

Deposit Formation of Flex Fuel Engines Operated on Ethanol and Gasoline Blends

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ABSTRACT

A test procedure was developed to assess the deposit-forming tendencies of gasoline/ethanol fuel blends, ranging from 0 % to 100 % ethanol (E0 to E100). The test engine was a Ford 1.8l - 4 cylinder -16 valve -natural aspirated flex fuel engine, which is used in various vehicle models, such as the European Focus and C-MAX.

The test cycle, a realistic engine speed/torque profile, based on an urban driving pattern, provided good differentiation between different gasoline/ethanol fuel blends as well as between additized and non-additized fuel blends. With unadditized E85 critical deposits were found in the intake system, on the intake valves, in the combustion chamber and on the injector tips. Well known deposit control additives (DCA) used in gasoline such as PIBA (polyisobutyleneamine) and PEA (polyetheramine) were examined in E85 for deposit control effectiveness of intake valves, injectors and combustion chambers. The hump effect, known to occur in gasoline fuels at very low additive dosages, could not be found with this particular test engine and test cycle.

The main cause of injector deposits with E85 fuel was identified to be engine design related.

Going from E10 to E100 it was shown that higher ethanol contents tended to decrease intake valve and combustion chamber deposits, both with and without additives.

Another test series simulated switching between E85 fuel and E5 fuel from one tank to another. This tanking pattern is

relevant for markets where E85 and E5 (or E10) fuels are available at similar prices.

The suitability of PIBA-containing additive packages for deposit control could be demonstrated for fuel blends up to 100 % ethanol content. For fuel blends of very high ethanol content, however, alternative additive packages were shown to provide improved compatibility with the fuel. An in-house filtration test permitted differentiation between conventional and new additive packages.

INTRODUCTION

The world-wide personal vehicle fleet is growing significantly until 2030 and with it also the demand for secured supply of gasoline (see Fig. 1).

(See [Figure 1](#) after last section of paper)

With renewable fuel mandates in the world and, in particular, with the US Energy Independence and Security Act 2007 (see [Fig. 2](#)) the amount of ethanol produced for use in gasoline will grow disproportionately and thus the volume of high concentration blends (E85 in the US and Europe, E100 in Brazil) will grow.

(See [Figure 2](#) after last section of paper)

Automakers have responded by increasing the number of flex fuel vehicles (FFV) produced. In 2008 more than 6 million FFVs were on the roads of the United States, the largest market for gasoline and gasoline powered cars in the world. In March 2006, Chrysler, GM and Ford pledged to convert 50 percent of each company's fleet to FFVs by 2012 [3].

Negative impacts of deposits formed in the intake system of spark-ignited engines have been widely investigated in the past [4,5,6]. There is some evidence that low levels of ethanol (like E10) can cause increased deposits. In [7] significantly increased intake valve deposits (IVD) were reported in a BMW 318i (following the ASTM D5500 driving cycle) after 8,000 km when it was operated with E10 instead of E0 fuel. The increased deposits were found with unadditized as well as with additized fuels.

In [8] the effects of E10 were evaluated in the M111E engine (following the CEC F-020-98 test procedure). In this study unadditized E10 was found to increase IVD compared to unadditized E0. When the fuels (E0 and E10) were additized the difference vanished.

In [9] E0, E10, E25 and E85 fuels were used in a 2006 FFV pickup truck with a 5.3 l V8 engine. E10 resulted in an increase of IVD compared to E0. Higher levels of ethanol reduced the level of deposits slightly below E0 level (unadditized fuel). IVD of unadditized E85 could be controlled by adequate doses of deposit control additive (DCA).

In [10] E0 and E85 fuels were used in a 2006 GM FFV passenger car with a 3.5 l V6 engine. The IVD forming tendencies were similar to a non-FFV when fuelled on E0. Sulfates from ethanol and certain types of corrosion inhibitor used in E85 contributed significantly to the formation of IVD. Polyisobuteneamine (PIBA) and polyetheramine (PEA) based DCAs were effective in minimizing IVD formation with E85. No injector deposits were found except for some sodium and potassium sulfates in a circular pattern around the injector tip when the ethanol contained 4 ppm sulfates.

There is still not much known about the interactions between varying gasoline/ethanol blends and additives on the deposit forming tendency of FFVs.

This paper describes the deposit forming tendency of IVD and injectors with E0 and E85 for the test engine. The IVD hump effect, which is a potential risk for FFVs, was investigated with very low dosages of DCA. Furthermore the origin and composition of injector deposits were analyzed. The ability to control injector deposits was investigated by means of PIBA and PEA based DCAs.

Systematic test series with gasoline/ethanol blends were carried out:

- With E10 to E100 in order to look into the effect of increasing ethanol contents on IVD in the absence or presence of a DCA.
- A test series in order to simulate a highly dynamic refueling behavior of a typical FFV driver by switching between E5 and E85 for each refueling.

- The dependency of combustion chamber deposits on the ethanol content and DCA was investigated.
- An in-house filtration test was utilized in order to investigate potential issues about the mixing behavior of conventional DCAs with fuel blends of high ethanol content.

1. BASICS OF DEPOSIT FORMATION

1.1. DEPOSIT FORMATION AND ADDITIVE PERFORMANCE

Deposits in spark ignited engines are formed not only by the fuel but also by oil flow, blow-by gases (positive crankcase ventilation, PCV) and combustion gases (exhaust gas recirculation, EGR). The carbon-like deposits are typically found in the intake ports, on intake valves (see Fig. 3) and on injector tips (see Fig. 4).

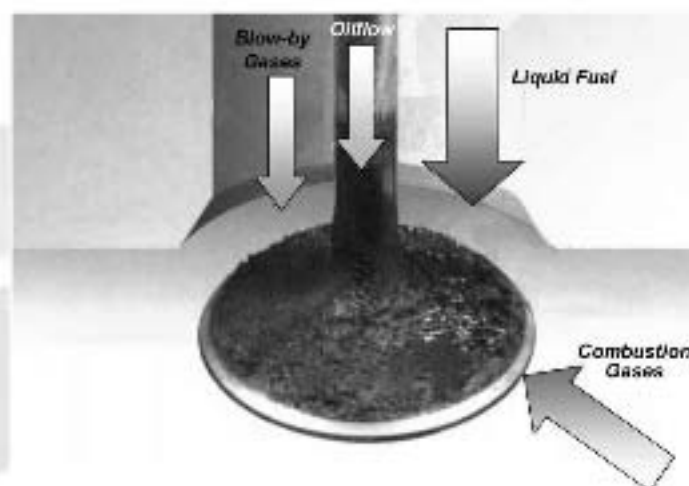


Figure 3. Deposit formation on intake valve



Figure 4. Injector deposits

Modern DCAs designed for gasoline fuels and for blends with low volumes of ethanol (typically E10) prevent deposit build-up during engine operation by creating a protective film on the metal surface and are also able to remove existing deposits when used in higher dosages. In [10] the US rules

and recommendations for obligatory E85 DCA additization are discussed. In Sweden the gasoline part of E85 (15 % gasoline by volume) has to be additized according to the Swedish EPA ruling. Fuel grade ethanol (without additive) is splash blended on top of the additized gasoline but the end product E85 is not additized furthermore [11]. According to our knowledge currently no regulations exist for the additization of E85 in the rest of the world.

1.2. THE IVD HUMP EFFECT

With low dosages of DCA it is a known risk that a hump effect can exist for engines running on E0, E5 or E10 fuel [12]. The amount of additive is not sufficient to create a protective film on the metal surface. The additive itself accumulates in the deposits. The result is higher IVD than without additive.

We found higher IVD with the Ford 2.3 l engine (following the ASTM D6201 test procedure) at low dosages (129 ppm) of three different DCAs (Additives A, B and C, commercial additives which are used in the market) compared to unadditized test fuel (RON 95 according to EN 228) (see Fig. 5). Low dosage in this case means LAC, the lowest additive concentration allowed according to the rules of the US EPA. At higher dosages of 190 and 247 ppm the additives reduce the IVD effectively compared to the unadditized test fuel.

(See [Figure 5](#) after last section of paper)

2. FLEXIFUEL TECHNOLOGY & TEST ENGINE

In cooperation with FORD, a 1.8-l-Duratec Flexi Fuel engine was set up on one of BASF's test benches. The engine is a naturally aspirated (NA) 4 cylinder- 16 valves- in-line type with port fuel injection (PFI). It is flex fuel capable from E0 up to E85 (85 %vol ethanol content). The rated power is 92 kW @ 6000rpm, while maximum torque is 170 Nm @ 4500 rpm.

Compared with the base engine the Flexi Fuel engine is equipped with especially hardened exhaust valve seat inserts, corrosion resistant injectors with increased flow rate, and an ethanol resistant fuel system. The Flexi Fuel engine control unit (ECU) is featured with two sets of calibration tables, one for E0 and another for E85. By means of the lambda oxygen sensor and a special type of software algorithm the ethanol concentration of the fuel is recognized permanently during engine operation. Based on the determined ethanol content the actual spark and fuel settings are derived by interpolation between the tables.

On the test bench (see [Fig. 6](#)) the engine was also operated with E100, using the E85 calibration with closed loop lambda control.



