

**PROTOCOL FOR THE EVALUATION OF  
EFFECTS OF METALLIC FUEL-ADDITIVES ON THE EMISSIONS  
PERFORMANCE OF VEHICLES**

*This test protocol is the outcome of an intense consultation with relevant stakeholders and it is based on the procedures prescribed by the new Euro5/6 regulation for durability demonstration.*

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## 2 LIST OF SPECIAL TERMS AND ABBREVIATIONS

AMA	US Automobile Manufacturers Association
CEC	Coordinating European Council
CO	Carbon Monoxide
CO <sub>2</sub>	Carbon Dioxide
CVS	Constant Volume Sampler
DF	Deterioration Factors
DPF	Diesel Particulate Filter
ECE	Economic Commission for Europe
ECE-cycle	Urban Driving Cycle (Part 1 of the NEDC driving cycle), aka "UDC"
EGR	Exhaust Gas Recirculation
ELR	European Load Response Test
EPEFE	European Programme on Emissions Fuels and Engine Technologies
ESC	European Steady State Cycle
ETC	European Transient Cycle
EU	European Union
EUDC	Extra Urban Driving Cycle (Part 2 of the NEDC driving cycle)
HC	Hydrocarbons
MIL	Malfunction Indicator Light
MTBE	Methyl Tert-Butyl Ether
NEDC	New European Driving Cycle
NO <sub>x</sub>	Nitrogen Oxides (NO, NO <sub>2</sub> )
PM	Particulate Matter
PN	Particle Number
SAS	Statistical Analysis Software
SBC	Standard Bench Cycle

SCR	Selective Catalytic Reduction
SRC	Standard Road Cycle
UN	United Nations
WHSC	World Harmonized Stationary Cycle
WHTC	World Harmonized Transient Cycle

### **3 SCOPE OF THE PROTOCOL FOR EVALUATION OF METALLIC FUEL- ADDITIVE EFFECTS ON EMISSIONS**

#### **3.1 Foreword**

This protocol is designed to evaluate two different aspects related to the use of metallic additives in fuels:

1. Short-term effects of metallic fuel additives on vehicle regulated and unregulated<sup>1</sup> emissions
2. Long-term effects of metallic fuel additives on the emissions performance of vehicles, so-called “durability” test

A first objective of the test protocol is to define a sound and repeatable way of measuring the effect of metallic fuel additives in fully formulated fuels on regulated exhaust emissions from vehicles in the European market. Regulated emissions include Hydrocarbons (HC), Carbon Monoxide (CO), Nitrogen Oxides (NO<sub>x</sub>), Particulate Matter (PM), Particle Number (PN) and Carbon Dioxide (CO<sub>2</sub>) measured using the methods prescribed by the applicable regulation. In addition, this test protocol requires also the measurement of the mass of the particle-bound metal(s) as produced by the combustion of the fuel containing metallic additive, the speciation of the combustion products using acknowledged analytical techniques and particle number.

Secondly, this protocol defines a test procedure to measure the long-term effects of metallic fuel additives on the emission performance of the vehicle's engine and emission control system.

For the purpose of this protocol the term “additive” means any material added to a fuel for general market distribution at a level of no more than 1% volume.

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#### **3.2 Scope**

**As required by Directive 2009/30/EC of the European Parliament and of the Council, the European Commission shall conduct an assessment of the risks for health and the environment from the use of metallic additives in fuel and, for this purpose, develop a test methodology.**

**However, compliance with this protocol does not absolve the fuel/additive supplier from ensuring that the use of metallic additives in fuels is in compliance with the**

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<sup>1</sup> For the purpose of this test protocol the term “unregulated” emissions is limited to the mass of particle bound metal(s) emitted as a consequence of the use of the metallic additive.

**European Union policy of vehicle performance standards and it has no adverse environmental or health effects.**

The scope of this protocol is threefold:

- i. evaluate the short term effects on regulated emissions of a candidate metallic additive in a fully formulated fuel;*
- ii. generate data on the emissions of the particle-bound metal(s) emitted as a consequence of the combustion of the fuel containing the metallic additive and on the speciation of the combustion products;*
- iii. demonstrate that there are detrimental effects of the metallic additive on long term emission performance of the vehicle's engine and emission control system.*

**The principle of this protocol's evaluation is that the emissions using the fuel containing metallic additive are compared with emissions from the same fuel without additive.**

**A testing programme carried out according to this test protocol can be expected to produce varying results on different vehicles. Therefore the results shall be analysed for statistically significant effects, and a detailed report shall be produced. Engineering judgement of significant effects can then be made by the appropriate experts leading to a report based on all of the results.**

**Metallic additives added to the fuel inside the tank by means of automatic dosing systems, as used in order to promote the regeneration of particulate filters, are *not* included in the scope of this test protocol.**

### **3.3 Project management aspects**

**An advisory board is to be established to assist in and advise during the preparation and development of the test. The advisory board is nominated by COM and JRC and it is expected to be an impartial, representative, objective group separated from the sponsor, project manager and the expert group. Its key role is to ensure that the test adheres to the protocol's requirements and to decide on any request to deviate from the test protocol methodology. Deviations from the test methodology must be justified and limited as far as possible; however some degree of flexibility is to be foreseen in case feasibility issues arise. Special attention shall be given by the advisory board in case the selection of fleet includes very small car that might have issues concerning the SRC cycle.**

**The advisory board has also the role to propose the candidates for the expert group. An expert group is to be gathered to assess the statistical relevance of the data gathered through out the test phase and to prepare the report summarizing the results. The expert group is nominated and stewarded by COM and JRC.**

**While the project management will be responsibility of the sponsors, the advisory body will have to monitor any test programs and insure independent data analysis.**

### **3.4 Scope of applicability**

The current version of this test protocol is mainly designed for light duty vehicles (LDV) and, notably the part addressing the long-term effects on the vehicle's emissions performance, is not applicable to heavy duty vehicles (HDV) for various reasons. Firstly, in the current European legislation, the durability demonstration procedure for heavy duty vehicles is based on either assigned deterioration factors or on the use of extrapolated deterioration factors calculated from emission data measured over a fraction of the useful life of the vehicle. This approach cannot be used to evaluate metallic additives since there is no proof that the effect of additive on emission control technologies is linear and no extrapolation of emission data generated over a fraction of the vehicle's useful life is allowed.

Secondly, given the considerable useful life of heavy duty vehicles as defined in relevant legislation (160000 km, 300000 km or 700000 km depending on the category which the vehicle belongs to), the efforts required to demonstrate that a metallic additive has no harmful effects on their emission control systems would lead to disproportionate costs, especially considering the limited number of vehicles of HDV compared to LDV.

Thirdly, to the knowledge of the Commission no metallic additive in diesel fuel is in current use for HDV (excluding the fuel-born catalysts used to promote the regeneration of DPFs). Actually combustion improver metallic additives added to the fuel with the purpose of reducing particulate emissions are used in some captive fleets (especially for passenger transportation) where the share of old technology vehicles is still high. However it is very likely that these metallic additives will not be used in vehicles equipped with advanced after-treatment systems and specifically with diesel particulate filters.

Further, as the after treatment technologies for LDV and HDV are converging, results from tests on LDV may be extrapolated – to a certain degree – in order to gain reasonable assumptions of the effects on emission control systems on HDV when using of metallic fuel additives.

Once a suitable test procedure has been demonstrated, HDV will be included in the scope of the test protocol.

Nevertheless, heavy duty engines can be tested using the fuel containing metallic additive to complement LDV data on short term effects on emissions. In this respect this test protocol does indeed contain a section dedicated to heavy duty vehicles (see Appendix 4).

## **4 PROTOCOL FOR THE EVALUATION OF METALLIC FUEL ADDITIVE EFFECTS ON VEHICLE EMISSIONS**

The test procedures and protocols have been based on the well-established EPEFE<sup>1</sup> methods, simplified and modified where appropriate, in order to meet the needs of this specific Protocol. The initial draft was prepared by the Special fuels/Vehicles/Engines Industry technical Expert in 2002 and then modified under the auspices of MVEG. The protocol has then been modified based on the procedure prescribed by the new Euro 5/6 Regulation for durability demonstration.

When followed carefully by a qualified laboratory, these procedures should assure sound test data and statistically valid interpretation of effects, so that the emissions performance of a candidate additive-treated fuel can be accurately assessed in a vehicle or engine test fleet. It is recognised that the specified tests involve a substantial effort to quantify the emissions performance of an additive. This is inherent in the nature of the subject - attempts to reduce the number of tests or vehicle fleet size will, according to today's experiences, lead to unreliable results.

It should also be noted that fuel effects on emissions can be complex. A key finding of the EPEFE study was that the same fuel change can have different effects in different vehicles. Selection of a sufficient number and representative test vehicles is therefore important to obtain valid results. Extrapolation of the test fleet results to the general vehicle population inevitably introduces some uncertainty.

In addition to the regulated emissions, this test protocol aims at generating data on the amount of the metal(s) emitted as a consequence of its combustion and on the chemical constitution in which the metal is emitted (speciation).

Currently there is no standard or legislative procedure to chemically/physically characterize particle-bound metal(s) emitted by combustion engines. However there are many studies publicly available describing different methodologies or techniques that can be used to investigate different aspects of the combustion products of ash-forming additives (particularly mass and speciation). This test protocol contains a short description of the main techniques.

<sup>1</sup> European Programme on Emissions, Fuels and Engine Technologies (published by the European motor and oil industry associations, ACEA & Europaia)

## 4.1 Test Fleet Selection

The number of vehicles or engines needed to achieve the objectives defined in the scope of the test protocol depends on three necessities:

- (i) the number of vehicles or engines needed to assure that the results are representative of the technologies available on the European market
- (ii) the fleet size needed to obtain statistical significance for the measured emission effect , and
- (iii) the characteristics of the road cycle used for the aging procedure

All the above criteria should be satisfied, so the total number of test vehicles or engines should be the higher than those required by the two criteria considered separately and they shall take into account the characteristics of the road cycle.

A test programme to investigate the long-term effects of the metallic fuel additive on emission control systems is extremely expensive and time consuming since two identical test fleets (hereinafter “no-additive fleet” and “additive fleet”) have to be aged for 160000 km with the very same procedure but with different fuels (without and with the additive respectively). Moreover, the test fleets should not only comprise different vehicle models to cover the main engine and after-treatment technologies, but more vehicles per each of the selected models should be tested in order to reduce the risks of invalid results in case of major breakdowns or accidents that could occur during the mileage accumulation to one or more vehicles. In order to keep the time needed to complete the test programme and its cost within reasonable margins, it is therefore necessary to search for a compromise on the number of vehicles to be tested.

It is recommended that the fleet is selected according to the following guidelines:

- **The chosen vehicles must be new and representative of those vehicles in the European market where the additive treated fuel may be applied**
- **At least four different models should be selected, each covering a specific technology or range of technologies. The technologies to be taken into consideration must be those that will be widely used in future vehicle generations available in the EU. In addition, as far as the after-treatment devices are concerned, the technologies that are potentially more sensitive to the effects of the metallic additive should be selected (e.g. high cell density catalysts). A non-exhaustive list of current technologies that should be covered as much as possible by the selected models is given in the following table.**

<u>Petrol vehicles</u>	<u>Diesel vehicles</u>
Port fuel injection engines	High pressure injection systems
Direct injection engines (lean and stoichiometric)	Diesel particulate filters
Turbo-charged engines	Lean NOx traps
Three way catalyst (>600 cpsi )	Selective Catalytic Reduction (SCR)
Lean NOx traps	Diesel Oxidation Catalyst (DOC°)
Particle Filters (fitted as a separate device or included within a four-way catalyst)	

- **At least one of the models selected shall fall in each of the three following categories based on engine size: Small (range up to 1.4 litres), medium (range from > 1.4 to < 2.0 litres) and large (range from >2.0 litres).**
- **Per each model, six different vehicles shall have to be tested and aged. Three vehicles will be tested and aged with the untreated fuel, while the other three vehicles will be tested and aged with the additive-treated fuel. It is essential to ensure that the individual vehicles of the same model match in all aspects relevant to emissions i.e., engine type, transmission, emission control technology, catalyst type, model year, tyre size/type, etc.**

By strictly following the above guidelines, the test fleet will comprise 24 individual vehicles that will be tested and aged as described in the next chapters. However some additional flexibility in the selection of the test fleet could be considered by the advisory board if the fleet is composed only of Euro 5 and 6 vehicles (for reference year 2011/2012) or future performance standards applied at EU level. Since this test protocol aims not only at investigating the long term effects of the metallic additive on the efficiency of emission control technologies, but also at generating emissions data for a more comprehensive risk assessment, it is recommended that other vehicle models different from those selected for the durability demonstration are tested with and without the metallic additive. These additional tests, not followed by the vehicle aging, have the objective of generating additional data on the instantaneous effect of the additive on regulated and unregulated emissions.

