

# Quality Test Procedures & Emissions with DPF+SCR Systems

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**Abstract** The combined exhaust gas aftertreatment systems (DPF+SCR) are the most efficient way and the best available technology (BAT) to radically reduce the critical Diesel emission components particles (PM&NP) and nitric oxides (NO<sub>x</sub>). SCR (selective catalytic reduction) is regarded as the most efficient deNO<sub>x</sub>-system, diesel particle filters are most efficient for soot abatement. Today, several suppliers offer combined systems for retrofitting of HD vehicles.

Quality standards for those quite complex systems and especially for retrofit systems are needed to enable decisions of several authorities and to estimate the potentials of improvements of the air quality in highly populated agglomerations.

The present paper informs about the VERTdePN \*) quality test procedures, which were developed in an international network project with the same name (dePN ... decontamination, disposal of PM / NP and of NO<sub>x</sub>) 2007-2011. Some interesting results of research on the engine dynamometer from the last test period 2011-2013 are given as a complement of the already published results.

The objective to introduce the SCR-, or combined DPF+SCR-systems in the VERT verification procedure was accomplished. During the tests additionally to the regulated gaseous emissions several unregulated components such as NH<sub>3</sub>, NO<sub>2</sub> and N<sub>2</sub>O were measured. The analysis of nanoparticle emissions was performed with SMPS and NanoMet.

The most important statements are:

- the procedures for the quality verification of SCR-, or (DPF+SCR) - systems are developed and confirmed,
- these test procedures on HD-chassis dynamometer and on-road are useful for OEM- and for retrofit systems,
- engine dynamometer testing enables the deepest insight in the investigated system concerning: secondary- and non-legislated emissions, variations of feed factor, analysis on different sampling positions and at specific engine operating conditions (like legal test procedures),
- testing on HD-chassis dynamometer can partially replace the engine dynamometer depending on the possibilities of the installation,
- testing of SCR-systems on vehicle is important, because of urea dosing, urea mixing and electronic control,
- the filtration efficiency of a DPF is independent of the operating condition (except of regeneration period, or passing over the maximum space velocity),
- the NO<sub>x</sub> reduction efficiency of SCR-systems is dependent on the operating conditions, because of the optimal temperature window of the SCR-catalysis; at the conditions with exhaust temperature below 200°C the urea dosing is stopped.

There is an intense further development of those aftertreatment systems and their electronic control, which opens further potentials of improvements.

**Keywords** SCR ... Selective Catalytic Reduction, Quality Procedures, Retrofitting, DPF ... Diesel Particle Filter

## 1. Introduction & Objectives

The use of deNO<sub>x</sub> (especially SCR) systems and the combinations with DPF's offer a large number of variants and technical complexity representing new challenges not

only for the manufacturers, but also for the users and for the responsible authorities.

Retrofit with those combined systems is quite challenging and it is possible, in general opinion, mostly through incentives, or restrictions with respect to low emission zones LEZ,[1] and regulations of the respective authorities.

The Swiss Federal Office of Environment BAFU and the Swiss Federal Roads Office ASTRA decided to support the activities of VERT to develop appropriate testing

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procedures and to define the quality criteria of dePN systems.

Laboratories for IC-Engines and Exhaust Emission Control of the University of Applied Sciences Biel, Switzerland (AFHB) participate since 1992 at the Swiss activities about nanoparticle analytics and DPF verification and since 2006 about the quality of SCR- and (DPF+SCR) - systems.

The need of testing the SCR-systems together with vehicle became stronger and the supporting Federal Offices accorded a supplementary project TeVeNO<sub>x</sub> (Testing of Vehicles with NO<sub>x</sub> reduction systems). In this project 2012-2013 several HD vehicles with SCR (OEM and retrofit) were tested and the test methods on HD chassis dynamometer and on-road were confirmed.

There is an intense research and development of SCR systems and their implementation,[2-13]. As effect significant reduction of the target emission parameters is possible.

The objectives of this paper are to show the last state of the VERTdePN testing procedures and to represent some new findings from different research subjects on engine like:

- results in different test cycles,
- secondary nanoparticles,
- comparison of different aftertreatment systems,
- demonstration of NH<sub>3</sub>-storage,
- cleaning of the system,
- results at different steptests,
- secondary emissions with Cu-Fe-zeolithes,
- quality of a retrofit system after 1000 hours.