Figure 6. Test engine (FORD 1.8-l-Duratec Flexi Fuel) on test bench

3. TEST PROCEDURE

3.1. TEST PATTERN

The first test applied was a 60 hours simulated M111 IVD test cycle (following the CEC F020-98 test procedure) because of the very similar engine design. Both engines are 4 cylinder NA PFI in-line types with 16 valves and a displacement between 1.8 and 2.0 liter.

During these sequences some intake valve deposits were observed for both unadditized E0 and unadditized E85 but very little injector deposits were found. In order to achieve more injector deposits the test cycle was modified from the quasi static operation pattern to a more dynamic pattern which simulates urban driving (see [Fig. 7](#) and [8](#)). The newly established 60 hours test cycle is prone to build up injector as well as intake valve deposits in the required amounts.

(See [Figure 7](#) after last section of paper)

(See [Figure 8](#) after last section of paper)

3.2. TEST RATING

In order to evaluate the additive effectiveness intake valve deposits (IVD) and total combustion chamber deposits (TCD) were quantified by deposit mass determination following the CEC F-020-98 test procedure (see [Fig. 9](#)) and a visual rating of the injectors was performed (see [Fig. 10](#)).



Figure 9. Deposit mass determination of IVD



Figure 10. Visual rating of injectors. Dirty Injector (left) and Clean Injector (right)

3.3. TEST FUELS

The E10 - E85 fuels used were splash blends consisting of fuel grade ethanol from a German manufacturer and conventional RON 95 unleaded gasoline from the German market (E0 or E5). In some tests technical grade ethanol was used instead of the fuel grade ethanol. Control experiments showed no significant differences between the two ethanol types. In the course of the test work no indication was found for excessive sulfate or corrosion inhibitor content of the fuel grade ethanol. Neither the ethanol nor the gasoline fuels contained any deposit control additive (DCA).

E85 is defined by different standards, for example ASTM D5789 for the USA or DIN 51625 for Germany and EN 15293 (draft version only) for the European Union. Denatured ethanol for blends with gasoline is also defined by different standards, for example ASTM D4806-06c for the USA or EN 15376 (draft version only) for the European Union.

4. TEST RESULTS AND DISCUSSION

4.1. DEPOSIT FORMING SENSITIVITY WITH E85 AND E0

After 60 hours of operation with unadditized E85 significant deposits were found on the intake valves (see Fig. 11) and on the injectors (see Fig 12),



Figure 11. IVD after 60 hours with unadditized E85



Figure 12. Injector deposits after 60 hours with unadditized E85

whereas with unadditized E0 significant deposits were only found on the intake valves (see Fig 13) and not on the injectors (see Fig 14).

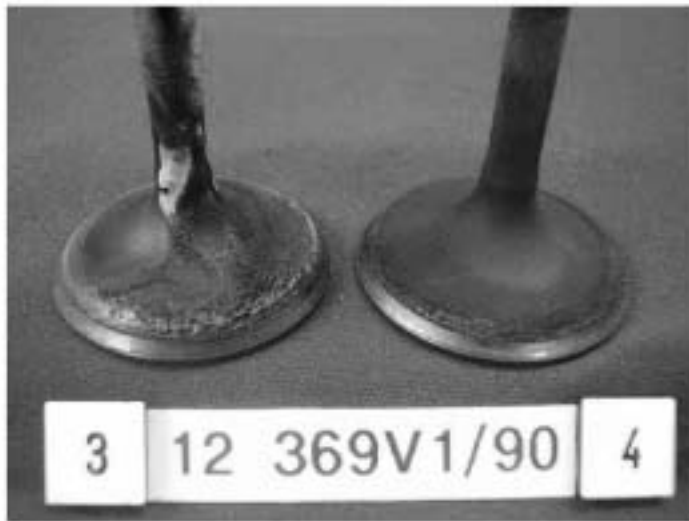


Figure 13. IVD after 60 hours with unadditized E0



Figure 14. Injector appearance after 60 hours with unadditized E0

showing reduced IVD even at very low dosages of 15 or 30 ppm in E85 (see Fig. 15).

(See Figure 15 after last section of paper)

This result, however, must not be generalized for other combinations of FFV engines, E85 test fuels and test cycles. There could be combinations where an IVD hump effect would appear.

4.3. ORIGIN AND COMPOSITION OF INJECTOR DEPOSITS

In 4.1 it was shown that unadditized E85 creates injector deposits on the test engine but unadditized E0 does not. In another experiment with unadditized E85 the EGR and blow-by gases were deliberately removed from the intake system and redirected to the environment, which resulted in significantly less injector deposits. For comparison see Fig. 16 and Fig. 17.

4.2. RISK OF IVD HUMP EFFECT IN FFVS

Due to the usual blending of additized gasoline with unadditized ethanol the E85 fuel as used for flexi fuel vehicles (FFV) has a considerable potential to contain too low dosages of DCA and subsequently to create an IVD hump effect issue.

In Sweden, for example, additized gasoline from the market is splash blended with fuel grade ethanol without further additizing the E85. If the gasoline portion of the E85 is estimated to be additized in a range of 400 to 800 ppm then the E85 can be estimated to be additized in a range of 60 to 120 ppm, clearly at or close to LAC dosages.

Therefore it has been important to have a closer look at the response behavior of the test engine towards low dosages of DCA in E85. Surprisingly no hump effect occurred at low and ultra-low dosages of a PIBA based DCA. Instead the engine turned out being very responsive to the DCA and



Figure 16. Injector deposits after 60 hours with unadditized E85 / with EGR and blow-by



Figure 17. Injector deposits after 60 hours with unadditized E85 / without EGR and blow-by

Thus there must be an interaction of EGR and blow-by gases which are directed into the intake system with E85 to form injector deposits, whereas there is no such interaction with E0 (see 4.1).

Therefore injector deposits from a test run “unadditized E85 / with EGR and blow-by” were analyzed by different means (test run “unadditized E85 / without EGR and blow-by” did not provide enough deposits for analysis).

X-ray fluorescence analysis (XRF) revealed metallic and semi metallic elements like sulfur, calcium, zinc, phosphorous and iron which are typical for engine oil or engine wear (see Fig. 18).

(See Figure 18 after last section of paper)

The IR analysis of the injector deposits revealed a close similarity to the spectrum of iso-butylene (from database). In addition the injector deposits have a band at 1702 cm⁻¹ which is typical for an imide band (see Fig. 19).

(See Figure 19 after last section of paper)

The conclusion from the analytical results and from the engine experiment is

- the injector deposits consist, at least to a large extent, of “PIB” material (polyisobutene)
- the imide band is an indication for the “PIB” material to be “PIBSI” (polyisobutenesuccinimide) which is the dispersant in all engine oils
- the origin of the injector deposits from the engine oil is further evidenced by the elements found by XRF
- PIBSI is the only possible source of PIB material in this experiment since the fuel was not additized

- PIBSI from the engine oil is not compatible with high volumes of ethanol in the fuel which causes the injector deposits

4.4. CONTROL OF INJECTOR DEPOSITS WITH DCA

Two different types of DCA, namely a PIBA based DCA and a PEA DCA were used to reduce the injector deposits when the test engine was operated with E85. A high dosage of the DCAs was chosen since in [13] it has been concluded that PIBA based DCAs could cause injector deposits whereas a PEA DCA would not. If there is such a detrimental effect of PIBA our experiments should have been able to confirm it.

In our experiments the injector deposits with unadditized E85 were significantly reduced when 1000 ppm of a PIBA based DCA or a PEA DCA were used in E85 (see Fig. 20). The positive result for PIBA is surprising in view of [13] but can be explained by the fact that the authors of [13] have not been able to obtain E85 completely free of PIB based additive for back to back tests.

(See Figure 20 after last section of paper)

4.5. ETHANOL CONTENT AND DEPOSIT CONTROL WITH DCA

In a systematic test series with unadditized gasoline/ethanol blends from E10 to E100 (E0 not tested; no tests between E10 and E50) we found that increasing ethanol content above E10 decreases IVD (black/dark bars in Fig. 21). Above 50 % the ethanol effect flattens out. Qualitatively the same result is found with DCA at 200 ppm (green/grey bars in Fig. 21) but IVD are much lower than without DCA. At a higher dosage of DCA the IVD can be reduced further (not shown here).

(See Figure 21 after last section of paper)

4.6. SWITCHING BETWEEN E85 AND E5

In this test series a highly dynamic refueling behavior of a typical FFV driver was simulated where the driver switches between E5 and E85 for each refueling, a behavior possible in markets like Sweden or the USA.

As described in 4.2 this behavior can mean in reality that the car is fuelled by low additized E85 and by regularly additized E5. For our test series we chose 60 ppm of a commercial PIBA-based DCA for E85 and 388 ppm for E5 (instead of E5 also E0 or E10 could have been chosen). A test time of ten hours was the equivalent to one tank of fuel.

The gravimetric evaluation of IVD after each test interval and also the visual inspection of the injectors revealed that the

additive provides excellent control of IVD and injector deposits and no problems are expected in the field (see [Fig. 22](#)).

(See [Figure 22](#) after last section of paper)

4.7. TOTAL COMBUSTION DEPOSITS (TCD)

Total combustion deposits (TCD), piston top deposits plus cylinder head deposits plus fire land deposits plus cylinder head gasket deposits, from the test series 4.5 were evaluated gravimetrically following the CEC F-020-98 test procedure both for the unadditized (black/dark bars in [Fig. 23](#)) and additized (green/grey bars in [Fig. 23](#)) gasoline/ethanol blends from E10 to E100.

