typical pressure traces, it can be seen that the combustion is retarded (PMAX is approx. 40 °CA ATDC, despite the ignition timing being 7 °CA ATDC).

5. RESULTS

A general summary of all of the engine testing results can be found in Figure 7. Unless stated, the presented results represent the average values from 4 realisations of the 270 minute test cycle. The first reference test was 102 RON fuel and clean oil, and found to have an average of 6 LSPI events. A second group of test using the same fuel resulted in an average of just over 1 event, highlighting that even with the same fuel there can be test-to-test variability.

The fuel was changed to 95 RON E10 and tests conducted firstly with new oil (2 events), then followed by used oil (3 events). Whilst the test with used oil is on average 1 event higher, this thought not significant as the absolute number of events are low. Given the test cycle is designed to provoke LSPI behaviour, the number of events experienced using these fuels was deemed acceptable to proceed with testing of the more extreme fuels.

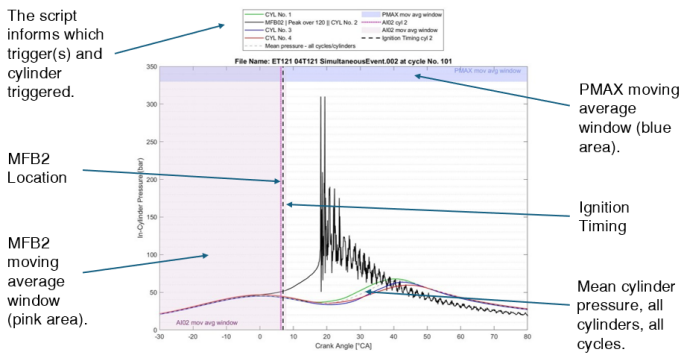

FIGURE 8: Result plot and triggers explanation. X-axis is crank angle, Y-axis is cylinder pressure. The significant of the lines and shaded areas are explained in the captions.

China LSPI Fuel

China LSPI fuel, in combination with clean oil, produced very high levels of LSPI with 168 events observed, Fig. 7. The absence of repeated or run-away events points to this being most likely attributed to the oil accumulation and release mechanism [7]. The following test with China LSPI fuel and used oil yielded 131 events, Fig. 7, an unexpected result. Based on the understanding reported in the literature, the addition of used oil was expected to have increased the number of events . This fuel had a particularly low RON (90.2) in contrast to the 102 RON reference fuel, resulting in very late combustion phasing. The effect on the calibration, was very active knock control, increased exhaust temperature resulting from retarded ignition, which in-turn required increased fuel enrichment. An example LSPI cycles of particular interest is shown in Figure 9. Cylinder 2 (black) triggered on both MFB02 and peak pressure >120 bar. This was a pre-ignition cycle as MFB02 was 12 °CA before the ignition timing and the cylinder pressure was rising significantly before the spark timing. The rate of pressure increase was relatively smooth until approx. 8°ATDC whereafter the rate of pressure rise was extreme, leading to aggressive mega knock by 10°CA ATDC.

The mean peak pressure of the normal combustion cylinders was 60 - 70 bar, whereas the mean peak of the abnormal cylinder

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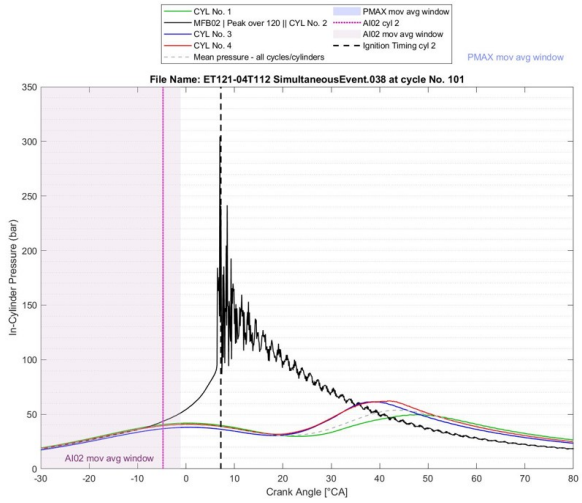

FIGURE 9: China LSPI fuel, clean oil, example of pre-ignition event captured from the continuous log file.