## 2. VERTdePN

A general objective of VERTdePN is to include the SCR-, or the combined DPF+SCR systems in the test procedures, which were previously developed for DPF retrofitting.

### 2.1. The Subjects & Procedures

For the VERT DPF quality procedure (SN 277206),[14] the research objectives are:

- filtration quality
- durability
- control - & auxiliary systems
- secondary emissions.

The objectives for a SCR system in the VERTdePN tests are:

- NO<sub>x</sub> reduction efficiency
- NO<sub>2</sub>- and / or NH<sub>3</sub>- slip
- emission of N<sub>2</sub>O
- operating temperature window
- dynamic operation
- field application & durability
- auxiliary systems

- further secondary emissions.

The main structure of VERTdePN tests for combined DPF-SCR is similar, as the preceding VERT activities for DPF.

Fig. 1 shows the scheme of the product standard testing and the main performed subprojects.

There are following test procedures:

1) VERTdePN test 1 (VPNT1) on engine dynamometer, or on chassis dynamometer with equivalent measuring possibilities. VPNT1 contains stationary engine operation in step test, including load transitions and urea switch on & off. Additionally to the reduction efficiencies of NO<sub>x</sub> & NO<sub>2</sub>, the measurement of some non-legislated emission components is required. These components are: Ammonia NH<sub>3</sub> and nitrous oxide N<sub>2</sub>O.

If the tested DPF is not yet approved by VERT/OAPC, the measurements of nanoparticles and the estimate of PCFE are necessary.

The demonstration of deNO<sub>x</sub>-efficiency at dynamic operation (ETC, WHTC, or FIGE) is recommended.

2) VERTdePN Secondary Emissions Test (VPNSET) is applied, if necessary, to state, if there are any potentials of secondary emissions of dioxines and furanes, which are considered as particularly toxic.

VPNSET needs for the analytics of traces a long and well controlled sampling procedure. For this purpose an ISO 8178 8-pts tests, including the load transitions is used. VPNSET can only be performed on engine dynamometer.

3) VERTdePN Test 2 (VPNT2) is performed on vehicle. This is a preliminary control before field test and a real world durability test of 1000h, or 50'000 km.

The preliminary control has to be performed on a HD chassis dynamometer at stationary OP's with SWON & SWOFF and with exhaust gas measuring technics, which enable at least the estimate of the deNO<sub>x</sub>-rate (KNOX). Also the datalogging, which is prepared for the field test shall be applied and evaluated. The results of the preliminary control are reference values for the final control after field test (VPNT3).

During the field operation a continuous datalogging of NO<sub>x</sub>-signals before and after system with periodical controls has to be performed. All results of controls, service & maintenance, or failures shall be documented in a log-book.

4) VERTdePN Test 3 (VPNT3) is a repetition of the preliminary control of VPNT2 on the same chassis dynamometer, at the same operating conditions (OP's & tamb) and with the same measuring apparatus, in order to demonstrate the system efficiency after the long run.

These test procedures can be used to recognize a product standard and to recommend the retrofit systems to the users.

The formal procedures of approval and the used criteria are to be decided by the implementing authority and are not a subject of this paper.