Increasing the ethanol content above E10 decreased the TCD with or without additive. The ethanol effect for TCD, though, is not as pronounced as for IVD. The presence of the additive caused a slight increase in TCD at the E10 level which is inline with experience from non FFV engines (e.g. the M111E engine following the CEC F-020-98 test procedure) and a somewhat larger increase at E50 to E100. It is worth pointing out that for E85 and E100 the TCD with additive are below the level of E10 without additive.

(See [Figure 23](#) after last section of paper)

4.8. ETHANOL ADDITIVE PACKAGE

We found that high dosages of PIBA based DCAs have a compatibility problem with E85.

For example see the photo in [Fig. 24](#): with 1000 ppm of PIBA based DCA in E85 a visible haze is apparent (bottle on the right side) whereas with 1000 ppm of PIBA based DCA in the gasoline portion (which is equivalent to about 150 ppm in E85) no haze is apparent (bottle in the middle).

It must be pointed out that the hazy material can only be a small fraction of the total PIBA material since the performance in E85 still exists (see 4.4).

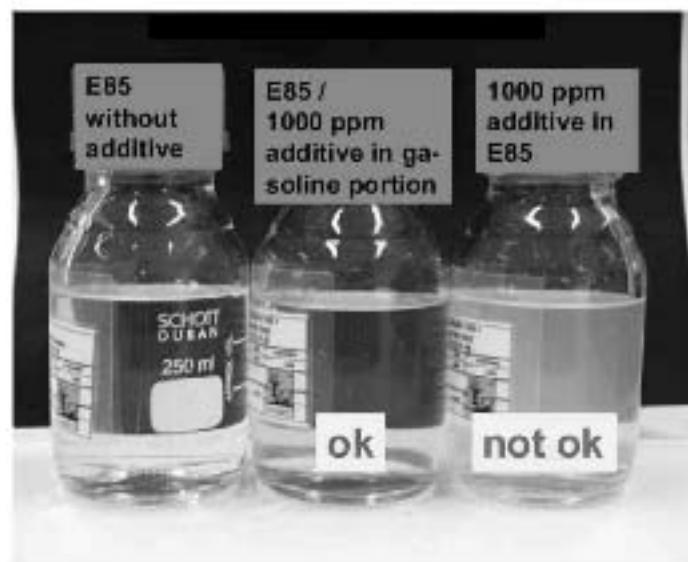


Figure 24. Clear E85 without additive (left), clear E85 with 1000 ppm PIBA DCA in the gasoline portion, equivalent to 150 ppm in E85 (middle) and hazy E85 with 1000 ppm PIBA DCA (right)

In order to evaluate the compatibility problem further we set up an in-house filtration test:

A SEDAB filtration test set-up [14] was modified with a 0.2 micron filter (regenerated cellulose) to check the filtration time of 100 ml increments of E85, either unadditized or additized with increasing dosages (250, 500 and 1000 ppm) of PIBA based DCA. A deviation from a linear increase in filtration time with volume would indicate some sort of “filter plugging” which in turn would be a proof that insoluble parts of the PIBA material could pose a problem in the field.

In fact we found that with a rather high dosage of 1000 ppm of a PIBA based DCA in E85 there was a significant deviation from the linear curve (light blue/upper curve in [Fig. 25](#)) whereas up to 500 ppm there was no large deviation from the base which was unadditized E85 (purple/lower curve in [Fig. 25](#)).

(See [Figure 25](#) after last section of paper)

It has to be pointed out that this test set-up is a sort of “worst-case” since a typical requirement for a fuel filter in a FFV vehicle is a retention efficiency of 82 % for particles larger than 5 micron with a filter module made from Nylon 6 [15]. In fact we did not find any indication for filter plugging in our in-house test with filters of larger pore size like 1 micron or even 0.5 micron.

An especially formulated ethanol additive package did not show any filtration problem in our in-house test even at a dosage of 1000 ppm ([Fig. 26](#)).

(See [Figure 26](#) after last section of paper)

Also the visual appearance of the new additive package, even at a dosage of 1000 ppm in E85 confirms the improved compatibility (see Fig. 27).

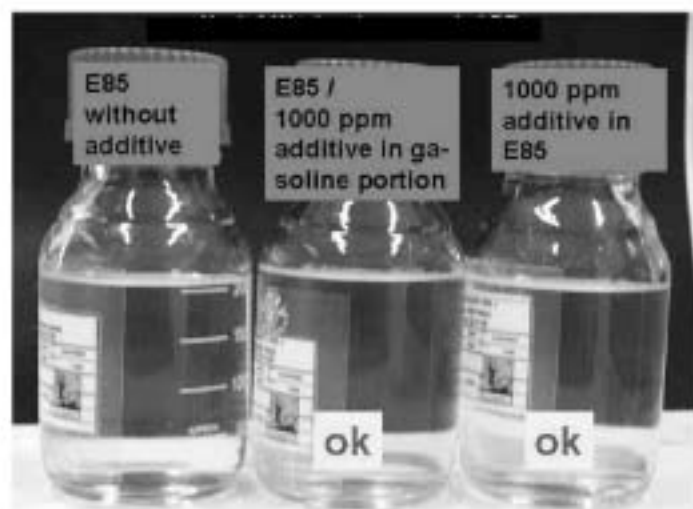


Figure 27. Clear E85 without additive (left), clear E85 with 1000 ppm ethanol additive package in the gasoline portion, equivalent to 150 ppm in E85 (middle) and clear E85 with 1000 ppm ethanol additive package (right)

SUMMARY/CONCLUSIONS

For the test engine and test cycle used in this work, and for the fuels and additives used we found

- With unadditized E85 the test engine was prone to accumulate deposits on intake valves and injectors, whereas with unadditized E0 deposits were found only on the intake valves but not on the injectors.
- The IVD hump effect, which is a potential risk for Flexi Fuel Vehicles, could not be found with this specific combination of test engine, E85 test fuel and test cycle.
- EGR and blow-by gases were identified as the main cause for injector deposits when using unadditized E85
- PIBSI from engine oil was identified to be the cause for injector deposits due to its incompatibility with ethanol.
- Injector deposits can be effectively controlled by PIBA and PEA based DCAs in E85.
- Higher contents of ethanol than E10 decrease IVD in the absence or presence of a DCA. Above 50 % the ethanol effect flattens out.
- PIBA based DCA can control IVD at all ethanol contents.
- Switching tank fillings between low additized E85 and regularly additized E5 provided excellent control of IVD and injector deposits.
- Higher contents of ethanol than E10 decrease TCD in the absence or presence of a DCA although the effect is not as pronounced as for IVD.

- A high dosage of PIBA based DCA has problems with E85 compatibility and in a simulated E85 filtration test. An especially formulated ethanol additive package avoids these problems.

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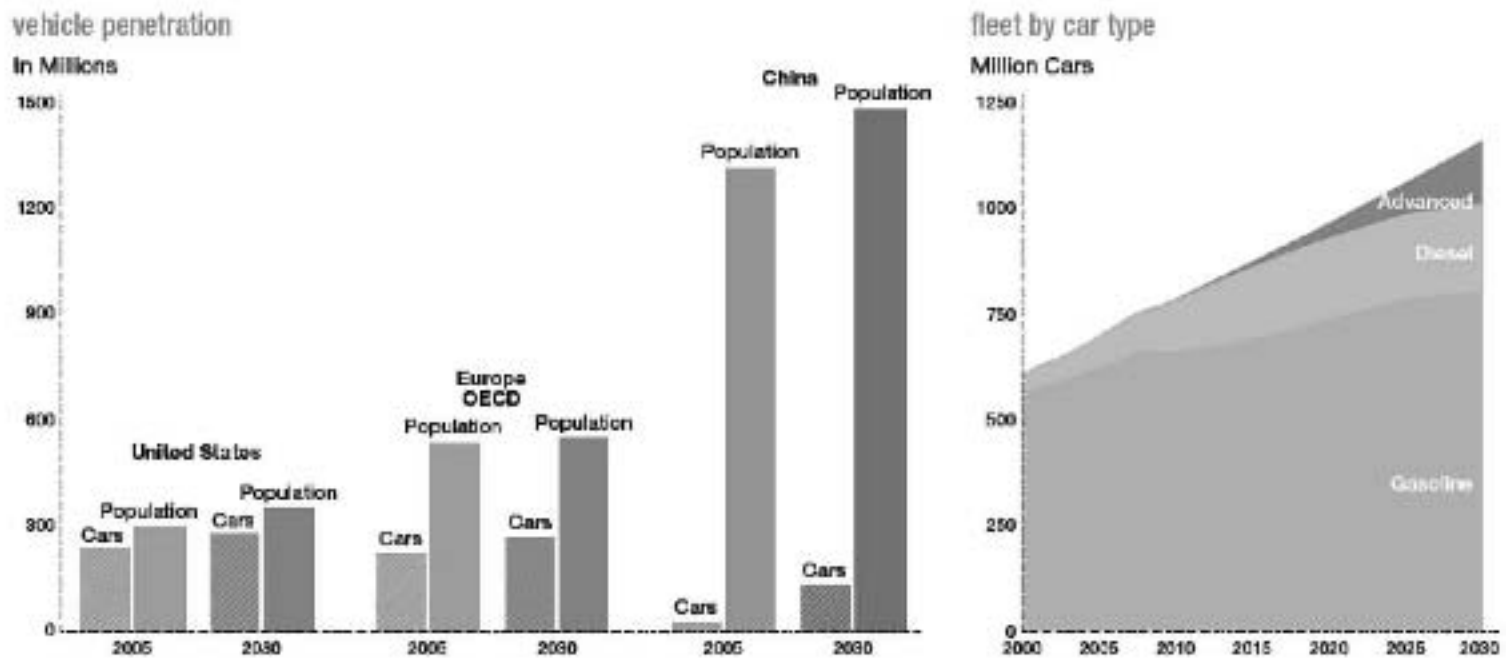