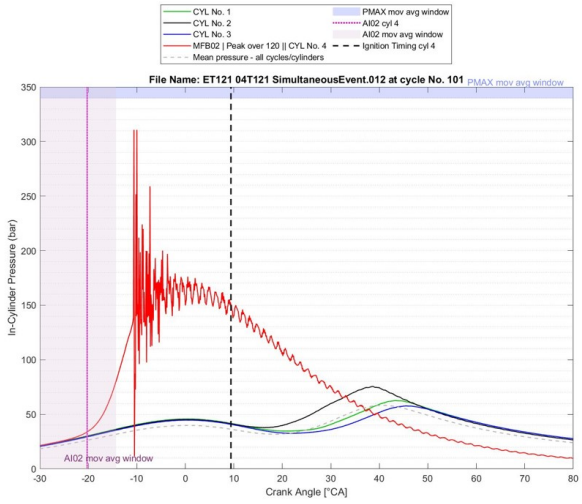

FIGURE 10: China bad fuel with 55 ppm MMT. Example of pre-ignition events captured from the continuous Indimaster log files.

was 150 bar. The acoustic ringing of the mega-knock peaked at over 300 bar.

China LSPI Fuel 55 PPM MMT

A reference test was conduced between each fuel to check the health of the engine. It can be seen from, Fig. 7, that the results from the second 102 RON reference test were consistent with the initial baseline test indicating that the engine health had been maintained despite prior exposure to a 315 bar PI event. Following this reference check, fuel with China LSPI base fuel specification plus 55 ppm MMT, was tested in combination with new oil. This testing was necessarily aborted after only a quarter of the first test had been completed due to failure of the electro-hydraulic valve-train, hence only a single test was completed with this fuel. Subsequent analysis of the sump oil indicated extremely high levels of FiO dilution (15%). It is possible that this oil dilution was due to the prevalence of LSPI with China LSPI fuel including MMT triggering an 'anti-runaway PI' calibration strategy that adds significant enrichment to cool the combustion chamber with fuel. Nonetheless sufficient LSPI data were captured during the short test time prior to failure, that some anaysis of the fuel's representative LSPI characteristics are possible, Fig. 10.

The cycle reported in Fig. 10 shows a very significant PI event on cylinder 4 triggered on both MFB02 and PMAX >120 bar. This is believed to characterise a new behaviour for fuel containing MMT not previously observed. Note the extremely early start of combustion – around 23 °BTDC – approximately 32°CA before the requested ignition timing. This suggests a different LSPI trigger mechanism to oil release or particle based LSPI which tend to occur closer to the spark timing. The resulting peak cylinder pressure of 160 - 170 bar occurred close to TDC (for reference the design pressure limit of the engine is 120 bar). The peak pressure of the acoustic ringing was 310 bar, which represents an extreme mega-knock event.

High LSPI Fuel

CAF 'High LSPI' fuel, a commercial reference fuel with known and repeatable LSPI promoting attributes, was tested in combination with new and used oil. The respective results were that an average of fifteen PI events were observed over 4 repeat test cycles with clean oil, which consistent with prior testing with this fuel, with an average of three events observed for the used oil case. As per the previous 'China LSPI' fuel tests, the used oil tests showed less propensity for LSPI than the clean oil cases – in direct contrast to results previously reported in the literature [10].

High LSPI Fuel 55 ppm MMT

A 102 RON reference test was again run to verify engine health prior to testing the high LSPI reference fuel with MMT addition. The resulting average of 5 LSPI events for the 102 RON fuel was consistent with the engine being healthy and fit to proceed with the testing despite having been previously exposed to an approximate 374 significant PI events.

During the first test on the high LSPI fuel with MMT and clean oil, the engine reached 226 PI events before having to be halted prematurely due to cooling system pressurisation, hence only a single test was completed with this fuel. After investigation the problem was found to be due to a cracked cylinder head, cylinder-2, exhaust side as shown in Figure 11. The deposits on the chamber roof on physical inspection appeared to be 'damp' due to coolant leakage in the region close to the visible cracks, highlighted in yellow. Also of note, is the red discolouration of the exhaust valves, consistent with manganese oxide deposits. To the authors knowledge this is the first such failure (cylinder head cracking directly attributable to LSPI) reported in the literature. The most commonly recorded damage due to LSPI being associated with the pistons and spark plugs [7] [14].