In the case of metallic additives intended for diesel fuel may arrive to the EU market, the test fleet may be complemented by some heavy duty diesel engines in order to generate data on the instantaneous effects of the additive on their exhaust emissions performance.



## 4.2 Test Fuel and Metallic Additive

### 4.2.1 Base or Reference fuel

The principle of additive evaluation is that the emissions using the fuel containing additive are compared with emissions on the same fuel without additive. This base or reference fuel should be chosen to be representative of fuels in the relevant market. For current European gasoline and diesel the fuel should meet the standard requirements of EN228 or EN590 *as appropriate to the relevant market*. The Coordinating European Council (CEC) reference fuels specified in the emission regulations meet this requirement, and should be used whenever possible.

Testing to establish emissions performance of an additive represents substantial effort and the fuels can vary from batch to batch, even when the specification is nominally the same. It is therefore important that a single batch of fuel is obtained from the same blender, sufficient in quantity to complete the entire test programme, i.e., the instantaneous effects assessment and longer term durability test).

However, given the substantial amount of fuel needed (estimated to be in the range of 300000-350000 litres), it may be not feasible to obtain and store a single batch of fuel for the entire test programme. In this case it is of eminent importance that the fuel used in the tests is kept within strict margins of deviances and that each vehicle sees the same amount of each different fuel batch used. A viable solution to minimize fuel variability is to use CEC certified reference fuel batches purchased from the same supplier. Each single batch of CEC reference fuel supplied is then split into two parts that will be used for the “no-additive” fleet and the “additive fleet” for both mileage accumulation and emission tests. It is essential that the fuel is changed from a batch to another batch at the same time/same mileage for both the two test fleets. This possible approach is exemplified below:

Fuel	Batch 1	Batch 2	Batch 3	Batch 4
“No-additive” fleet	Run-in, initial emission tests and aging up to 30000 km	30000 – 80000 km aging and periodic emission tests	80000 – 120000 km aging and periodic emission tests	120000 – 160000 km aging, periodic/final emission tests
“Additive fleet”	Run-in, initial emission tests and aging up to 30000 km	30000 – 80000 km aging and periodic emission tests	80000 – 120000 km aging and periodic emission tests	120000 – 160000 km aging and periodic/final emission tests

The number of fuel batches needed to complete the test programme should be kept to the minimum feasible value.

A certificate of analysis shall be obtained for each batch of fuel used, and from a qualified laboratory certified to EN45000. The analysis shall include all the parameters specified in EN228 or EN590. This certificate of analysis must be submitted with the test results. In addition, a one litre sample of fuel must be retained for a period of one year after the submission of test results to allow re-checking and confirmation of fuel properties if necessary.

The reference fuel may contain additives essential for good fuel quality as typically used in the market e.g., antioxidant, diesel cold flow additive, lubricity additive.

It is recommended that a deposit control additive is used to avoid potential problems with inlet system deposit build-up during such a long duration test. The additive, preferable from the same batch, should be in use in Europe and used at the additive manufacturer's recommended treat-rate to achieve a 'protection' or 'keep-clean' level of performance. High performance premium 'clean-up' treat rates should not be used. The same additive and treat rate must be used in all mileage accumulation fuels, both with and without the metallic additive.

The Tester shall publish a complete table of components added to the fuel, together with detailing the use during the test; e.g. volume percentage and other relevant to ensure conformity with the test protocol and comparability of results data.

#### 4.2.2 Candidate additive

The candidate, metallic fuel-additive must only be splash blended into the fuel batch (off-vehicle), respectively batches of the reference fuel used for the test programme at a treat rate representative of that commercially used<sup>2</sup>.

- **Where a range of additive treat rate levels is envisaged, the level tested for instantaneous emission effects and the "durability" demonstration shall be considered to be the maximum treat rate for which no-harm has been demonstrated.**

Blending of the additive into the fuel must use methods that are practical in service. The blending method and treat rate must be documented and submitted with the test results. The certificate of analysis and batch number of the metallic additive must be submitted with the test results. Artificial methods to ensure that the additive is homogeneously dispersed in the test fuel during the test programme are not permitted.

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<sup>2</sup> For manganese, ACEA recommends a maximum rate of 6mg/l +/-0.5 mg/l tolerance.

### 4.3 Test Programme General Outline

The test programme is split into two main phases:

➤ Phase 1: Running-in and baseline emissions test

The purpose of this phase is to adequately prepare the vehicles before commencing the actual emission testing and vehicle aging in order to ensure that the catalyst and the engine combustion chamber deposits have stabilised.

After the running-in, the vehicles are tested for emissions:

- For the “No-additive fleet”: In order to establish a reliable base line for regulated emissions
- For the “Additive fleet”: In order to establish a reliable base line for regulated emissions and to later assess the instantaneous effect of the metallic additive on regulated and unregulated emissions

All the emission tests are carried out according to the relevant European type approval procedure. Each legislative test has to be immediately followed by a second emission test using a hot start “real-world” driving cycle. The vehicle shall be checked for OBD defaults (with/without MIL) in the OBD memory.

➤ Phase 2: Durability test procedure including periodic emission tests

This phase has the two-fold target of assessing the potential impact on emission control devices by the metallic additive and to generate additional data on unregulated emissions from these same additives.

Upon the completion of Phase 1 and validation of the baseline emissions results, the vehicle shall start Phase 2, i.e. the mileage accumulation phase using the procedure described in chapter 4.5.

At pre-defined intervals the vehicles shall be tested for emissions to check the evolution of the regulated pollutants against accumulated mileage as well as to generate additional data on unregulated emissions.

All the emission tests shall be carried out according to the relevant European type approval procedure. Each legislative test has to be immediately followed by a second emission test using a hot start “real-world” driving cycle.

The total test procedure is different for the two test fleets. A summary of the complete test procedure is given below:

- For vehicles aged without use of metallic additive (only the untreated fuel has to be used for all the operations)
  - Running-in (3000 km)
  - Four repetitions (base line) before starting the mileage accumulation
  - Mileage accumulation using the standard road cycle (SRC)
  - Every 30000 km up to 160000 km included: at least two back-to-back emissions tests including the legislative cycle + real-world cycle
  - Check and record any OBD faults using service \$06
  
- Vehicles aged with use of metallic additive
  - Running-in (3000 km on untreated fuel)
  - Four repetitions (base line) before starting the mileage accumulation (untreated fuel)
  - Use the test protocol for instantaneous effects to assess the impact of the metallic additive on regulated and unregulated emissions (both untreated and additized fuel)
  - Mileage accumulation using the SRC cycle
  - Every 30000 km up to 160000 km included: at least two back-to-back emissions tests including the legislative cycle + real-world cycle using the additized fuel followed by other two emission tests (legislative cycle+ real-world cycle) using the untreated fuel  
If needed, additional PM samples should be taken in order to characterize metal emissions.
  - Check and record any OBD faults using service \$06

## 4.4 Phase 1: Running-in of the Vehicles and Initial Emission Tests

### 4.4.1 Phase 1: Test Vehicle/Engine Preparation

**Before commencing the test programme to evaluate a fuel containing additive, the emissions performance of the vehicle or engine must be measured and confirmed to meet the limits for which the vehicle/engine was certified. *For this purpose, the test procedure and reference fuel appropriate to the certification should be used.***

#### 4.4.1.1 Light Duty Vehicles

Vehicles are to be prepared in strict accordance with the European Union (EU) procedures to which the vehicle was originally type approved (Refer to Appendix 1) with the following additional requirements:

1. The new vehicles selected for the test programme must have completed at least 3000 km mileage accumulation prior to testing them for emissions to ensure that the catalyst(s) and the engine combustion chamber/injector deposits have stabilised. The selected test fuel (or its first batch) without the metallic additive shall be used for the running-in phase for all the vehicles. The initial mileage accumulation may be carried out on the road or on a mileage accumulation dynamometer. No specific driving cycle is prescribed by the current legislation for the running-in phase but the use of the Standard Road Cycle is highly recommended. In any case the driving cycle used must be representative of road conditions appropriate to the expected use of the candidate additive treated fuel. If the vehicle has been left stationary for an extended period of time (more than 5 days) it shall be driven at least 100 km immediately before the start of an emissions test sequence. The same running-in procedure is used for all test vehicles.
2. In case of additional vehicles taken from the road and tested in order to expand the emission data set, they must be presented in good mechanical condition and must have completed at least 8000 km prior to testing them for emissions to ensure that the catalyst and the engine combustion chamber deposits have stabilised. The last 1000 km of this 8000 km shall be conducted with the selected test fuel (or its first batch), in any case without the metallic additive. The 1000 km mileage accumulation may be carried out on the road or on a mileage accumulation dynamometer. The use of the Standard Road Cycle is highly recommended. If the vehicle has been left stationary for an extended period of time (more than 5 days) it shall be driven at least 100 km immediately before the start of an emissions test sequence.
3. The fuel tank must be modified to accept a drain valve to allow the tank to be completely emptied between test fuels. Where it is not possible to fit a drain valve in the bottom of the fuel tank due to safety reasons, it is recommended that the fuel pump/fuel tank sender module is modified to accept an additional pipe, located so as to allow the tank to be drained by means of a separate suction pump or an equivalent procedure to ensure the fuel is completely drained. Alternative fuel handling procedures may be used, but advice from the vehicle's manufacturer is necessary and any intervention must be authorized by the manufacturer.
4. The setting of the engine and of the vehicle's controls shall be those prescribed by the manufacturer and shall be checked, adjusted and recorded immediately prior to testing. No further adjustments are permitted during the test programme. If downtimes of >30 days occur during the test programme, rechecking of the vehicle settings is mandatory.
5. Check and record any OBD faults using service \$06
6. Change the engine oil and filter according to the manufacturer's recommendations. The oil must then be aged by driving a minimum of 1000 km on the road or on a mileage accumulation dynamometer before any emissions test. The engine oil should be that recommended by the vehicle manufacturer, and appropriate for normal vehicle

service. The same manufacturer-recommended engine oil should be used in all six vehicles of the same model.

7. Install a new air filter at the intervals recommended by the manufacturer and before any emission test sequence.
8. Check and adjust cold tyre pressures to 3.5 bar before any emission test sequence. The same set of wheels and tyres must be used throughout the test programme on the dynamometer, others must be used for any mileage accumulation on the road.
9. The evaporative emissions system must remain connected and functioning throughout the test programme. The canister may, however, be relocated to a more convenient position to allow easy access for purging and weighing.
10. All emission control systems (Three Way Catalyst (TWC), Exhaust Gas Recirculation (EGR), Diesel Particulate Filter (DPF), Selective Catalytic Reduction (SCR), etc.) must remain intact and in the same configuration and functioning throughout the test programme.
11. In case of a vehicle equipped with periodically regenerating devices, the test procedure to be followed is described in Annex 13 of the UN-ECE Regulation 83. If the vehicle is fitted with a DPF, an appropriate procedure has to be adopted to ensure that the vehicle is tested with the DPF in similar conditions in terms of fill state. In this case advice from the manufacturer is necessary.
12. Ensure the dynamometer load and inertia settings are set accurately to provide the correct run down characteristics. It is recommended that periodic checks (at least weekly) are carried out throughout the programme to ensure consistent dynamometer performance. Variations of 5% or more in vehicle run down characteristics must be corrected and recorded.
13. The test equipment must be in accordance with Annex III of Directive 70/220/EEC [1] as modified by its subsequent amendments. All calibrations shall be conducted prior to and during the test programme according to the provisions of Annex III and the test laboratories' internal quality assurance system.
14. The calibration reports shall be filed at the test laboratory for a period of 6 months after the end of the test programme, and shall be available for inspection upon request.

#### **4.4.2 Diesel Heavy Duty Engines**

The engines are to be prepared in strict accordance with the EU procedures to which the engine was originally typed approved (Refer to Appendix 1), with the following additional requirements:

1. The test engine shall be presented in good mechanical condition and must have completed the running-in procedure prescribed by the manufacturer.