Figure 1. Personal vehicle fleet is growing. Source [1]

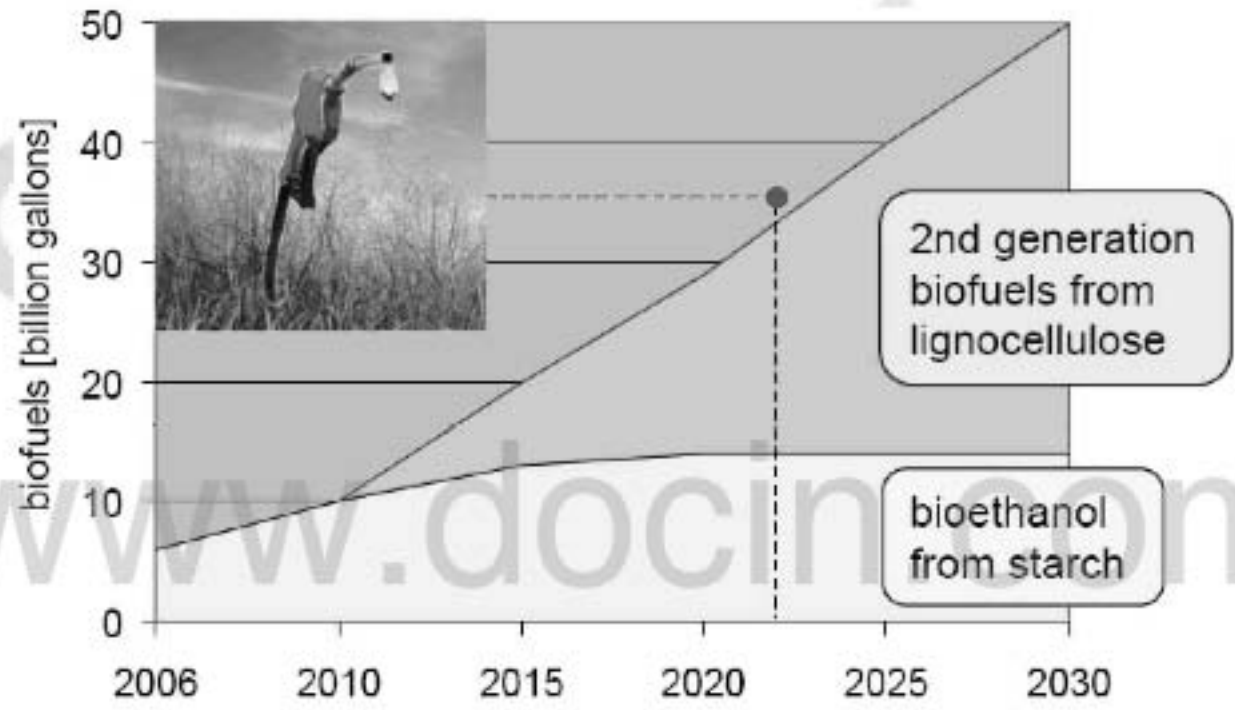


Figure 2. US Energy Independence and Security Act 2007: 36 Billion gallons of biofuels in 2022 (equivalent to 20% of US gasoline consumption). Source [2]

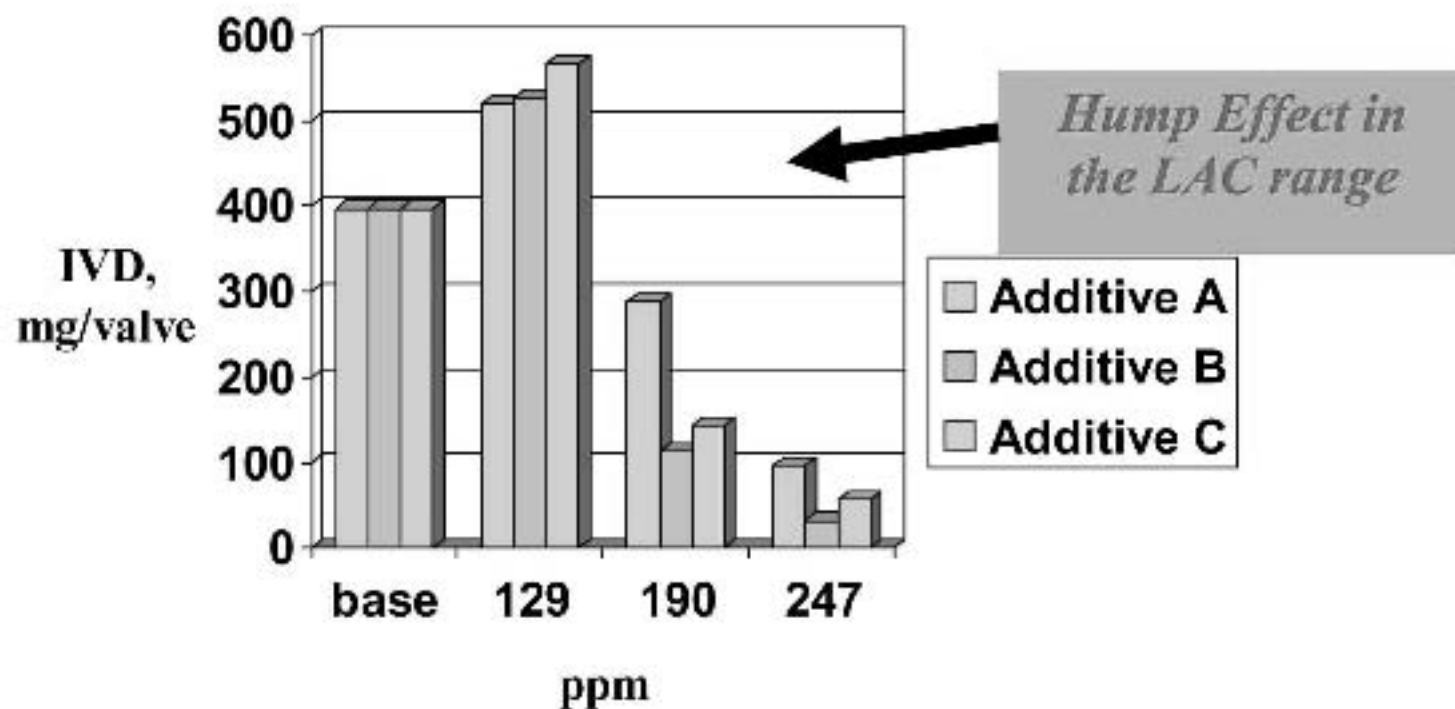


Figure 5. IVD hump effect with three different commercial additives

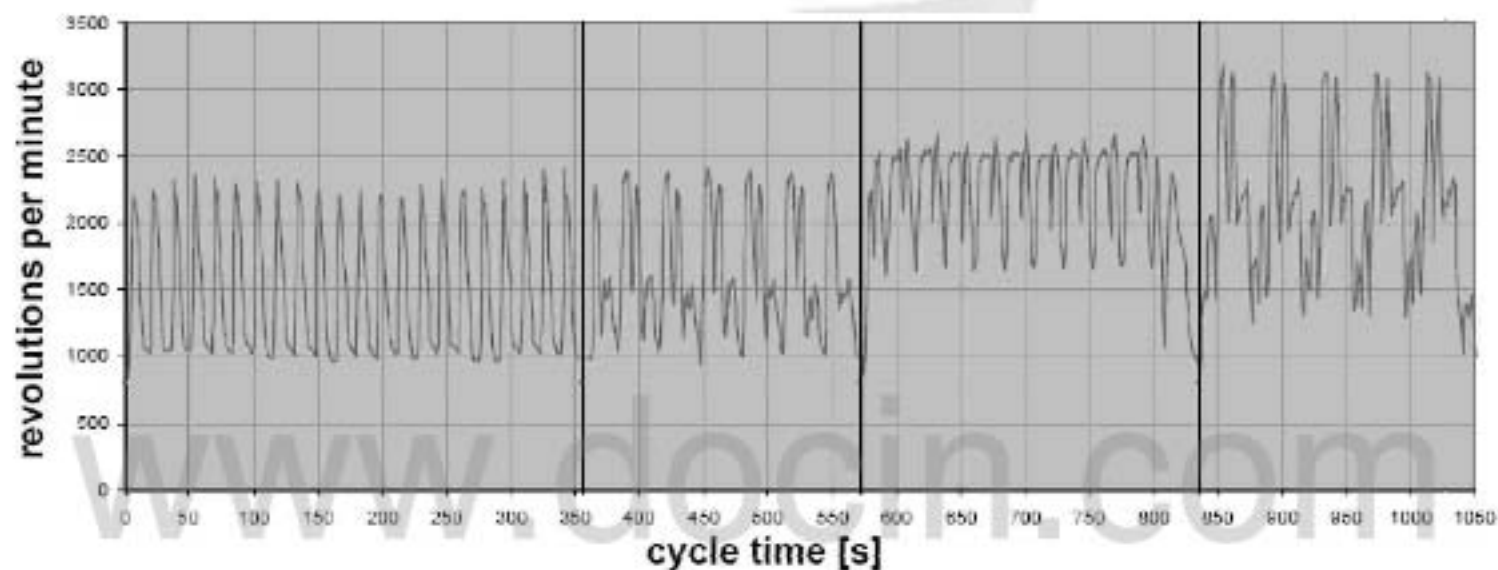


Figure 7. Dynamic test cycle, revolutions per minute in 1052 s cycle, total test time 60 hours