It can be seen from the representative example shown in Figure 12 that very severe LSPI events were encountered with High LSPI fuel containing MMT. This cycle triggered on cylinder 4

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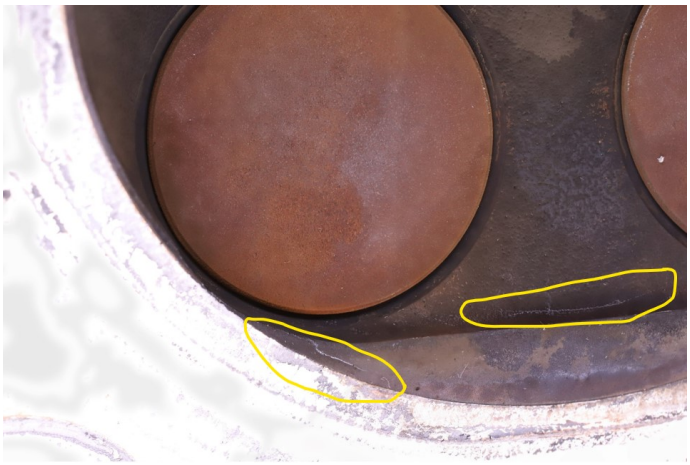

FIGURE 11: Cracked cylinder head, cylinder 2 exhaust side - High LSPI Fuel 55 ppm MMT

(red) due to PMAX exceeding 120 bar using the MFB02 moving average window. The mean peak cylinder pressure reached 160–170 bar, with a peak mega-knock pressure of 200 bar. Combustion occurred extremely early, estimated at 30 ° CA BTDC, despite a requested ignition timing of 3 ° CA ATDC, representing a 33 ° CA advance. Consequently, peak pressure occurred before TDC, subjecting the engine to severe mechanical loading – well beyond intended design limits and consistent with the observed engine damage.

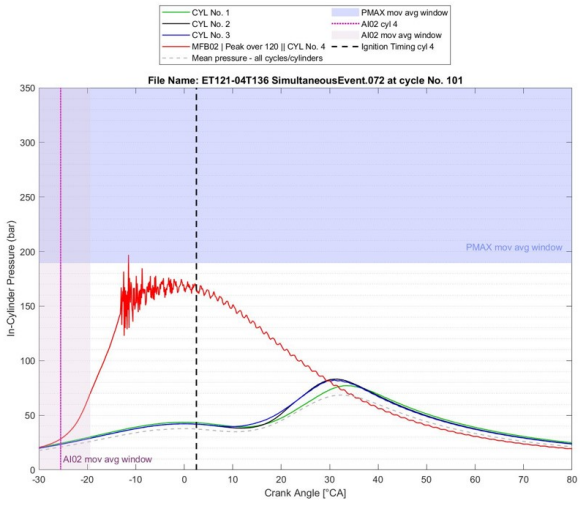

FIGURE 12: High LSPI fuel with 55 ppm MMT, examples of pre-ignition events captured from the continuous Indimaster log file.

Africa LSPI Fuel 30 ppm MMT

Prior to the testing of the Africa LSPI fuel, the engine was carefully inspected and rebuilt. Particular attention was paid to the bottom end. The high peak pressures encountered near TDC in previous test (leading to the reported cylinder head failure) are also transmitted directly from the piston crown through the gudgeon pin into the connecting rod, generating a significant

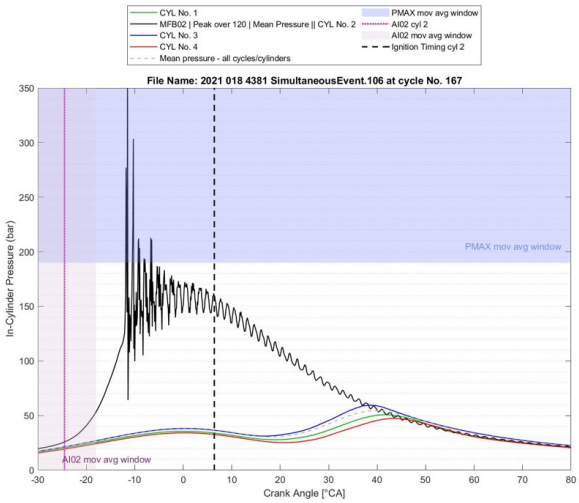

FIGURE 13: Africa LSPI fuel with 30 ppm MMT, example of pre-ignition events captured from the continuous Indimaster log file.

bending moment when the piston–pin–rod–crank system is close to vertical. The inspection confirmed that the bottom end was serviceable, allowing fitment of a new cylinder head and rebuild. Following reinstallation on the test bed, a 102 RON reference test confirmed acceptable engine health; albeit with an elevated average LSPI count of 11 events, the highest recorded value on this reference fuel.