2. The exhaust system may be a chassis type or test cell system, provided it is representative of the road vehicle system.
3. A chassis type or test cell type inter-cooler may be used. The test cell type inter-cooler (typically water-to-air system) shall be so designed as to produce temperature profiles and temperature differences similar to the chassis type.
4. The setting of the engine and of the vehicle's controls shall be those prescribed by the manufacturer and should be checked, adjusted and recorded immediately prior to testing. **No further adjustments are permitted during the test programme.** If downtimes of >30 days occur during the test programme, rechecking of the vehicle settings is mandatory. Change the engine oil and filter. Prior to the test programme, the oil must be aged by running the engine between 10 and 20 hours at rated speed and load. The engine oil should be that recommended by the engine manufacturer and appropriate for normal engine service. The test equipment must be in accordance with Annex III of Directive 88/77/EEC as modified by its subsequent amendments. All calibrations shall be conducted prior to and during the test programme according to the provisions of Annex III and the test laboratories' internal quality assurance system. The calibration reports shall be filed at the test laboratory for a period of 6 months after the end of the test programme, and shall be available for inspection upon request. All emission control systems (EGR etc.) must remain intact, in the same configuration and functioning throughout the test programme.

#### **4.4.3 Phase 1: Establishing the Regulated Emission Base Line**

Before commencing the mileage accumulation for investigating the effect of a metallic additive on emission control technologies, the base line levels of the regulated emissions have to be established.

Upon the completion of the running-in phase and after having prepared the vehicles according to the Section 4.4.1 all the vehicles are tested for emissions following the procedure prescribed by the European type approval emission legislation (Directive 70/220/EEC and subsequent amendments or UN-ECE Regulation 83).

The selected test fuel without the metallic additive must be used for these emissions tests.

At least four emissions tests should be repeated following the daily schedule described in section 4.4.5 in order to obtain a reliable base line.

#### **4.4.4 Phase 1: Test Design for Instantaneous Effect from Metallic Additives on Regulated and Unregulated Emissions**

The vehicles belonging to the "additive test fleet" will be also tested with the treated fuel containing the candidate additive following the procedure described below. The four emissions tests carried out to establish the base line can be used as initial tests for the test sequence described in the rest of the paragraph.

Statistical considerations dictate that multiple tests are needed to establish the single-case variances and ultimately the significance of observed emissions effects. Such multiple tests are needed for each fuel on each vehicle or engine, with more than one vehicle or engine to be tested. The test outline for each individual vehicle/engine indexed 1, 2, ... is

(i)            A<sub>1</sub>A<sub>1</sub>    B<sub>1</sub>B<sub>1</sub>    A<sub>2</sub>A<sub>2</sub>    B<sub>2</sub>B<sub>2</sub>  
or (ii)        B<sub>1</sub>B<sub>1</sub>    A<sub>1</sub>A<sub>1</sub>    B<sub>2</sub>B<sub>2</sub>    A<sub>2</sub>A<sub>2</sub>

Each letter represents a single test on the Additive treated fuel ("A") or Base fuel ("B"). Short-term repeats (A<sub>1</sub>A<sub>1</sub> etc) are included to ensure an absolute minimum of variance control. Where there is a significant difference between the two results in a pair, a third test must be performed before proceeding to the next fuel. The criteria for deciding if additional testing is needed are explained in Section 4.4.5.4. Variations between blocks of tests on the same fuel, but different vehicles/engines (e.g., A<sub>1</sub>A<sub>1</sub> varying from A<sub>2</sub>A<sub>2</sub>, etc) provide the measure of test variability that is normalised in later statistical analysis, thus permitting some basic judgement about statistical significance of the test programmes' results. Once again, in the event that large variability is seen between the two pairs (blocks) of tests, a third block may be needed. The criteria for deciding if additional testing is needed are explained in Section 4.4.5.4.

Statistical evaluation of the earlier EPEFE data showed that most of the variability is to be expected between vehicles rather than in tests on a single vehicle. Therefore, little is gained by carrying out further repeat tests on the same vehicle - rather additional vehicles need to be tested. As an additional guarantee of balance in the results, some vehicles should be tested in order (i), some in order (ii).

Strict adherence to the vehicle preparation and testing requirements outlined in Sections 4.4 and 4.4.5 is essential to produce consistent and reliable data. It is an important principle that each test must be performed in exactly the same way with regard to both the testing and the preparation of the vehicle beforehand, to ensure that spurious factors do not affect the quality of the results. Accurate and traceable reporting is also essential; the requirements for data reporting and interpretation are outlined in Section 4.4.6.

Alternative test designs may be considered provided they provide the same technical rigour. For example, additional repeat tests on the same vehicle may be performed, or additional provisions made for tests on additives that may have a carry-over or 'memory' effect. Statistically sound designs, which test more than one additive and/or different concentration levels of additives in a particular reference fuel, may also be used.

## 4.4.5 Phase 1: Daily Test Schedule

### 4.4.5.1 Test Outline

For each emissions test, it is important that the vehicle or engine is presented and prepared in exactly the same way so that a true comparison of fuel effects is obtained. The order of steps is

1. Change test fuel (if needed)
2. Condition vehicle/engine
3. Cold soak (for light duty tests)
4. Emissions test

All these steps must be performed in a controlled and repeatable manner.

### 4.4.5.2 Regulated and Unregulated Emissions

The following regulated pollutants have to be measured in each emission test according to the relevant European type approval procedure:

- Carbon monoxide (CO)
- Un-burnt hydrocarbons (total and non-methane hydrocarbons)
- Nitrogen oxides (NO<sub>x</sub>)
- Particulate mass (PM) (Including port fuel injection vehicles using the same legislative procedure as for direct injection vehicles)
- Particle number (PN)
- Carbon dioxide (CO<sub>2</sub>)

In addition to what is prescribed by the type approval procedure, the following unregulated emissions should be measured:

- Mass of particle-bound metal(s) emitted from the vehicle's tailpipe as a consequence of the combustion of the fuel containing metallic additive
- Speciation of the combustion products of the metal(s) contained in the vehicle's exhaust emissions is also encouraged if the results can be reliably and routinely measured on the combustion products. The project team should consider the available techniques and recommend analytical measurements to the advisory board, taking into account information that will be beneficial to health-related studies.
- Distribution of particle number

For this purpose, appropriate, acknowledged methods should be used (e.g. ICP-MS; PIXE; XAS; ESCA-XPS, etc.). Any method selected should be accompanied by an explanatory note.

Some of the techniques mentioned above require special filter media not compatible with the filters prescribed by the current legislative procedure for particulate mass determination (e.g. quartz; PTFE; polycarbonate; etc.). This may require the collection of additional samples during the emission tests. In general this can be easily accomplished by using additional sampling probes that are very often available in emission test facilities.

A short description of the most used techniques, best practice and the requirements in terms of filters is given in Appendix...

It is essential that all the analyses for the determination of the mass of metal emitted and/or for the speciation of the combustion products are carried out by one laboratory to ensure the comparability of the results.

#### 4.4.5.3 Test Cycles

Light duty gasoline and diesel vehicles must be tested according to the EU Procedures to which the vehicles were originally typed approved. Refer to Appendix 1. Detailed additional instructions are in Appendix 3.

Upon the completion of the emission test performed according to the type approval procedure, a second emission test has to be carried out using a hot start “real-world” driving cycle in order to obtain emission data more representative of the in-use emissions. The time interval between the first and the second test depends on the time needed to the operator to prepare the test cell (analysis of the emission, reset and preparation of the equipment, calibration of analysers, etc.) but it should be kept constant as far as possible.

Examples of hot start driving cycles that can be used for this purpose are the US06<sup>3</sup> complementary driving cycle, representative of aggressive highway driving conditions, or the Artemis<sup>4</sup> driving cycle.

#### 4.4.5.4 Criteria for Additional Repeat Testing

To assure the integrity of the emissions data each pair of back to back tests must be compared to ensure that they fall within the expected test error range. If they do not fall within this range a third repeat test should be run before proceeding to the next stage after the second of the back to back tests. The data must be reported for all three tests.

After completion of the second block of testing on the same fuel, a comparison between the mean of the first block and that of the second must be made. If these two mean values

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<sup>3</sup> US06 Supplemental FTP Driving Schedule, <http://www.epa.gov/oms/sftp.htm#nprm>

<sup>4</sup> Real-world driving cycles for measuring cars pollutant emissions – Part A: The ARTEMIS European driving cycles  
ARTEMIS - Assessment and reliability of transport emission models and inventory systems  
Report INRETS-LTE 0411 June 2004

do not fall within the expected error range, a third pair of back to back tests must be conducted.

Criteria for determining if a repeat test is required are applied by comparing the ratio of the two test results for each emission (larger/smaller) with the tabulated data below. If the ratio is greater a repeat test must be conducted.

Light Duty <sup>1</sup> Gasoline	HC	CO	NO <sub>x</sub>
Back to back	1.28	1.26	1.32
Block to block	1.33	1.29	1.36

Light Duty Diesel <sup>1</sup>	HC	CO	NO <sub>x</sub>	PM
Back to back	1.40	1.32	1.09	1.28
Block to block	1.49	1.41	1.11	1.29

Heavy Duty Diesel <sup>1,2</sup>	HC	CO	NO <sub>x</sub>	PM
Back to back	1.12	1.09	1.02	1.10
Block to block	1.14	1.17	1.04	1.14

<sup>1</sup> The acceptable level of variability between tests has been established based on the variability seen in the EPEFE test programme.

<sup>2</sup> The tabulated data for heavy-duty diesel were developed for stationary cycles and for engines without after-treatment systems. They will be revised when more experience has been gained with the transient test procedure and new engine and after-treatment technologies.

#### 4.4.6 Phase 1: Test Data Reporting

##### 4.4.6.1 Test Results

A test report must be submitted for each individual emissions test using a format substantially similar to and containing all the information shown in Appendix 5 for Light Duty Vehicles and Appendix 6 for Heavy Duty Engines.

##### 4.4.6.2 Test Result Summary

A summary table must be provided showing the emissions results for the whole programme, in the format given in Appendix 7. Results must be presented for the regulated pollutants HC, CO, NO<sub>x</sub>, PM, PN (where applicable) and for CO<sub>2</sub>.

##### 4.4.6.3 Calculation of Measured Emissions Effect

The test results will have to be professionally processed and analysed by using acknowledged statistical methods in order to identify the statistically significant effects which can be attributed to the metallic additive. The following guidelines based on the EPEFE programme are given as reference.

For each pollutant, the effect of an additive on emissions will normally be calculated by comparing the geometric (logarithmic) mean of the emissions on the additivated fuel with

the geometric mean emissions on the base fuel. The results from each vehicle or engine should be combined to give an overall estimate of the effect of the additive across the test fleet. The necessary calculations are explained in Section 4.4.6.4.

Where additional repeat tests have been required (see Section 4.4.5.4), test results should be rejected only where there is engineering or strong statistical evidence (e.g. from a statistical outlier test) to suggest that a result is invalid (see Appendix 9). In all other cases the result should be retained and included in the average. The geometric mean figures for each vehicle should be included in the test result summary (Appendix 8).

A one-sided *t*-test should be used to determine whether the reduction in emissions is statistically significant. This is illustrated in Appendix 9. Appendix 9 also shows how a lower 95% confidence bound (one-sided) may be derived for the true reduction in emissions.

#### 4.4.6.4 Methodology for Calculating Emissions Effect and Statistical Significance

Data collected under this protocol require careful statistical analysis and it is recommended that this be conducted by a statistician.

This Section gives details of a quick analysis, which can be used to determine the average emissions for the various fuels under test and their likely significance. A spreadsheet illustrating these calculations is given in Appendix 8. More detailed guidelines vis-à-vis the steps which must be followed in a fuller and correct statistical analysis are explained in Appendix 9.

##### **Quick analysis**

The basic steps needed to calculate the percentage reduction in emissions for a test fuel A relative to a reference fuel B from the results of a test programme conducted following the experimental design in Section 4.4.4 are listed below. It is assumed implicitly that the variations in emissions measurements follow the log-normal distribution (standard deviation proportional to the mean) and therefore geometric (logarithmic) means are used instead of arithmetic means and reductions are expressed on a percentage basis.