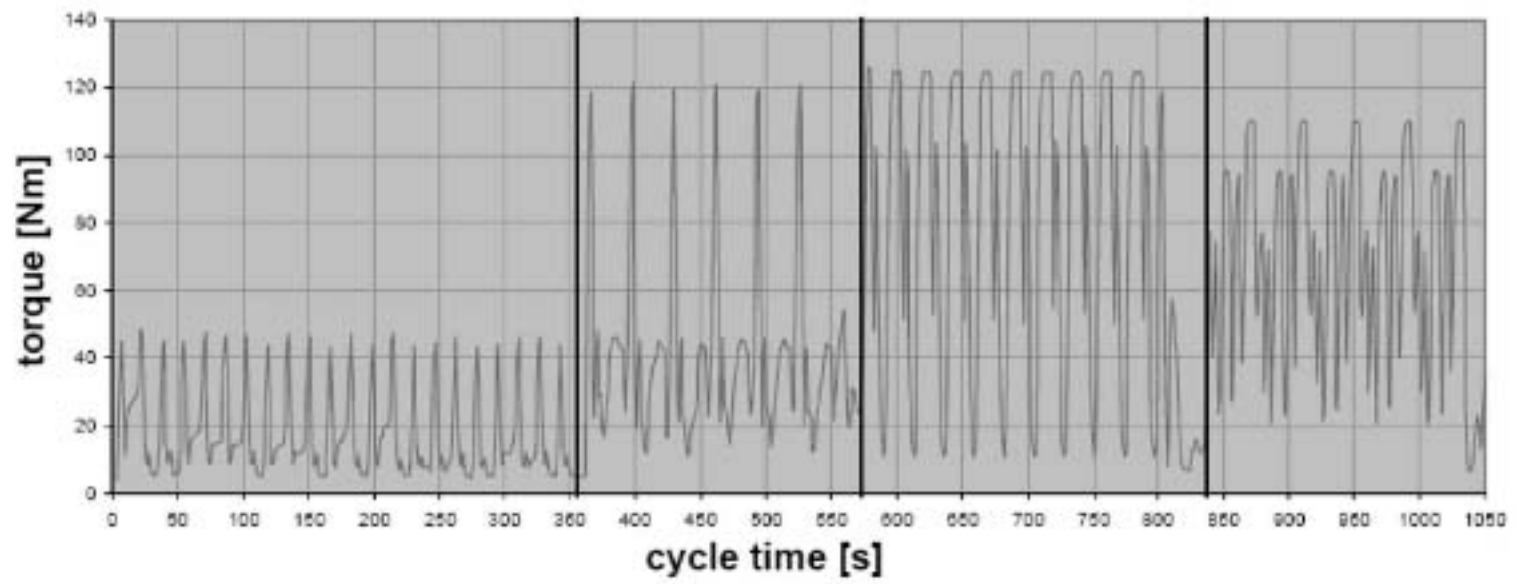


Figure 8. Dynamic test cycle, torque in 1052 s cycle, total test time 60 hours

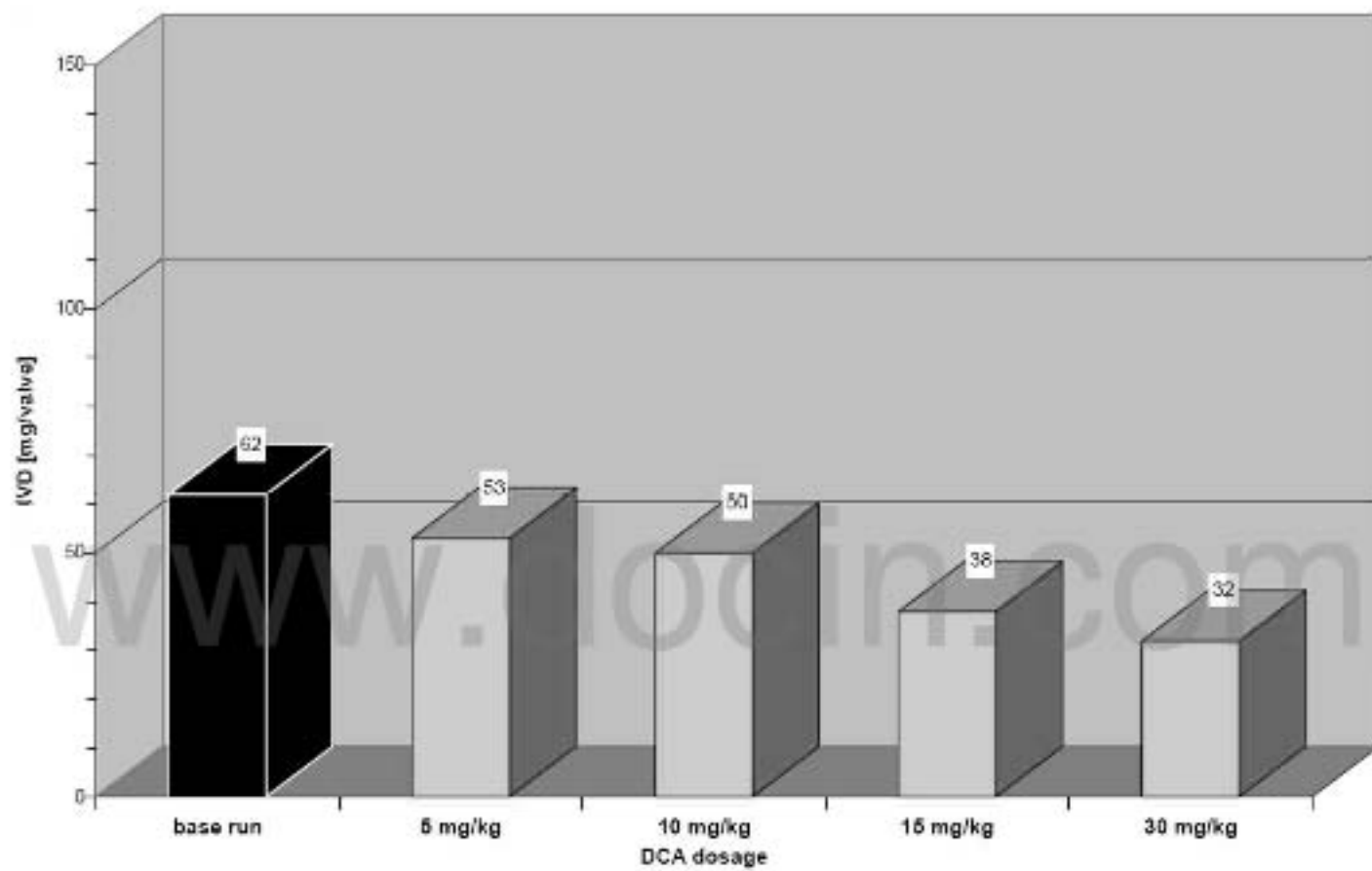


Figure 15. IVD with E85 at low and ultra-low dosages of DCA.

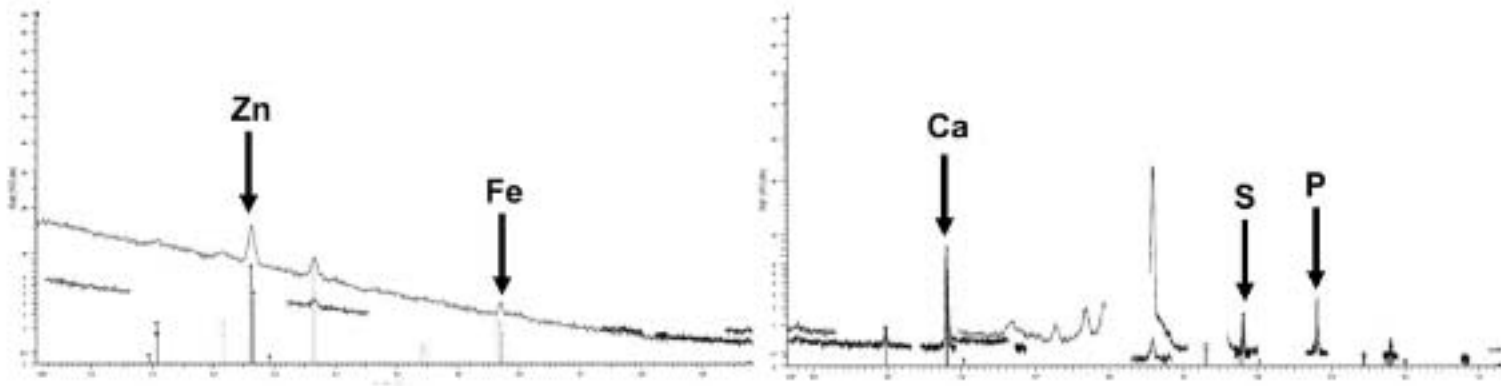


Figure 18. XRF of injector deposits from a test run "unadditized E85 / with EGR and blow-by".