The behaviour of this fuel was similar to all of the fuels containing MMT, characterised by: very early start of combustion, high mean pressure and very high acoustic ringing. The severity of in-cylinder pressure peaks from mega-knock events of 300 - 350 bar with this fuel were so extreme that engine failed (again on the first test) after just 107 events. This was an estimate due to sensor damage, constant errors from the indicating system and due to being a single test result does not allowing for any averaging. The engine was inspected and after the cylinder head was removed and pressure tested it was confirmed that micro cracks in the cylinder head were the cause of failure. This was unexpected as the replacement new cylinder head hadn't accumulated the hours of damage that the previous cylinder has been exposed to. This supports the view that the cylinder head damage was due to overload rather than fatigue.

The frequency and severity of high LSPI fuels containing MMT, and the resultant engine damage, was sufficient as to preclude further testing within the available project budget. In addition to the reported cylinder head failure consistent damage to the in-cylinder pressure transducers, caused by mechanical damage of the transducer diaphragms and evidenced by divergence in peak values of the TDC pressure at the end of compression, was noted during the 'Africa LSPI' fuel tests..

5.1. Run-away LSPI Events

A stand-out feature of the fuels containing MMT were run-away LSPI events. This phenomenon was described by Okada *et al.* [1] whereby a particle based ignition hypothesis is proposed as the cause of LSPI. Figure 14 shows the approximate number

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of multiple LSPI events recorded. It can be seen that no runaway events were present when using the 95 RON pump fuel. A very small number of consecutive or run-away events were also recorded for China LSPI and High LSPI fuels. In contrast, the same fuels with the addition of MMT exhibit high numbers of consecutive or run-away events. Test results from the Africa LSPI fuel could not be included in this figure due to the aforementioned damage to the in-cylinder pressure sensors. This resulted in the number of events being inaccurately captured due to indicating system synchronisation errors, meaning that most Africa LSPI run-away events were lost or corrupted. A proposed hypothesis for these run-away events is that an oil or hot particle based LSPI event leads to the so-called 'snow shaker' effect [1], whereby the first LSPI event dislodges particles on the combustion chamber surface that heat up and are retained. These particles then go on to cause run-away behaviour. This run-on behaviour is typically intermittent as once the dislodged surface particles are consumed they require a period of time to reform or redeposit. It is worthy of note that red manganese particles are clearly visible on the exhaust valves and combustion chamber surfaces in Fig. 11.

Fuel in Oil Dilution MMT Fuels

Following the failure of the valve-train, oil samples were analysed for FiO dilution using GC MS, for 95 RON and China LSPI fuel, Fig. 15. It can be seen that fuel in oil is relatively low for the typical dedicated LSPI test cycle (3.5 & 3.7% respectively), due to lower levels of knock retard and fuel enrichment. In contrast, China LSPI fuel resulted in very retarded ignition timing and fuel enrichment, leading to very high fuel in oil levels (15.2 & 18.2% respectively). Whilst the data is unavailable, it is anticipated that the FiO levels for the other fuels would be at least as high as China LSPI, but potentially higher. Based on guidance from the manufacturer, a value of 9% FiO dilution is considered excessive and puts the engine at significant risk of severe damage due to the reduction in kinematic viscosity of the oil. The KV100 is shown in Table 6 for 95RON E10 and China LSPI fuels, and illustrates the resulting difference between the KV100 at the start of test vs. the end of test.

[1](named by the author after the popular desk ornament which is shaken for snow particles to fall on the subject in a closed volume)

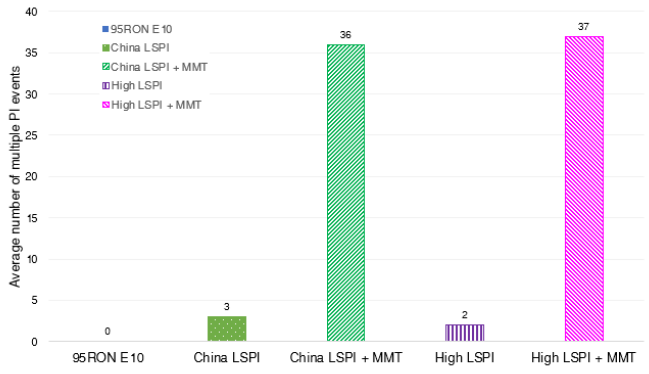

FIGURE 14: Comparison of run-away LSPI event tendency of fuels, with and without MMT.