1. Perform the log transformation  $y = \ln(x)$  for each emission measurement  $x$ .
2. Calculate the average value of  $y$  for each set of back-to-back tests (usually this will be the mean of two values, but occasionally there may be 1 or 3).
3. Calculate the average of the back-to-back averages computed in step 2 for each vehicle  $\times$  fuel combination (usually this will be the mean of two values, but occasionally there may be 1 or 3). Give each back-to-back average equal weight irrespective of the number of tests involved in its computation.
4. Perform the inverse transformation  $x = \exp(y)$  to determine the geometric mean emissions for each vehicle  $\times$  fuel combination.
5. Calculate the percentage reduction in emissions between fuels for each vehicle from the appropriate geometric means.

6. Calculate the geometric mean emissions across vehicles and for each fuel by averaging the log-scale averages calculated in step 3; then perform the inverse transformation  $x = \exp(y)$ . Give each fuel and vehicle equal weight in the averaging process irrespective of the number of back-to-back tests and true repeats actually conducted.
7. Calculate the percentage reduction in fleet emissions between fuels from the appropriate geometric means calculated in step 6.

A spreadsheet illustrating these calculations is given in Appendix 8. Further calculations are given in Appendix 8 to determine whether fuel A gives significant reductions in emissions relative to fuel B. These calculations will be statistically exact if:

- exactly two pairs of back-to-back tests have been conducted on each fuel in each vehicle and there are no missing values or outliers and
- the variability in emissions measurements (log scale) does not vary from vehicle to vehicle (engine to engine).

The analysis will only be approximate if (a) a third back-to-back test, or a third pair of back-to-back tests have been conducted in accordance with the protocol for certain vehicle/fuel combinations and/or (b) certain values are missing or have been rejected as outliers.

## **4.5 Phase 2: Vehicle Aging and Periodic Emission Test**

### **4.5.1 Phase 2: Need for Additional Durability Test**

This protocol does not attempt to evaluate all aspects of additive behaviour necessary to assure effective and harm-free operation in the field - this remains the responsibility of the additive supplier.

However, where a metallic additive is used, it is important that possible short-term benefits are not obtained at the expense of longer-term deterioration of the vehicle's emission control system. This is particularly important for modern vehicles where low emissions are achieved with the help of sophisticated exhaust after-treatment devices. Test requirements have therefore been included in this protocol to address this question and are described in the following sections for light-duty vehicles. This part of the test protocol is also designed to generate data on unregulated emissions over the life of the vehicles when using the treated fuel.

As described in the scope (see Section 3.2) heavy duty vehicles are for the moment not included in this part of the protocol. Regulatory authorities for heavy-duty vehicles and engines are developing detailed homologation procedures for durability demonstration. Upon completion of this work, it will be possible to define the necessary procedures for 'no-harm' testing and for long term durability of emissions benefit from the use of fuel additives in heavy-duty vehicles and engines. Until such procedures are defined, additives producers must be prepared to demonstrate 'no-harm' and maintenance of the benefit by appropriate fleet or demonstration tests on relevant engines and vehicles.

### **4.5.2 Phase 2: Tests to Demonstrate 'Durability' of the Vehicle's Emission Control System (Light Duty Vehicles) to Metallic Additive**

Long term tests need to evaluate the potential for:

1. Chemical poisoning of emission system components (catalyst, oxygen sensor, traps).
2. Physical blocking/glazing of emission control system components (including catalyst & oxygen sensor).
3. Chemical effects on sensor or wash-coat and/or substrate integrity (e.g. leaching).
4. Effects on the base engine combustion process.

### **4.5.3 Phase 2: Rationale for the Selection of Test Procedure**

Test procedures need to be able to evaluate low temperature adsorption/deposition processes, high temperature glazing, desorption and oxidation and alloying processes.

The test procedures described in the following chapters are based on the provisions for emission durability demonstration provided in the Annex 9 of the UN-ECE Regulation 83 [2] and in the Annex VII of the Commission Regulation (EC) No 692/2008 [3] implementing the Euro 5/6 emissions standards.

According to the above mentioned Regulations, three different aging procedures can be used in order to demonstrate that a light duty vehicle complies with the durability requirements:

- Mileage accumulation with a full vehicle using the AMA<sup>5</sup> cycle
- Mileage accumulation with a full vehicle using the Standard Road Cycle (SRC)
- Accelerated bench aging test based on the Standard Bench Cycle (SBC)

The AMA vehicle test cycle was developed before vehicles were equipped with catalytic converters and therefore contains a substantial portion of low speed driving, designed to address concerns about engine deposits. In fact it provides a regime with low operating temperatures and low space velocities that should reproduce plugging and glazing phenomena and could replicate deposit-forming processes in the engine for both gasoline and diesel light duty vehicles. While engine deposits were a major source of emissions deterioration in pre-catalyst vehicles, the advent of catalytic converters, better fuel control, and the use of cleaner fuels shifted the causes of deterioration from low speed driving to driving modes which include higher speed/load regimes that cause elevated catalyst temperatures. Consequently the effect of deterioration has to be verified more on the after treatment devices than on the engine-out emissions. The AMA driving cycle does not adequately focus on these higher catalyst temperature driving modes<sup>6</sup>. For this reasons a test to evaluate the effect of fuel additives on emission control technologies cannot be based only on the AMA cycle.

The Standard Bench Cycle (SBC) has been introduced in the European legislation in order to provide an alternative to the manufacturer with the aim of reducing the time needed to run a durability demonstration programme and the related costs. The SBC is based on the principle that it is possible to reach a given level of thermal deactivation of a catalyst in a much shorter time by exposing the catalyst to a higher exhaust gas temperature. The aging time needed to reach the given deactivation level as a function of the exhaust gas temperature is provided by the Arrhenius equation which relates chemical reaction rates with temperature.

However, an accelerated bench cycle does not adequately address the fuel poisoning and deposit formation aspects and for this reason in the US-EPA Emission Durability Procedure, which first introduced the SBC, the catalyst aging time is adjusted with the multiplicative factor 1.1 to account for deterioration from sources other than thermal aging of the catalyst. This has been also introduced in the European legislation.

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<sup>5</sup> US Automobile Manufacturers Association

<sup>6</sup> Environmental Protection Agency, 40 CFR Part 86, Emission Durability Procedures and Component Durability Procedures for New Light-Duty Vehicles, Light-Duty Trucks and Heavy-Duty Vehicles; Final Rule and Proposed Rule Federal Register / Vol. 71, No. 10 / Tuesday, January 17, 2006 / Rules and Regulations, pag. 2811

Furthermore, if the objective is to demonstrate the no-harm of a fuel additive, an accelerated aging procedure like the SBC may be not suitable to replicate the interactions between the candidate additive and the emission control system for two main reasons: the amount of additive seen by the after-treatment systems during the accelerated test would be much lower than in the real world use and, moreover, the higher exhaust gas temperature may result in chemical interactions between the additive and the after-treatment systems that do not occur at the lower exhaust gas temperatures typical of real world vehicle driving patterns.

Therefore, the AMA cycle and the Standard Bench Cycle should be considered as not suitable to address the effects of a fuel additive on the emission control system of modern vehicles unless they are run in combination as complementary test procedures.

The Standard Road Cycle (SRC) described in Appendix 10 is a mileage accumulation cycle combining low and high temperature poisoning that was first introduced in the US-EPA Emission Durability Procedure to replace the obsolete AMA cycle. With the entry into force of the Commission Regulation (EC) No 692/2008 this cycle can be now used in Europe as an alternative cycle for emission durability demonstration.

The SRC, providing a balanced mix of high and low exhaust gas temperatures, can be considered the most suitable aging procedure among those prescribed by the current European emission legislation to address the 'no-harm' aspect of fuel additives on modern emission control systems. It is suitable for both petrol and diesel light duty vehicles.

#### **4.5.4 Phase 2: Test Procedure for 'No-harm' Demonstration for Light Duty Vehicles**

This section of the test requires the evaluation of emissions over 160,000 km of on-road, track or chassis dynamometer driving to a pre-determined driving pattern. The test programme compares the results from two identical test fleets, one running on the additive-treated fuel, the other on base fuel. Test fleet and fuel selection has been already described in chapters 4.1 and 4.2.

##### **4.5.4.1 Outline of Phase 2 of the Test Programme**

1. Upon completion of phase 1 of the test protocol, start the mileage accumulation phase using the SRC cycle for mileage accumulation (until reaching 160000 km). Throughout the test programme, a test fleet is continually operated using the base reference fuel only, the other with the additive treated fuel.

**It is essential to ensure that every vehicle is operated ONLY and continuously, on the fuel for which it had been nominated. Any change from the additive treated fuel to non-treated fuel or vice versa, will nullify the tests with this vehicle.**

2. The vehicles have to be aged for 160000 km. Under no circumstances can the test distance be shortened. No extrapolation of the test results based on an early stop of the programme is allowed.

3. During the mileage accumulation OBD monitors capable of logging the main engine parameters and any malfunction code have to be fitted to each vehicle.
4. Any MIL (Malfunction Indicator Light) events shall be monitored and recorded. All MIL events must be investigated within 10km of occurrence and a Report included with the test data. Rectification must be undertaken before continuing with the test.
5. Measure emissions every 30000 km up to and including 160000 km. When possible high definition colour pictures shall be take of any contamination of the catalyst front face, of the spark plugs and of the sensors.
6. In case of major breakdowns of a vehicle or of an unacceptable increase of emissions leading to its exclusion from the continuation of the test programme, post-mortem analyses should be carried out in order to identify the likely reason of the problem and a justification of the exclusion has to be provided to the advisory board. A list of parts that should be checked is given below:
  - Catalist
  - Spark plugs.
  - Oxygen and other sensors.
  - Fuel injectors (internal and external).
  - Cylinder head – exhaust outlets.
  - Turbo charger.
  - Exhaust manifold

#### 4.5.4.2 Mileage Accumulation and Operating Cycle

The mileage accumulation has to be conducted by driving the vehicles over the Standard Road Cycle (SRC) as described in Appendix 1 (See also Annex VII to the Commission Regulation (EC) No 692/2008).

The mileage accumulation should be preferably carried out on a chassis dynamometer since this allows a better level of repeatability and reproducibility and a higher degree of test condition stability compared to driving the vehicles on the road. However, the availability of an adequate number of chassis dynamometers to run 24 vehicles in parallel is an issue. If the use of only chassis dynamometers turns out to be unfeasible, the mileage accumulation can be conducted on a test track. Driving the vehicles on the road is the least preferred option and should be avoided, if possible. In case of a limited number of chassis dynamometers available, the vehicles that will be aged using this approach shall be selected in order to ensure the highest degree of comparability (e.g. if 6 dynamometers are available, three different models should be selected and 3 vehicles should be aged without the additive while the other 3 vehicles of the same models are aged with the treated fuel).

If the mileage accumulation is carried out on a chassis dynamometer, a load equal to their inertia weight class, as used for emissions testing, shall be set.

Between each of the test points on the mileage accumulation cycle, several full load checks over the entire engine speed should be undertaken at random intervals within the 30,000 Km.

In case of development of a new durability cycle (e.g. globally harmonized and/or more representative of the ‘real world’ driving conditions) for regulatory purposes, the SRC will be replaced by the new cycle. Since the car fleet for the test protocol has been defined taking into account the characteristic of the Standard Road Cycle, its composition and characteristics will have to be re-assessed each time a new cycle is developed and used for regulatory purposes.

#### 4.5.4.3 Emissions Test

Two back-to-back emission tests (both using the legislative driving cycle and the complementary “real-world” cycle) have to be periodically performed to monitor the evolution of the regulated emissions during the vehicle aging and, in case of the fleet running on the treated fuel, to generate data on unregulated emissions.

Once the mileage accumulation has started, the first two back-to-back emission tests have to be performed at a mileage of 30000 km and afterwards every 30000 km.

It is recommended that in case of significant variations of the emissions compared to the base line, the interval at which the emissions have to be carried out is reduced to 20000 km. In this respect a mid-term check of the programme should be conducted by the Advisory Board.

The emissions tests must be carried out in accordance with the EU procedures to which the vehicles/engines were originally type approved and following the daily schedule detailed in Section 4.4.5.