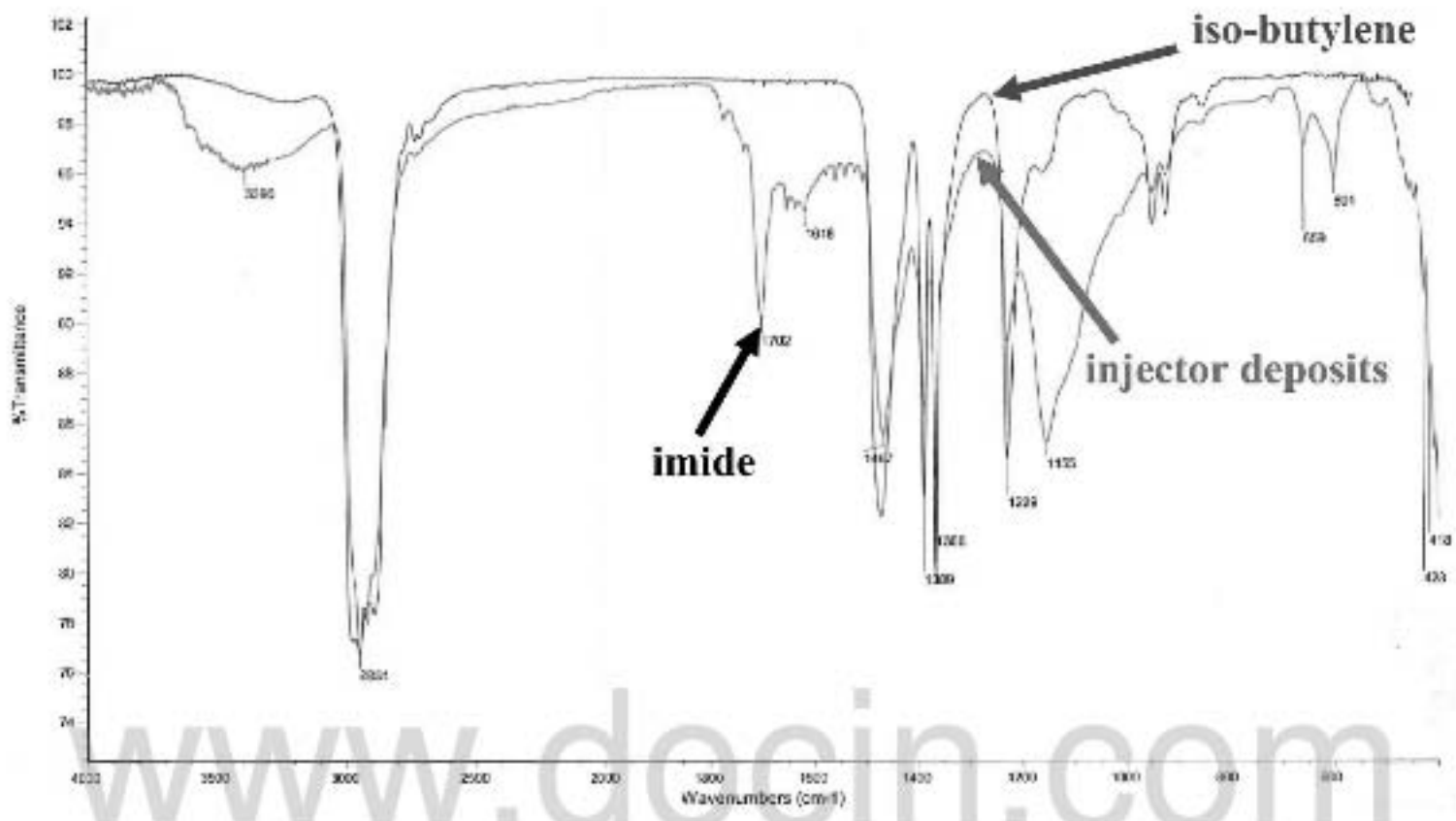


Figure 19. IR of injector deposits from a test run "unadditized E85 / with EGR and blow-by" and of iso-butylene (from database).

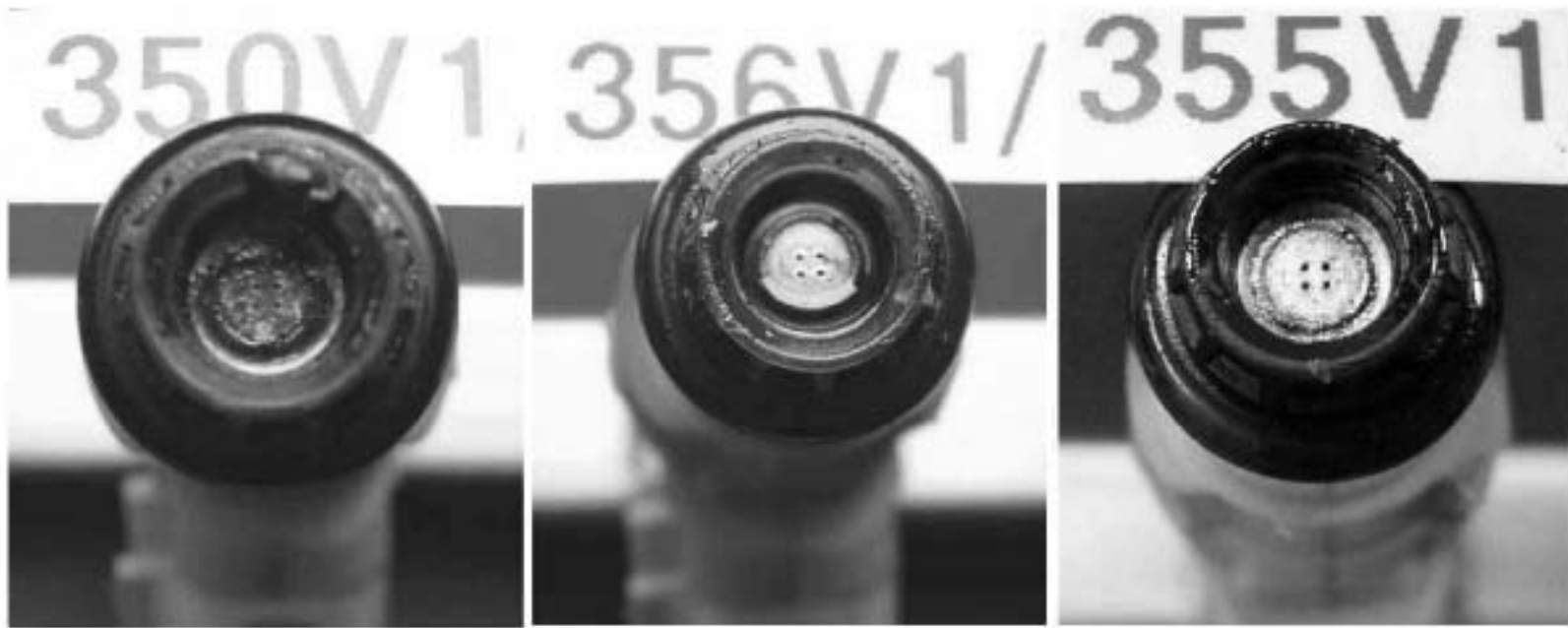


Figure 20. Injector deposits after 60 hours with unadditized E85 (left), with 1000 ppm PIBA DCA (middle) and with 1000 ppm PEA DCA (right)