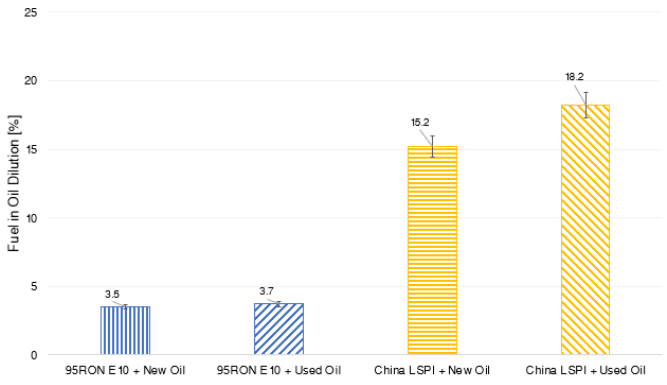

FIGURE 15: Fuel in Oil Dilution, 95 RON vs China LSPI Fuel, new and used fuel. Measured via GC MS, uncertainty ± 5% (D2887)

TABLE 6: Comparison of 95 RON E10 vs. China LSPI fuel: KV100 at start and end of test. KV (ASTM D445) uncertainty ±3%.

	Start of Test [cSt]	End of Test [cSt]	Δ [cSt]
95 RON E10 + New Oil	7.7	6.6	1.1
95 RON E10 + Used Oil	6.6	6.6	0.0
China LPSI + New Oil	7.7	5.0	2.7
China LSPI + Used Oil	6.6	4.5	2.1

It can been seen for the 95 RON E10 fuel that there is a drop of KV100 between the start of test to the end of test of 1.1 cSt. This is due to the new oil being diluted slightly with fuel, 3.5 %, Fig. 15. It is known from Figure 7 that only 2 events/test occurred and the resulting FiO dilution was comparable from the used service oil (3.75%), Tab. 5. When used oil was tested with the 95 RON fuel only 3.25 events/test occurred and there was no change in the KV100. China LSPI fuel had a high PI propensity with extremely high fuel in oil dilution. When new oil was tested there was a reduction in KV100 to 2.7 cSt, vs used oil 2.1 cSt respectively. Whilst there was a significant reduction in KV100 overall, the final viscosity of the used oil was only 0.5 cSt lower in the used oil than the new oil.

Fuel Injectors and Oxygen Sensors

The fuel injectors and oxygen sensors were changed with each fuel change, for the duration of the testing. The objective was to investigate if defective injection or control deviations were responsible for the LSPI events with fuels containing MMT. All parts evaluated post engine test and found to be fully functional and without defects. Hence is was concluded that malfunctioning sensors and actuators wer not the cause of abnormal combustion when using fuels containing MMT.

6. CONCLUSIONS

The results of the present study have shown the frequency and magnitude of LSPI events of fuels containing MMT to be significant and to cause engine damage as a result of mechanical stress. In fact, such was the severity of LSPI events encountered and the subsequent engine damage suffered that it wasn't possible

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to test all of the fuel and oil combinations planned for this study. A further significant finding of this work was the distinct difference in the type and phasing of LSPI in fuel containing MMT as opposed to those with no MMT content. In general, the average levels of PMAX weren't significantly higher than fuels without MMT, however, the highest recorded levels of PMAX were found when MMT was present in the fuel. Where MMT was present in the fuel the LSPI was characterised by very early MFB02 (~30 BTDC) and PMAX (150 - 170 bar) before TDC, >300 bar with the acoustic ringing caused by mega-knock. The gradient of the pressure rise was notably steeper with MMT present than without. The effect of used lubricating oil on the results for a 90 RON China LSPI fuel and a high-LSPI 96 RON reference fuel was to reduce the number of LSPI events recorded. This result is contrary to behaviour previously reported in the literature (and that which was observed in this study for a standard E228 based reference fuel with a 10% ethanol content). The cause of this behaviour is currently not understood. FiO dilution was extremely high in tests where MMT was added to the fuel (15 – 18 % v/v), far in excess of the safe maximum limit of 9 % v/v. The resulting reduction in oil viscosity (2 – 3 cSt) was such that mechanical failures of the electro-hydraulic valve train components occurred. A tenfold increase in the propensity for runaway LSPI events was observed when using fuels containing MMT compared with those without, suggesting the presence of a particle-based mechanism in addition to that proposed by Nomura [6]. The results demonstrate with reasonable certainty that the cause of LSPI, when MMT is included in the fuel, was not related to injector wear or misreading oxygen sensors. It is possible that these factors could lead to increased LSPI tendency to occur over extended engine lifetime, but are not the primary causal mechanism for increase LSPI frequency with MMT.

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