#### 4.5.5 Test Data Reporting

A test report must be submitted for each individual emissions test, using a format substantially similar to, and containing all the information shown in Appendix 5 for Light Duty Vehicles. It is important to include the following:

1. Deterioration plots of all relevant data.
2. Comparisons, which must also allow assessments to be made between the Initial and Final  $\Delta$  between the two different test fleets.

FULL data must be in any case reported.

#### 4.5.6 Evaluation Criteria

A testing programme carried out according to this test protocol can be expected to produce multiple results on multiple vehicles with a certain level of variability and uncertainty.

Therefore, it is considered inappropriate to set pre-defined pass/fail criteria to determine whether the tested additives are in line with existing EU legal requirements in terms of the effect on exhaust emissions and potential risk that they pose to the environment and health encompassing all the possible result combinations.

The following approach is suggested:

- The test results will have to be assessed and analysed by using acknowledged statistical methods in order to identify the statistically significant effects which can be attributed to the metallic additive.
- In particular the statistical analysis of the data will have to establish whether relevant emission limits are significantly exceeded and/or levels of regulated emissions are significantly different when comparing the two sets of vehicles aged with and without the metallic additive.
- Any MIL event that occurred during the test programme should be carefully analysed in order to check whether it is related to the use of the additive.
- The raw data and the statistically treated data will have to be reviewed by the expert group which will have to take into account the number and representativeness of the vehicles tested, the variability between test results, and the criticality of regulated emissions.
- A final report summarizing the findings will have to be generated by the expert group taking into account the number and representativeness of the vehicles tested, the variability between test results and the existing EU legal requirements.

**NB:** The test design allows for a distribution of emissions results in any population of vehicles of the same type at the zero km test point. It is not necessary for the two vehicles to have identical emission results at the zero km point.

The selection of vehicles to use base or additive treated fuel should be randomised - it is not necessary or desirable to base the selection on relative emissions results. Attention should be given to the fact that the average emissions of the two fleets of vehicles however should not be substantially different.



## **APPENDIX 1            European Emissions Regulations**

### **Passenger Cars and Light Duty Vehicle**

Directive 70/220/EEC and subsequent amendments

Regulations No 595/2009 and 715/2007

UN-ECE Regulation no.83

Note that the test procedure and driving cycle have varied throughout the various Directives amending the base Directive. It is therefore essential that the Directive to which the vehicle was originally typed approved is used.

### **Heavy Duty**

Base Directive 88/77/EEC and subsequent amendments

Regulations of the European Commission No 595/2009 and 582/2011

UN-ECE Regulation no.49

Since the test procedure and engine cycle have varied overtime as the base Directive has been amended various time. It is therefore essential that the specific Directive used is the one the engine was originally typed approved for.

## APPENDIX 2. Planning Guideline: Minimum Number of Vehicles/Engines Needed for Statistical Significance (based on the test-to-test variability found in the EPEFE programme)

**See Section 4.1 for overall minimum number of vehicles/engine requirements**

Emission	EPEFE mean	S.D. (back-to-back tests) (%)	S.D. (long repeats) (%)	S.D. (veh x fuel int'ns) (%)	S.E. (mean of 2 back-to-back tests) (%)	S.E. (mean of 2 pairs of back-to-back tests) (%)	S.E. (diff. in mean of 2 pairs of back-to-back tests) (%)	Vehicles required for a 10% reduction to be sig. at P < 5% (one-sided)	Vehicles required for a 20% reduction to be sig. at P < 5% (one-sided)	Vehicles required for a 30% reduction to be sig. at P < 5% (one-sided)	Vehicles required for a 50% reduction to be sig. at P < 5% (one-sided)
<b>Gasoline (ECE+EUDC)</b>											
<i>HC (g/km)</i>	0.173	8.31%	7.44%	15.25%	9.48%	6.71%	9.48%	4	2	1	1
<i>CO (g/km)</i>	1.417	7.67%	6.60%	11.03%	8.54%	6.04%	8.54%	3	2	1	1
<i>NOx (g/km)</i>	0.172	9.17%	7.82%	12.82%	10.16%	7.18%	10.16%	4	2	1	1
<b>Light-duty diesel (ECE+EUDC)</b>											
<i>HC (g/km)</i>	0.080	11.11%	10.78%	14.10%	13.34%	9.43%	13.34%	6	2	2	1
<i>CO (g/km)</i>	0.402	9.20%	9.42%	9.72%	11.45%	8.10%	11.45%	5	2	1	1
<i>NOx (g/km)</i>	0.541	2.81%	2.82%	3.40%	3.45%	2.44%	3.45%	1	1	1	1
<i>PM (g/km)</i>	0.054	8.13%	6.25%	5.68%	8.49%	6.00%	8.49%	3	2	1	1
<b>Heavy-duty diesel (R49; 13-mode)</b>											
<i>BSHC (g/kWh)</i>	0.253	3.64%	3.51%	7.08%	4.35%	3.08%	4.35%	2	1	1	1
<i>BSCO (g/kWh)</i>	0.611	2.98%	4.76%	5.35%	5.21%	3.68%	5.21%	2	1	1	1
<i>BSNOx (g/kWh)</i>	6.593	0.75%	1.06%	2.39%	1.19%	0.84%	1.19%	1	1	1	1
<i>BSPT (g/kWh)</i>	0.122	3.32%	3.54%	1.54%	4.25%	3.01%	4.25%	2	1	1	1
Notes:											
(1) It is assumed that inferences are to be made about the average percentage reduction in emissions across the tested vehicle/engine fleet only.											
(2) The additional SD (veh x fuel interactions) variance component needs to be taken into account if inferences are to be made about the general population.											
(3) It is assumed that significance tests will be based on fresh estimates of the long-repeat and back-to-back S.D.s obtained in each future test programme.											
(4) Larger numbers of vehicles may be needed to observe statistically significant effects if the S.D.s observed in future test programmes turn out to be greater than those obtained in EPEFE.											
(5) The probability of observing a statistically significant effect may be as low as 50% if the fleet is of the minimum size above											

(6) S.D.(Long repeats) represents the additional variability between tests conducted some time apart over and above the variability found in back-to-back tests S.D. (back-to-back tests)

## **APPENDIX 3 Emissions Testing Procedure: Light Duty Vehicles**

A flow chart for the test procedure is in Figure A.

### **A3.1. Test Sequence Start**

Each laboratory must follow the pre-determined test order specified in Section 4.4.4. In order to help minimise test variability all emissions tests for a given test vehicle must be conducted on the same dynamometer. Since drivers can have a significant impact on the emissions test results, laboratories are encouraged to use the same driver or nominated stand in throughout the test programme. If possible all conditioning should also be carried out on the same dynamometer with the same driver. Every effort should be made to ensure the consistency of the testing and any anomalies must be recorded on the test data log.

### **A3.2. Same Test Fuel?**

Two or four back to back tests will be conducted on each fuel depending on whether the vehicle belongs to the “additive fleet” or to the “non-additive fleet”. The NO option *in figure A* should be taken if the previous test was conducted on a different fuel or if this is the first test in the programme. The YES option should be taken if the previous test was conducted on the same fuel. An independent monitoring is recommendable to ensure that the fuel declared is actually used in the vehicle tests.

### **A3.3. Drain Fuel**

Remove all fuel from the fuel tank by means of the low point drain fitted during the vehicle preparation or using an alternative procedure recommended by the manufacturer. It is vital that as much fuel as possible is removed at this stage to minimise the cross contamination of the various fuel blends being tested.

### **A3.4. 10 Litre Fill**

Fill the vehicle fuel tank with 10 litres of test fuel. Carry out the fuel fill as quickly as possible and ensure the fuel filler cap is replaced immediately, to minimise the evaporative losses in case of gasoline vehicles.

### **A3.5. 5 Min Idle**

Start the engine and idle for 5 minutes to allow the new test fuel to flush the fuel injection system thoroughly.

### **A3.6. Drain Fuel**

Remove all fuel from the fuel tank by means of the low point drain fitted during the vehicle preparation or using an alternative procedure recommended by the manufacturer. It is vital

that as much fuel as possible is removed at this stage to minimise the cross contamination of the various fuel blends being tested.

### **A3.7. 25 Litre Fill**

Fill the vehicle fuel tank with 25 litres of test fuel. Carry out the fuel fill as quickly as possible and ensure the fuel filler cap is replaced immediately, to minimise the evaporative losses.

### **A3.8. Top Up Fuel as Required (Only diesel vehicles)**

Check fuel quantity in tank is sufficient to complete steps 6 through to 10 and top up with test fuel as required.

### **A3.9 Pre-condition**

Gasoline vehicles: After fuel change, carry out a vehicle pre-conditioning cycle to ensure the vehicle has fully adapted to the new test fuel and has taken account of any stoichiometric air/fuel ratio changes that may have occurred. [Consult OEM if necessary]. The pre-conditioning cycle used must be documented.

Diesel vehicles: In case of a vehicle equipped with a DPF particular attention has to be paid to the fill state and/or distance from the regeneration event. An appropriate procedure to ensure that at the moment of the emission test the filter is in similar conditions has to be adopted and carried out prior to the three EUDC test cycles pre-conditioning drive. In this case advice from the manufacturer is necessary.

### **A3.10 Pre-Conditioning According to the Relevant EU Directive**

Carry out one ECE and two consecutive EUDC test cycles for gasoline vehicle or three EUDC test cycle for diesel vehicles according to the emissions test procedure, but do not take exhaust gas samples. These test cycles are to ensure the vehicle is fully conditioned on the test fuel prior to commencement of the emissions test.

### **A3.11 Cold Soak**

The vehicle must be soaked overnight according to the EU test procedure (temperature 20 - 30°C) except that the soak period is restricted to 12 - 36 hours. If the soak period is outside this time envelope, the vehicle must be re-conditioned by repeating steps 10 through to 13. Every effort should be made to minimise the soak temperature variation during the test programme.

### **A3.12. Pre-conditioning of the Dilution Tunnel**

1. Clamp open end of exhaust transfer pipe 0.5 meter above floor level, ensure the pipe is horizontal (to avoid dust being drawn into the system).

2. Start CVS and draw air through the tunnel for 15 minutes with the dilution air inlet restricted by 90% of its area, to increase the flow of air through the exhaust transfer pipe.
3. Fit a weighed particulate filter into the sample holder and remove dilution air inlet restriction.
4. Sample particulate flowing through the tunnel for 10 minutes (do not start the engine yet).
5. Inspect and weigh the filter. In the event of abnormal amounts of particulate being present (>0.025 mg) repeat the conditioning process again. If laboratories cannot meet this specification they must apply a specification consistent with their experience and record the values on the emissions test log sheet.
6. Connect the exhaust transfer pipe to the vehicle exhaust. Care should be taken to ensure that the transfer pipe is moved and bent as little as possible.

**Note:** Steps 3, 4 and 5 are not required for every test but must be carried out at least once per day.

### **A3.13. EU Emissions Test**

Record the engine coolant temperature and oil sump temperature immediately prior to starting the test. Carry out an EU emissions test according to the legislated test procedure defined in the Directive to which the vehicle was originally typed approved. Refer to Appendix 1. Collect one bag sample (and integrated HC sample in case of diesel vehicles) for the urban (ECE) and one bag sample (and integrated HC sample in case of diesel vehicles) for the extra-urban (EUDC) test for the legislated emissions (CO, HC+NO<sub>x</sub> and CO<sub>2</sub>). Collect one particulate filter for the whole test. [If required, the first two cycles of the urban test may be collected separately from the third and fourth, in order to allow more detailed evaluation of the results. The extra-urban cycle should still be collected separately (i.e. three bags/filter pairs/HC data sets)].

Particulate emissions and particle number should be determined according to the legislated EU test procedure by the test laboratory. Filters must be weighed between 2 - 2.5 hours after test completion. The filter paper specification must be according to the EU test procedure (typically 47mm diameter) and must be from the same manufacturing batch. The batch number must be recorded on the test log sheet.

Particle counting equipment should be calibrated according to the procedure described in the relevant legislation.

In case the techniques selected for the determination of the particle-bound metal(s) and their speciation would require different filter media, additional samples should be taken from the dilution tunnel using appropriate additional sampling probes located very close to the sampling probe for particulate mass determination.

All the particulate filters collected during the test programme that will have to be analysed for metal emission characterization must be stored in appropriate single use containers in order to eliminate any risk of contamination.

Some flexibility shall be foreseen by the advisory group concerning determination of the particle-bound metal(s) and their speciation in case relevant data for a specific metallic additive is already available and considered sufficient.