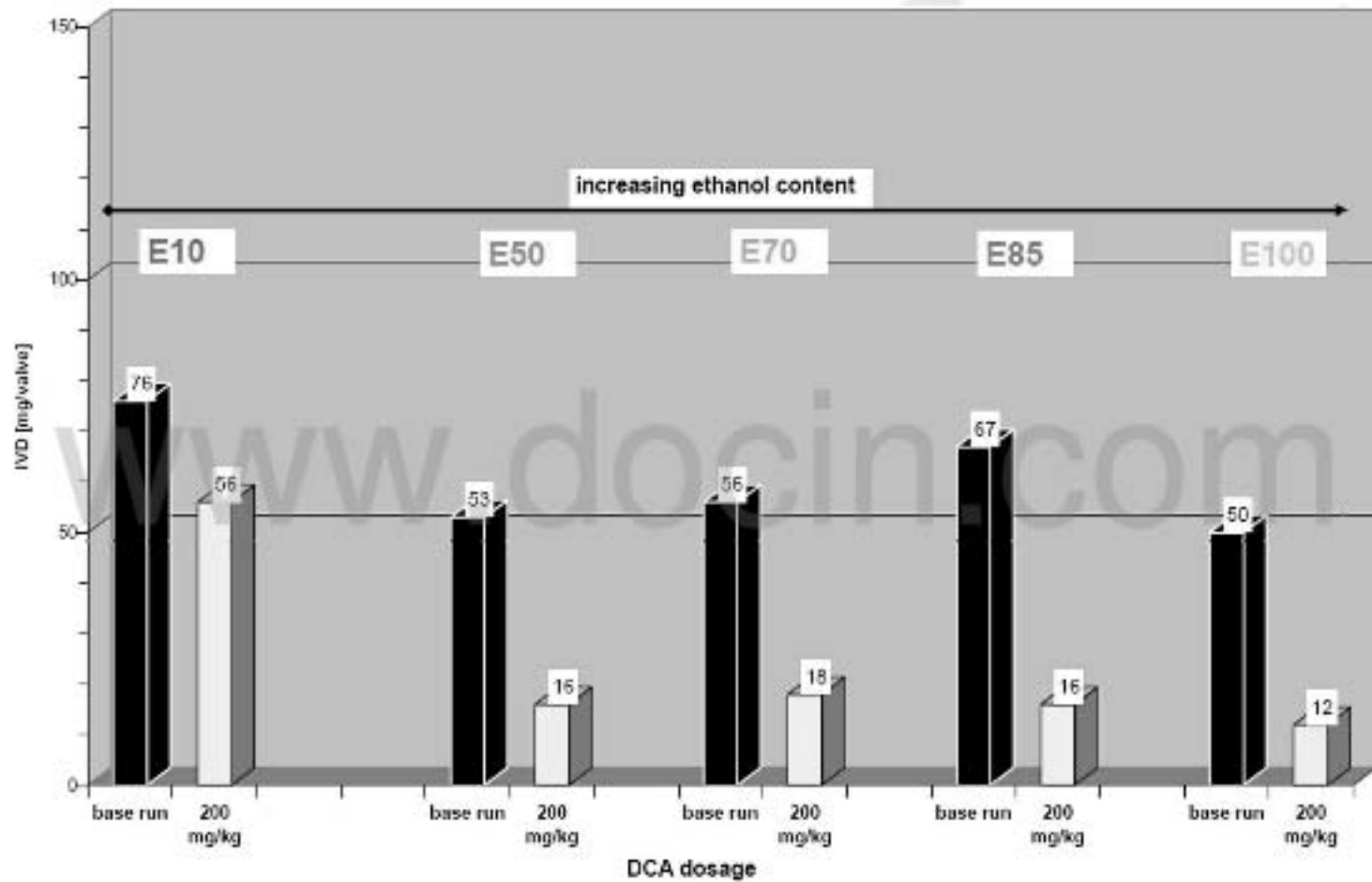


Figure 21. IVD with increasing ethanol content from E10 to E100. Matching pairs of test results with the same ethanol content are shown: without DCA (in black/dark, base runs) and with 200 mg/kg of DCA (in green/grey).

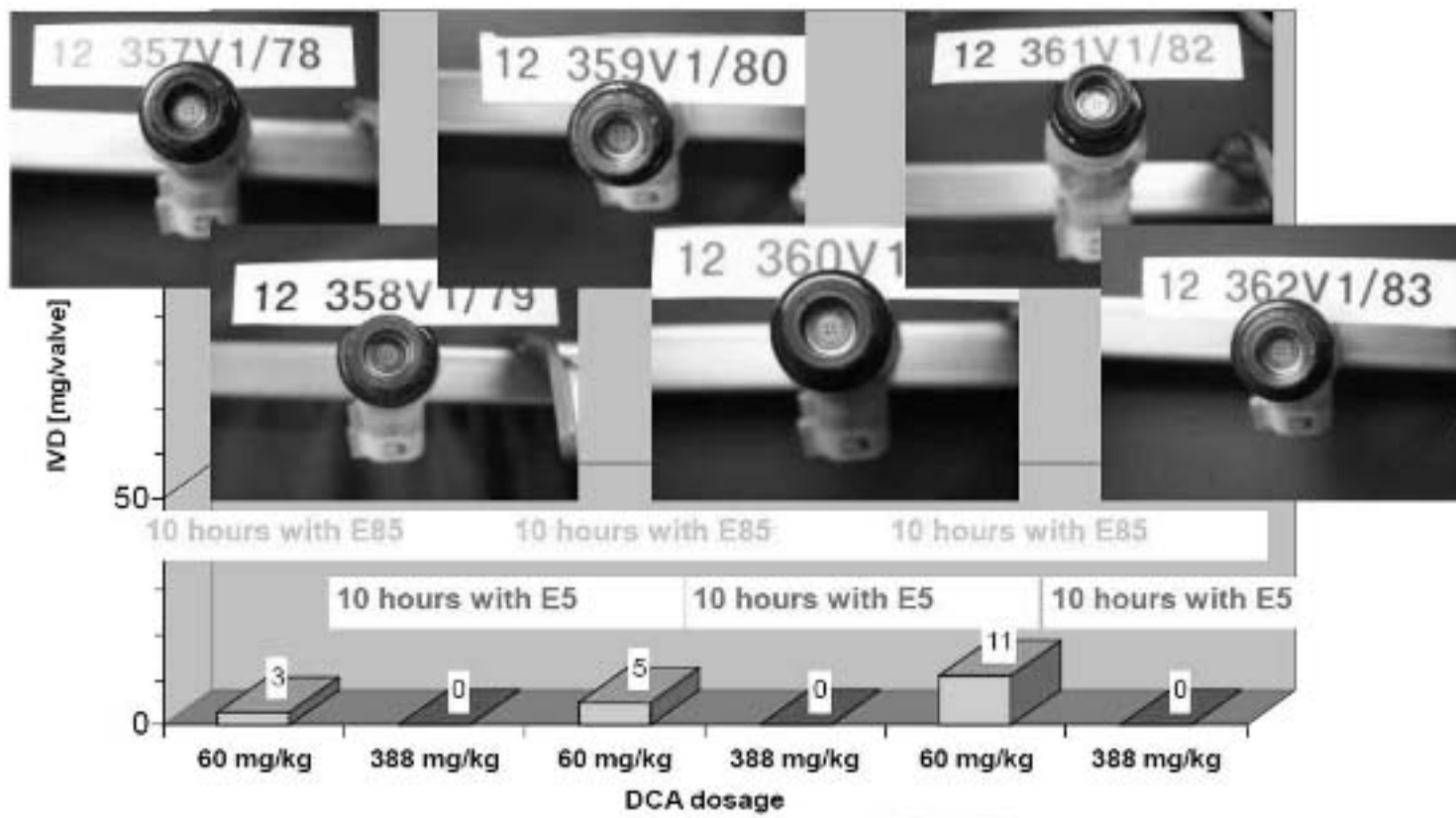


Figure 22. Switching between E85 (with 60 mg/kg DCA) and E5 (with 388 mg/kg of DCA) in 10 hours intervals. IVD and injector deposits after each interval.

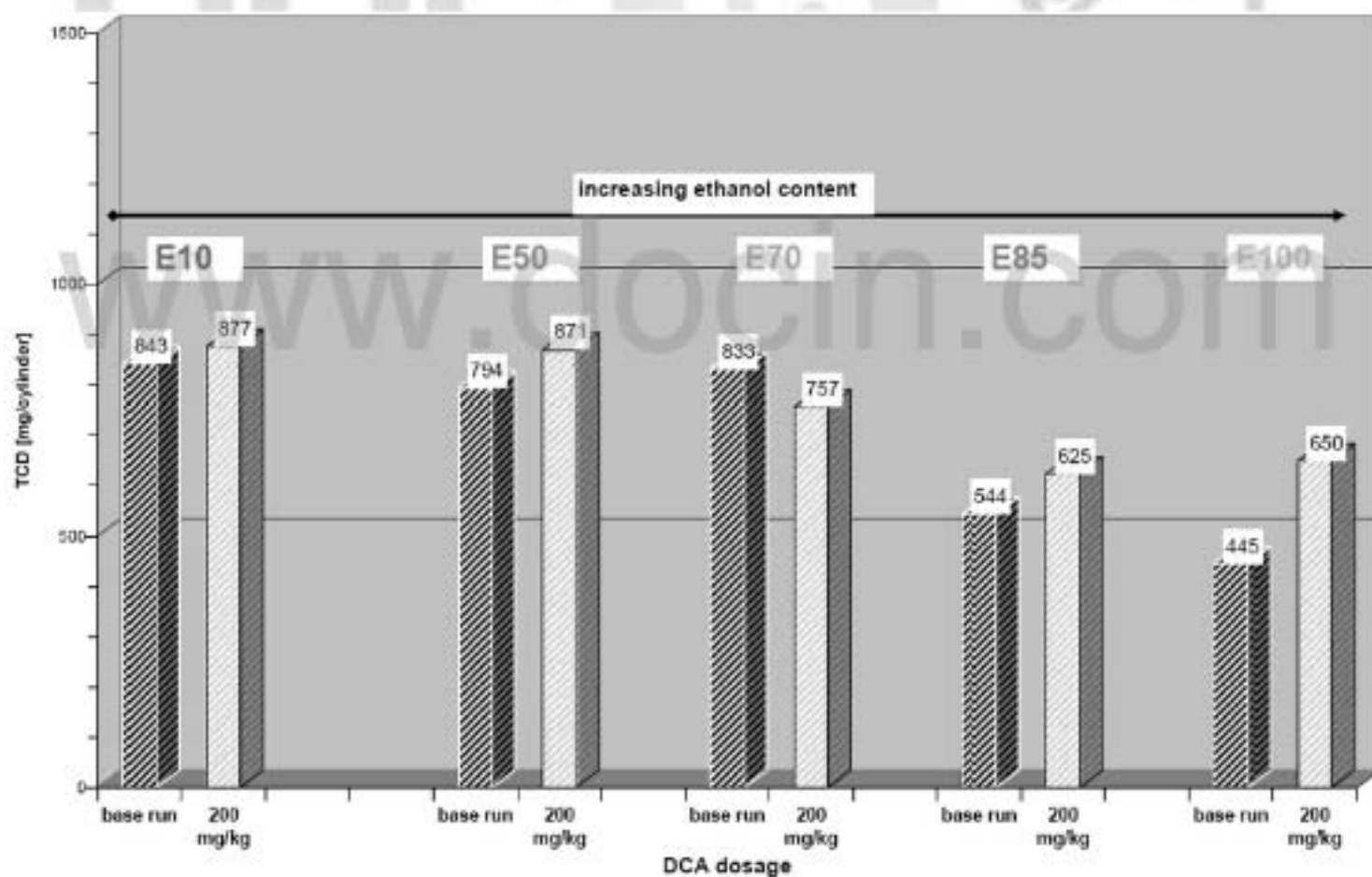


Figure 23. TCD with increasing ethanol content from E10 to E100. Matching pairs of test results with the same ethanol content are shown: without DCA (in black/dark, base runs) and with 200 mg/kg of DCA (in green/grey).