#### **A3.14 Real-world driving cycle**

Upon the completion of the legislative emission test and after all the operations required to prepare the test facility for a new emission test, carry out a second hot start emission test using a “real-world” driving cycle. Examples of cycles that could be used for this purpose are the US06 complementary cycle or the Artemis cycle.

The interval between the first cold start test and the complementary hot start test should be recorded and kept as much as possible constant.

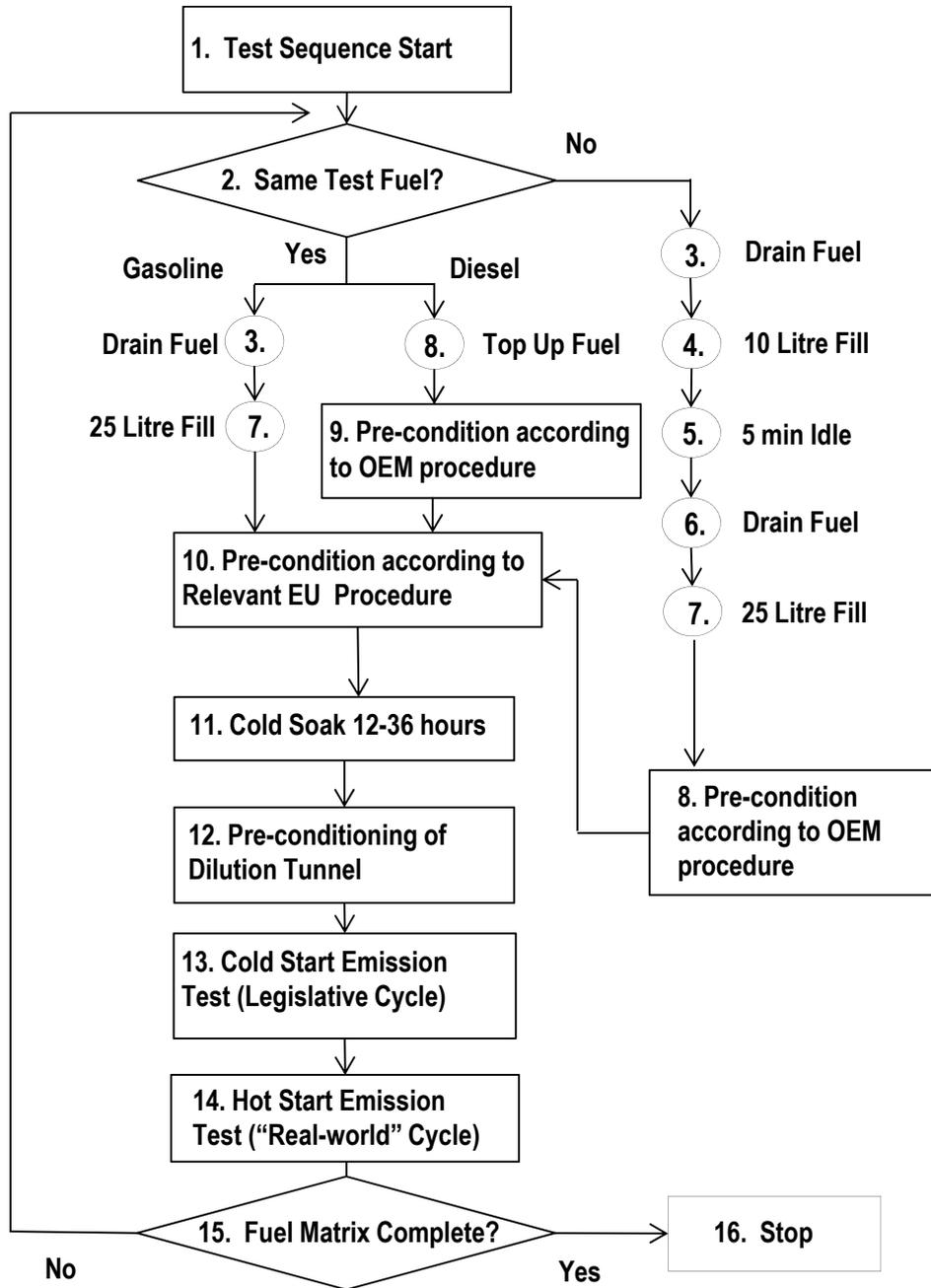
#### **A3.15 Fuel Matrix Complete**

Return to step 2 and continue testing according to the appropriate test order (Section 4.4.4) until the test programme has been completed.

#### **A3.16 Stop**

Testing complete, report test results using the procedures described in Section 4.4.6

### APPENDIX 3: Figure A: Flow chart for testing Light Duty Gasoline Vehicles



## **APPENDIX 4: Emissions Testing Procedure: Heavy Duty Diesel Engines**

### **A5.1 General Test Procedure**

*The emissions test for heavy-duty engines should be that used for certification of the engine tested. For older engines this will be the 88/77/EEC or 91/542/EEC procedure. For vehicles certified to Euro III/IV/V the European Steady State Cycle (ESC), European Load Response Test (ELR) and (if appropriate) European Transient Cycle (ETC) procedures defined in 99/96/EC [4] should be used as appropriate. For Euro VI engines the World Harmonized Transient Cycle (WHTC) and World Harmonized Stationary Cycle (WHSC) procedure should be used as appropriate (Regulations No 595/2009 and No 582/2011 and UNECE Regulation 49). The test shall be carried out according to the specified regulation immediately on completion of the dilution tunnel conditioning.*

The test protocol has been designed so that each set of two tests can be conducted in one day. A flow chart for the test procedure is in Figure C.

### **A5.2 Test Sequence Start**

The test laboratory must follow the pre-determined test order specified (Section 4.4.4).

### **A5.3 Fuel Drain/Change to Next Fuel**

The fuel system shall be drained as much as practical. Taking care to minimise contamination, the fuel drum shall be changed to the next test fuel. The fuel filter of the engine shall be changed.

### **A5.4 Warm-up Engine / Fuel Flush / Adjust Engine**

After starting the engine, it shall be operated 5 minutes at idle, 5 minutes at maximum torque speed/50% load, and up to 20 minutes at rated speed/full load until the oil and water temperatures are stabilised. The temperatures are defined as stabilised if they are maintained within 2% of point for 2 minutes.

If a fuel change is made, the warm-up period shall be used to purge the fuel system of the engine by flushing the new fuel through the engine in the most economical way. If possible, the re-circulating fuel shall be diverted to a waste drum, when the new fuel is first introduced. When most of the previous fuel is flushed out, normal fuel circulation shall be resumed for the remainder of the warm-up period, and the engine operated at least 10 minutes at 'rated speed full load' to remove any remaining fuel before starting the measurement series.

Whenever the engine is shutdown for a break during the daily test sequence (e.g. lunch), the warm-up procedure shall be repeated prior to an emissions test.

In general, the time required very much depends upon the engine and the test cell equipment. The indicated times are a conservative estimate of the maximum time required. It is recommended that *the tests* be conducted according to good engineering practice in the most time and fuel-efficient manner.

#### **A5.5 Condition Dilution Tunnel**

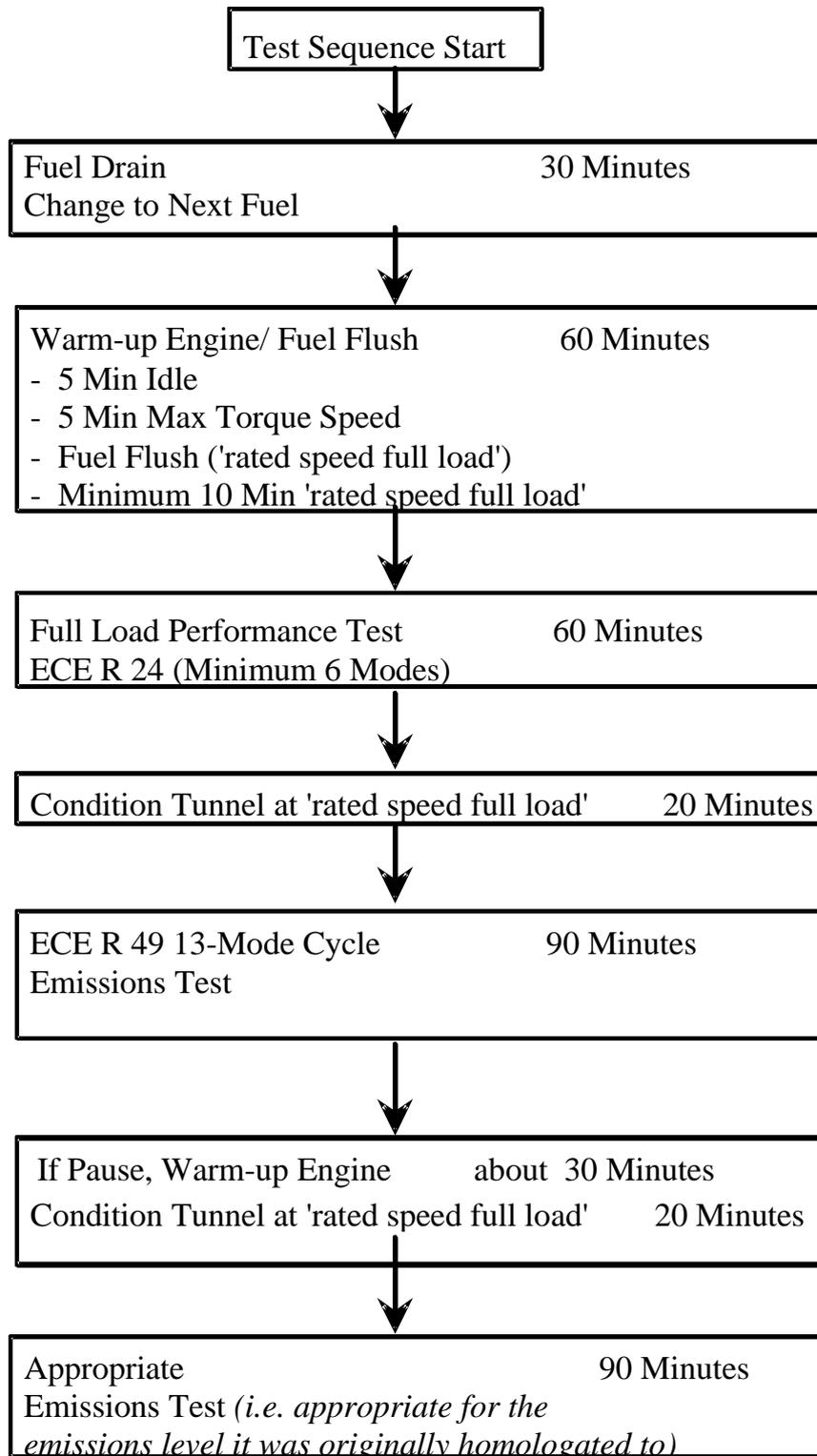
This procedure is essential in order to get repeatable PM test results. For all operation prior to the EU test cycle the tunnel should be by-passed in order to avoid particulate storage in the PM measurement system. When the engine is warm, exhaust gas shall be directed through the dilution tunnel for a period of 20 minutes at 'rated speed full load'. Immediately thereafter, the engine shall be set to idle and the *emissions* test be started. If the engine was shut-off for a break, the warm-up shall be repeated prior to the tunnel conditioning.

#### **A5.6 Emissions Test**

The fuel temperature shall be held constant within  $308 \pm 2\text{K}$  ( $35 \pm 2^\circ\text{C}$ ) during the test, and the measuring point *and conditions* specified by the engine manufacturer. For PM measurement, the single filter method applies. The filter papers must be from the same batch throughout the whole test sequence. The batch number must be recorded on the facility description sheet.

The particulate weight is the sum of the primary and back-up filters. The gaseous emissions shall be measured in the raw exhaust gas, and no measurement of unregulated components is to be done. The test results shall be reported using the format in Appendices 6 and 7.

## APPENDIX 4: Figure C: Flow chart for testing Heavy Duty Diesel Engines



# APPENDIX 5: Test Report, Light Duty Vehicles

## LIGHT DUTY EMISSIONS TEST REPORT

Sheet 1 of 2

DRIVE CYCLE:	(Specify)	VEHICLE ID:	XY27
COMPANY:	ANOEM	MODEL:	EUROCAR
GASOLINE/DIESEL	DIESEL	ODOMETER (km):	8000
TEST No:	OEM-X1-EA5-1a	FUEL CODE:	Abnn
DATE:	DD/MM/YY	CARBON (MASS%):	0,87
DRIVER:	A DRIVER	OIL CODE:	RLxxx
DYNAMOMETER:	PC1	COOLANT TEMP(C):	22,00
INERTIA: LOAD SETTINGS		OIL TEMP (C):	22,20
:			

	ECE Bag 1 (g)	EUDC Bag 2 (g)	ECE+EUDC Per Test (g)	ECE g/km	EUDC g/km	ECE+EUDC g/km
HC	0,000	0,000	0,000	0,000	0,000	0,000
NO <sub>x</sub>	0,000	0,000	0,000	0,000	0,000	0,000
HC+NO <sub>x</sub>	0,000	0,000	0,000	0,000	0,000	0,000
CO	0,000	0,000	0,000	0,000	0,000	0,000
PM	0,0000	0,0000	0,0000	0,0000	0,0000	0,0000
PN	0,000*	0,0000*	0,0000*	0,0000**	0,0000**	0,0000**
CO <sub>2</sub>	0,000	0,000	0,000	0,000	0,000	0,000

\* Total particle number

\*\* Particles/km

FUEL CON (l/100km) (93/116/EEC)	0,000	0,000	0,000
------------------------------------	-------	-------	-------

## LIGHT DUTY EMISSIONS TEST REPORT

RAW DATA	ECE	EUDC	ECE+EUDC		CELL CONDITIONS	
PM LOADING (mg)	00,000	00,000	00,000		ABSOLUTE PRESS (mbar)	0,0
DISTANCE (km)	0,000	0,0000	0,0000		CELL TEMP (°C) (start of test)	0,0
CVS VOLUME (l)	0,0	0,0	0,0		RELATIVE HUMIDITY (%)	0,0
AMBIENT HC (ppm PROPANE)	0,000	0,0000			GAS METER. TEMP (°C)	0,0
AMBIENT CO (ppm)	0,000	0,000				
COMMENTS						
TEST VALIDATION						
TEST ENGINEER: .....			DATE: .....			
TECHNICIAN: .....			DATE: .....			

## APPENDIX 6: Test Report, Heavy Duty Engines

### HEAVY DUTY DIESEL DATA REPORTING SHEET

HD Data Reporting Sheet						Company:	
TESTSITE		ENGINE TYPE		TEST NO.			
TEST CELL		ENGINE NR.		COMMENT			
TEST MANAGER		TEST(R24/R49)		FUEL NO.	ENG. HOURS		
OPERATOR		TEST DATE		ST. INJ. TIMING	TEST (valid/not)		

R49-MODE / R24-FULL LOAD POINT		1	2	3	4	5	6	7	8	9	10	11	12	13
Speed	[RPM]													
Torque	[Nm]													
Power	[kW]													
BSFC	[g/kWh]													
Fuel Delivery	[mg/inj]													
Fuel Temperature	[K]													
Water Temp. bef. Engine	[K]													
Water Temp. after Engine	[K]													
Intake Air Temp.a. Filter	[K]													
Air Temp.after Compr.	[K]													
Air Temp. in Manifold	[K]													
Exhaust Temp. a. Engine	[K]													
Exhaust Temp. a. Turbine	[K]													
Barometer Pressure	[mbar]													
Intake Air Pressure a. Filter	[mbar]													
Pressure a. Compressor	[mbar]													
Exhaust Pressure a. Engine	[mbar]													
Exhaust Pressure a. Turbine	[mbar]													
Boost Pressure in Manifold	[mbar]													
Begin of Injection	[c.a.D]													
Duration of Injection	[c.a.D]													

### HEAVY DUTY DIESEL DATA REPORTING SHEET (CONTINUED)

HD Data Reporting Sheet						Company:	
TESTSITE		ENGINE TYPE		TEST NO.			
TEST CELL		ENGINE NR.		COMMENT			
TEST MANAGER		TEST(R24/R49)		FUEL NO.	ENG. HOURS		
OPERATOR		TEST DATE		ST. INJ. TIMING	TEST (valid/not)		

R49-MODE / R24-FULL LOAD POINT	1	2	3	4	5	6	7	8	9	10	11	12	13
Atmos.Factor R49	[1]												
Fuel Flow	[kg/h]												
Air Flow (wet)	[kg/h]												
Exhaust Flow (wet)	[kg/h]												
Air/Fuel Ratio (dry)	[1]												
Intake Air Humidity	[g/kg]												
Cylinder Peak Pressure	[bar]												
Combustion Noise	[dbA]												
Bosch Smoke	[SZ]												
NO conc.	[ppm]												
HC conc. (C1 equivalent)	[ppm]												
CO conc.	[ppm]												
CO <sub>2</sub> conc.	[vol %]												
NO <sub>x</sub> Hum.corr.Fact.	[1]												
Dry/Wet corr.Fact.	[1]												
BSNO <sub>x</sub>	[g/kWh]												
BSHC	[g/kWh]												
BSCO	[g/kWh]												
BSCO <sub>2</sub>	[g/kWh]												

NO <sub>x</sub> –Measurement: [x]	dry:		wet:	
-----------------------------------	------	--	------	--

Standard Air Temp. in Manifold (as specified by manufacturer for 'rated speed full load' )	[K]	
--	-----	--

### HEAVY DUTY DIESEL DATA REPORTING SHEET (CONTINUED)

HD Data Reporting Sheet						Company:	
TESTSITE		ENGINE TYPE		TEST NO.			
TEST CELL		ENGINE NR.		COMMENT			
TEST MANAGER		TEST(R24/R49)		FUEL NO.	ENG. HOURS		
OPERATOR		TEST DATE		ST. INJ. TIMING	TEST (valid/not)		

R49-MODE (only)	1	2	3	4	5	6	7	8	9	10	11	12	13
Dilution Air Temp. [K]													
Exh. Temp. at PT-Samp. Probe [K]													
Temp. at Tunnel inlet [K]													
Filter Face Temp. [K]													
Sampling Time [s]													

PARTICULATE FILTER	Loading
Primary Filter [mg]	
Backup Filter [mg]	
<b>Total Filter Loading [mg]</b>	

	Weight empty	Weight loaded	Filter Number
Temp. [K]			
Humidity [%]			
Date Time			

R49-TEST RESULTS	
Weighted Power [kW]	
BSFC [g/kWh]	
BSCO [g/kWh]	
BSHC [g/kWh]	
BSNO <sub>x</sub> [g/kWh]	
BSPT [g/kWh]	
BSPN [# /kWh]	
BSPT ORG.FRAC. [g/kWh]	
BSPT Fuel [g/kWh]	
BSPT Lube Oil [g/kWh]	
BSPT SO4 (optional) [g/kWh]	

PT ORG.FRAC. (Prim.Filter) [%]		PT ANALLAB	
PT FUEL out of ORG.FRAC [%]		METHOD (VOF/SOF)	
PT OIL out of ORG.FRAC. [%]		LAB. TECH	
PT SO4 (Prim.Filter) [%]		DATE of analysis	



## APPENDIX 8. Calculation of Geometric Mean Emission Levels and Approximate Standard Errors, Significance Tests and Confidence Intervals

Vehicle												Average Across fuels
Fuel	B					A						
1	True repeat	1		2		1		2		3		
	Back-to-back	1	2	3	1	2	1	2	1	2	1	2
	Result	0.103	0.150	0.112	0.105	0.113	0.099	0.097	0.078	0.080	0.096	0.097
	Ln(Result)	-2.273	-1.897	-2.189	-2.254	-2.180	-2.313	-2.333	-2.551	-2.526	-2.343	-2.333
	Back-to-back average	-2.120			-2.217		-2.323		-2.538		-2.338	
	True repeat average	-2.168					-2.400					
	Geometric mean	0.114					0.091					
	% reduction (% of B)						20.7%					
												<b>-2.284</b>
												<b>0.102</b>
2	True repeat	1		2		1		2				
	Back-to-back	1	2	1	2	1	2	1	2			
	Result	0.113	missing	0.123	0.133		0.091	0.099	0.100	0.110		
	Ln(Result)	-2.180		-2.096	-2.017		-2.397	-2.313	-2.303	-2.207		
	Back-to-back average	-2.180		-2.056			-2.355		-2.255			
	True repeat average	-2.118					-2.305					
	Geometric mean	0.120					0.100					
	% reduction (% of B)						17.0%					
												<b>-2.212</b>
												<b>0.110</b>
3	True repeat	1		2		1		2				
	Back-to-back	1	2	1	2	1	2	1	2			
	Result	0.155	0.172	0.165	0.166		0.140	0.144	0.145	0.146		
	Ln(Result)	-1.864	-1.760	-1.802	-1.796		-1.966	-1.938	-1.931	-1.924		
	Back-to-back average	-1.812		-1.799			-1.952		-1.928			
	True repeat average	-1.806					-1.940					
	Geometric mean	0.164					0.144					
	% reduction (% of B)						12.6%					
												<b>-1.873</b>
												<b>0.154</b>
4	True repeat	1		2		1		2				
	Back-to-back	1	2	1	2	1	2	1	2			
	Result	0.072	0.077	0.080	0.082		0.067	0.061	0.049	0.059		
	Ln(Result)	-2.631	-2.564	-2.526	-2.501		-2.703	-2.797	-3.016	-2.830		
	Back-to-back average	-2.598		-2.513			-2.750		-2.923			
	True repeat average	-2.555					-2.837					
	Geometric mean	0.078					0.059					
	% reduction (% of B)						24.5%					
												<b>-2.696</b>
												<b>0.067</b>
Average across Vehicles/engines	Ln scale	<b>-2.162</b>					<b>-2.370</b>					
	Geometric mean	<b>0.115</b>					<b>0.093</b>					
	% reduction (% of B)						<b>18.8%</b>					
												<b>-2.266</b>
												<b>0.104</b>

The following calculations may be used to determine whether fuel A gives significant reductions in emissions relative to fuel B.

It is assumed that the results are log-normally distributed (S.D. proportional to the mean) and that the (log scale) variability in the results does not differ from vehicle to vehicle.

The analysis is exact when exactly two pairs of back-to-back tests have been conducted on each fuel in each vehicle and there are no missing values or outliers.

The analysis is approximate if (a) a third back-to-back test, or a third pair of back-to-back tests have been conducted in accordance with the protocol for certain vehicle × fuel combinations or (b) certain values are missing or have been rejected as outliers

$$\begin{aligned} \text{Sum of squares SS (true repeats)} &= [-2.120 - (-2.168)]^2 + [-2.217 - (-2.168)]^2 + [-2.323 - (-2.400)]^2 + \dots + [-2.750 - (-2.837)]^2 + [-2.923 - (-2.837)]^2 \\ &= 0.06522 \end{aligned}$$

$$\text{Residual degrees of freedom DF} = 17 \text{ (no. of true repeats conducted)} - 4 \text{ (no. of vehicles)} \times 2 \text{ (no. of fuels)} = 9$$

$$\text{Residual mean square MS} = \text{SS} / \text{DF} = 0.007247$$

$$\text{Root mean square RMS} = \sqrt{\text{MS}} = 0.08513$$

$$\text{SE (mean for one fuel on ln scale)} = \text{RMS} / \sqrt{2 \times \text{no. of vehicles}} = 0.0301$$

$$\text{SE (difference in means on ln scale)} = \text{RMS} / \sqrt{\text{no. of vehicles}} = 0.04256$$

$$t_9 = [-2.370 - (-2.162)] / 0.04256 = -4.89 \text{ (significant at } P < 0.1\% \text{ in a one-sided test)}$$

$$95\% \text{ confident that true reduction exceeds } [-2.370 - (-2.162)] + t_{9;95\%(1\text{-sided})} \times 0.04256 = -0.208 + 1.833 \times 0.04256 = -0.129 \text{ (ln scale)}$$

$$95\% \text{ confident that true reduction exceeds } (1 - \exp(-0.129)) = 12.2\%$$



## **APPENDIX 9: Methodology for Calculating Emissions Effect and Statistical Significance: Rigorous Statistical Analysis Guidelines**

It is difficult to formulate a statistical analysis protocol which will cover all eventualities. Therefore this Section confines itself to a set of guidelines to be followed when analysing emissions data collected under this protocol. Fuller details may be found in references [5, 6].