Filterability Tests of E85 with PIBA based Additive

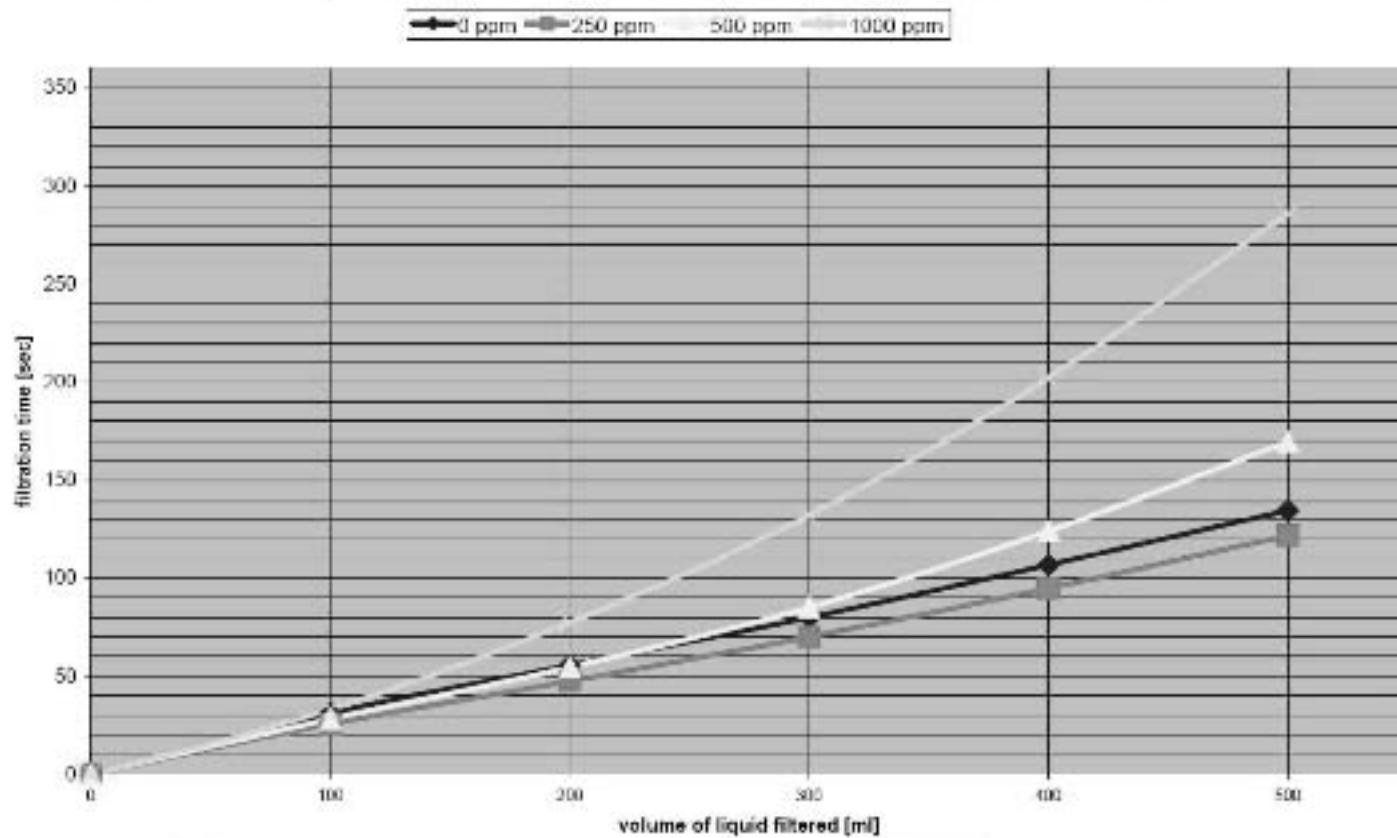


Figure 25. Modified SEDAB filtration test with E85. With 1000 ppm PIBA based DCA indication for filter plugging (upper curve). With 250 and 500 ppm PIBA based DCA (two middle curves) no large deviation from unadditized E85 (lower curve).

Filterability Tests of E85 with Ethanol Additive Package

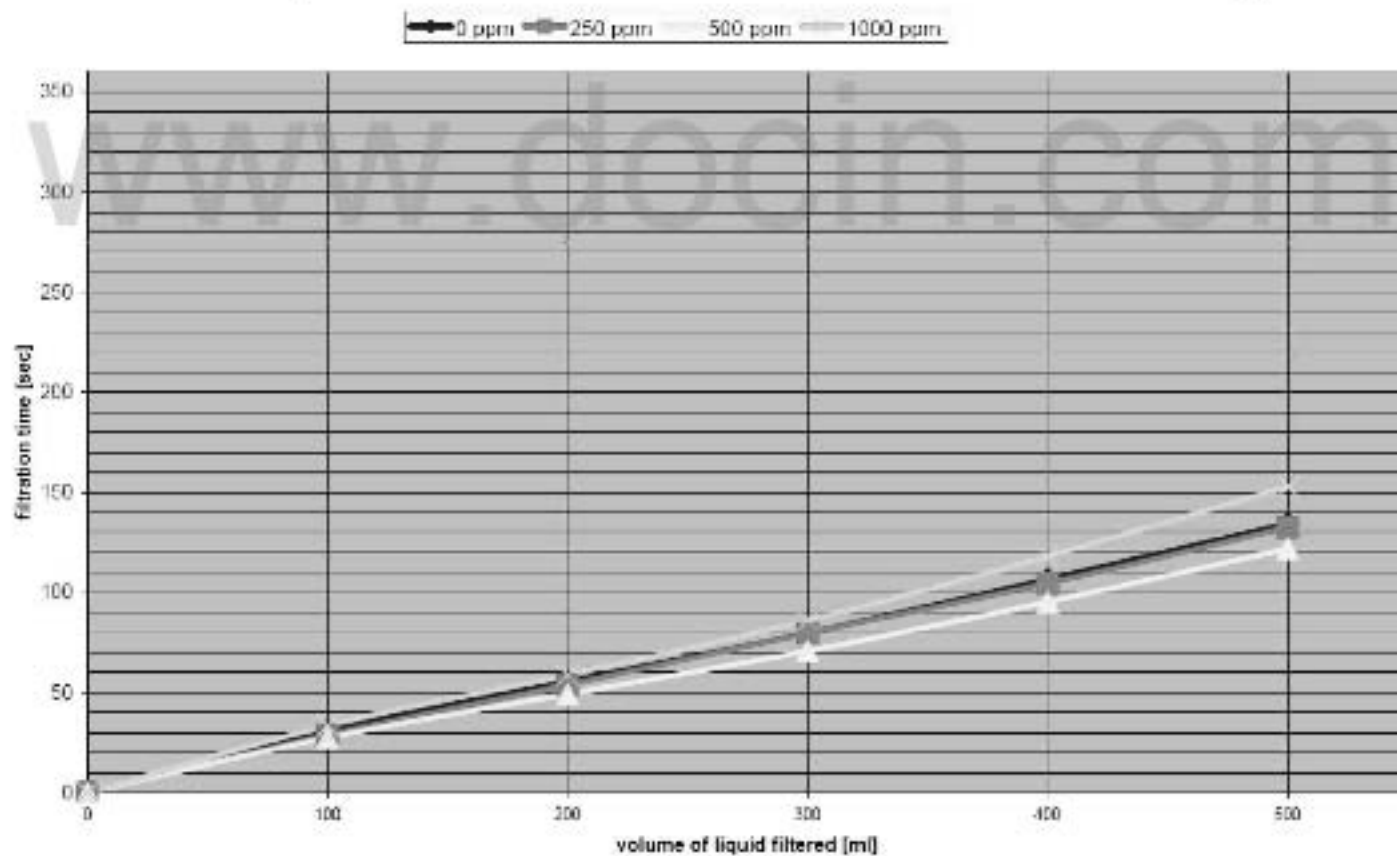


Figure 26. Modified SEDAB filtration test with E85. No indication for filter plugging with 250, 500 and 1000 ppm ethanol additive package.