### *1. Examination of the data for outliers*

The data should be examined for outliers using both statistical and graphical techniques [5, 6] and suspicious results should be queried with the test laboratory. Any transcription or similar errors should be corrected. Statistical outlier tests (e.g. Cochran's test) may be used to reject results which are so out of line from the remainder that it can only be concluded that some fault or abnormality must have occurred in the application of the test method or in the handling or labelling of the test fuel. Discrepancies should be sought both within pairs of back-to-back tests and between pairs of true repeats. When emissions are low, due attention must be paid to the possible influence of rounding on variability estimates/plots and outlier tests.

### *2. Examination and transformation of the data to ensure homogeneity of variance.*

Standard deviation Vs mean plots may be used to assess whether the variability in the data varies with the emission level or not. If it does vary, a suitable variance stabilising data transformation must be found. The main emphasis should be placed on stabilising the variability between true repeats. In the absence of strong evidence to the contrary, the standard deviation shall be assumed to be proportional to the mean and the in transform used.

### *3. Check that the variance is constant for different vehicles/engines.*

Standard deviation Vs mean plots and/or Bartlett's test may be used to assess whether the true repeat variability differs from vehicle to vehicle. If so, the results will need to be analysed on a vehicle by vehicle basis.

*Steps 1-3 are highly inter-dependent and need to be considered in toto. For example, a measurement might appear to be an outlier in its original units, but be concordant with the remainder after transformation. Likewise the apparent homogeneity of variance across vehicles will depend on the decisions taken re potential outliers and data transformation. Careful judgements need to be made vis-à-vis the order in which steps 1 to 3 are carried out and some recycling may be necessary.*

### *4. Analysis of variance or multiple regression*

The cleaned and transformed data from steps 1-3 should be analysed using two-way analysis of variance techniques. However, when third tests or missing or rejected results render the data unbalanced, multiple regression techniques must be used instead.

It is recommended that the analysis be conducted using a statistical package such as Statistical Analysis Software (SAS). The following SAS code

```
PROC GLM DATA=A;  
  
CLASS VEH FUEL LONG;  
  
MODEL LNY = VEH FUEL VEH*FUEL LONG(VEH FUEL);  
  
RANDOM LONG(VEH FUEL);  
  
LSMEANS VEH FUEL VEH*FUEL / STDERR E=LONG(VEH FUEL);  
  
RUN;
```

will calculate the means of the transformed values  $y = \ln(x)$  as described in the “Quick analysis” in section 8.4. PROC GLM will also compute correct standard errors for these means and perform significance tests for vehicle and fuel effects. Confidence intervals for differences in log emissions between fuels or between vehicles may be obtained by judicious use of the ALPHA, CL and PDIFF options to LSMEANS.

The standard errors and significance tests, computed using the code above, are only strictly correct when the variance of the transformed values  $y$  does not differ from vehicle to vehicle. When the variance is non-homogeneous, the techniques described in [5] (Annex 05) and [6] may be used to compute the standard error of the fleet average emissions for each fuel. Reference [5] also gives suitable SAS code.

## APPENDIX 10 : Standard Road Cycle (Light Duty Vehicles)

### 1. Introduction

The Standard Road Cycle (SRC) is a kilometre accumulation cycle. The vehicle may be run on a test track or on a kilometre accumulation dynamometer.

The cycle consists of 7 laps of a 6 km course. The length of the lap may be changed to accommodate the length of the mileage accumulation test track.

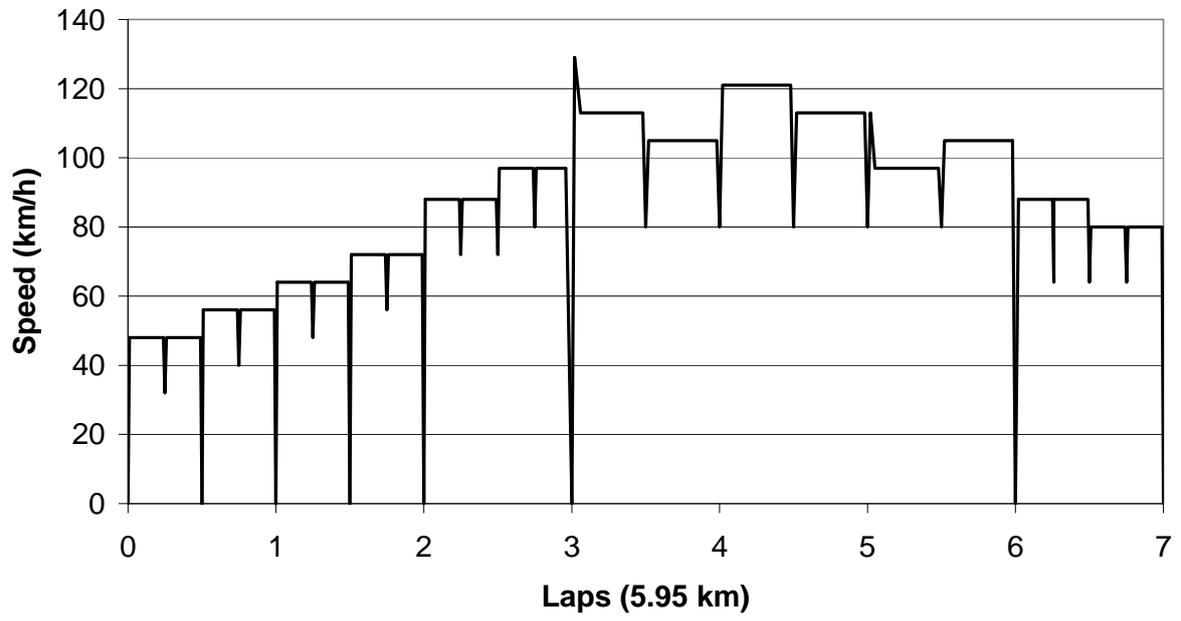
### 2. Standard Road Cycle

Lap	Description	Typical acceleration rate m/s <sup>2</sup>
1	(start engine) idle 10 seconds	0
1	Moderate acceleration to 48 km/h	1.79
1	Cruise at 48 km/h for ¼ lap	0
1	Moderate deceleration to 32 km/h	-2.23
1	Moderate acceleration to 48 km/h	1.79
1	Cruise at 48 km/h for ¼ lap	0
1	Moderate deceleration to stop	-2.23
1	Idle 5 seconds	0
1	Moderate acceleration to 56 km/h	1.79
1	Cruise at 56 km/h for ¼ lap	0
1	Moderate deceleration to 40 km/h	-2.23
1	Moderate acceleration to 56 km/h	1.79
1	Cruise at 56 km/h for ¼ lap	0
1	Moderate deceleration to stop	-2.23
2	idle 10 seconds	0
2	Moderate acceleration to 64 km/h	1.34
2	Cruise at 64 km/h for ¼ lap	0
2	Moderate deceleration to 48 km/h	-2.23
2	Moderate acceleration to 64 km/h	1.34
2	Cruise at 64 km/h for ¼ lap	0
2	Moderate deceleration to stop	-2.23
2	Idle 5 seconds	0
2	Moderate acceleration to 72 km/h	1.34
2	Cruise at 72 km/h for ¼ lap	0
2	Moderate deceleration to 56 km/h	-2.23
2	Moderate acceleration to 72 km/h	1.34
2	Cruise at 72 km/h for ¼ lap	0
2	Moderate deceleration to stop	-2.23
3	idle 10 seconds	0
3	Hard acceleration to 88 km/h	1.79
3	Cruise at 88 km/h for ¼ lap	0
3	Moderate deceleration to 72 km/h	-2.23
3	Moderate acceleration to 88 km/h	0.89
3	Cruise at 88 km/h for ¼ lap	0
3	Moderate deceleration to 72 km/h	-2.23
3	Moderate acceleration to 97 km/h	0.89
3	Cruise at 97 km/h for ¼ lap	0
3	Moderate deceleration to 80 km/h	-2.23
3	Moderate acceleration to 97 km/h	0.89

3	Cruise at 97 km/h for ¼ lap	0
3	Moderate deceleration to stop	-1.79
4	idle 10 seconds	0
4	Hard acceleration to 129 km/h	1.34
4	Coastdown to 113 km/h	-0.45
4	Cruise at 113 km/h for ½ lap	0
4	Moderate deceleration to 80 km/h	-1.34
4	Moderate acceleration to 105 km/h	0.89
4	Cruise at 105 km/h for ½ lap	0
4	Moderate deceleration to 80 km/h	-1.34
5	Moderate acceleration to 121 km/h	0.45
5	Cruise at 121 km/h for ½ lap	0
5	Moderate deceleration to 80 km/h	-1.34
5	Light acceleration to 113 km/h	0.45
5	Cruise at 113 km/h for ½ lap	0
5	Moderate deceleration to 80 km/h	-1.34
6	Moderate acceleration to 113 km/h	0.89
6	Coastdown to 97 km/h	-0.45
6	Cruise at 97 km/h for ½ lap	0
6	Moderate deceleration to 80 km/h	-1.79
6	Moderate acceleration to 104 km/h	0.45
6	Cruise at 104 km/h for ½ lap	0
6	Moderate deceleration to stop	-1.79
7	idle 45 seconds	0
7	Hard acceleration to 88 km/h	1.79
7	Cruise at 88 km/h for ¼ lap	0
7	Moderate deceleration to 64 km/h	-2.23
7	Moderate acceleration to 88 km/h	0.89
7	Cruise at 88 km/h for ¼ lap	0
7	Moderate deceleration to 64 km/h	-2.23
7	Moderate acceleration to 80 km/h	0.89
7	Cruise at 80 km/h for ¼ lap	0
7	Moderate deceleration to 64 km/h	-2.23
7	Moderate acceleration to 80 km/h	0.89
7	Cruise at 80 km/h for ¼ lap	0
7	Moderate deceleration to stop	-2.23

The Standard Road Cycle is represented graphically in the following figure:

### Standard Road Cycle



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## References

- 1) Council Directive 70/220/EEC of 20 March 1970. "On the approximation of the laws of the Member States relating to measures to be taken against air pollution by gases from positive-ignition engines of motor vehicles".
- 2) United Nations, Economic Commission for Europe, Regulation No 83, "Uniform Provisions Concerning the Approval of Vehicles with Regard to the Emission of Pollutants According to Engine Fuel Requirements".
- 3) Commission Regulation (EC) No 692/2008, 18 July 2008, "Implementing and amending Regulation (EC) No 715/2007 of the European Parliament and of the Council on type-approval of motor vehicles with respect to emissions from light passenger and commercial vehicles (Euro 5 and Euro 6) and on access to vehicle repair and maintenance information".
- 4) Council Directive 91/542/EEC of 01 October 1991. Amending Directive 88/77/EEC "On the approximation of the laws of the Member States relating to the measures to be taken against the emission of gaseous pollutants from diesel engines for use in vehicles".
- 5) Directive 1999/96/EC of the European Parliament and of the Council of 13 December 1999, "On the Approximation of the Laws of the Member States Relating to Measures to be Taken Against the Emission of Gaseous and Particulate Pollutants from Compression-Ignition Engines for Use in Vehicles, and the Emission of Gaseous Pollutants from Positive-Ignition Engines Fuelled with Natural Gas or Liquefied Petroleum Gas for Use in Vehicles and Amending Council Directive 88/77/EEC".
- 6) "*European Programme on Emissions, Fuels and Engine Technologies - EPEFE Report*". ACEA and Europa, 1996.
- 7) "European Programme on Emissions, Fuels and Engine Technologies (EPEFE) - Statistical Design and Analysis Techniques". SAE paper 961069, 1996
- 8) Regulation (EC) No 595/2009 of the European Parliament and of the Council of 18 June 2009 on type-approval of motor vehicles and engines with respect to emissions from heavy duty vehicles (Euro VI) and on access to vehicle repair and maintenance information and amending Regulation (EC) No 715/2007 and Directive 2007/46/EC and repealing Directives 80/1269/EEC, 2005/55/EC and 2005/78/EC
- 9) Commission Regulation (EU) No 582/2011 of 25 May 2011 implementing and amending Regulation (EC) No 595/2009 of the European Parliament and of the Council with respect to emissions from heavy duty vehicles (Euro VI) and amending

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Annexes I and III to Directive 2007/46/EC of the European Parliament and of the Council