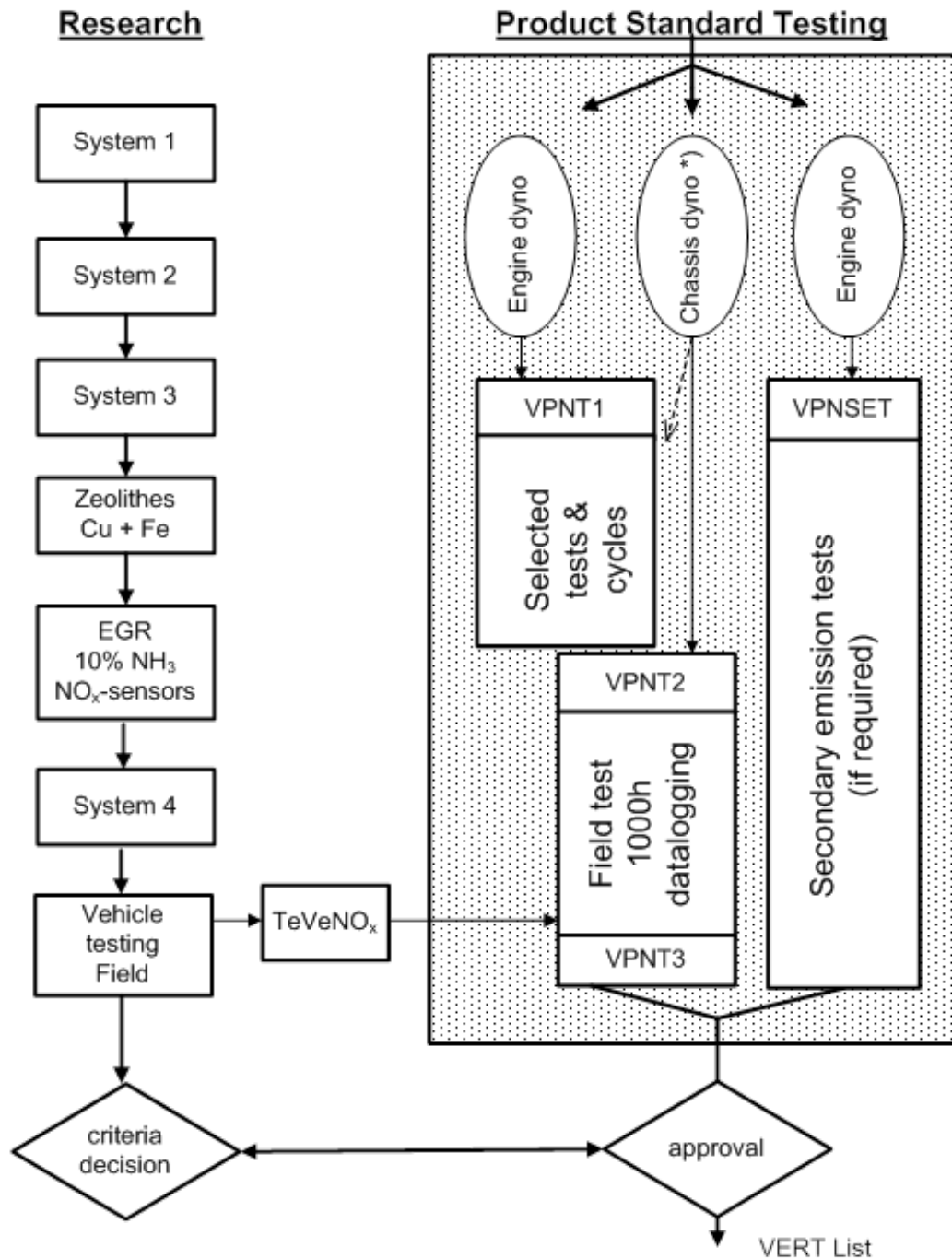


Figure 1. VERTdePN Testing Procedure for DPF+SCR Combisystems Product Standard (DPF VERT certified)

From the technical points of view some flexibilities of implementing of these test procedures are possible in view of promotion of the retrofitting. This concerns first of all the acceptance of other tests, which were already performed by the manufacturer and which deliver similar information about the system.

It is also to consider that the retrofitting can be done only for certain fleets, which are handled by specialized personal, which usually have their own quality control and do not need such rigorous OBD measures, like those introduced recently for OEM-SCR applications.

For each retrofitted SCR-system, which is put into

operation a simplified acceptance test on vehicle has to be performed. This test consists of checking the datalogger and the urea SWON & SWOFF during a short road trip and standstill.

## 2.2. Test Cycles

For the research on engine dynamometer different steady state test procedures can be used. The main objective is to have at least one urea switch-on (SWON) and at least one high load OP during the test.

The first 4-pts test proposed for VPNT1 is represented in Fig. 2,[15].

The four operating points were chosen in such way, that the switching “off” and “on” of the urea-dosing is included in the tests (pt. 7 → pt. 4 and pt. 4 → pt. 1).

For basic research of SCR-systems a 6-pts test at constant engine speed was used, Fig. 3. This enabled the representation of results in function of exhaust gas temperature.

If the DPF of a combined system has also to be approved it is necessary to apply the OP’s according to the norm,[14]. This requirement leads to the 6-pts test, Fig. 4. In following the result of 4-pts test (Fig. 2) and 6-pts test (Fig. 4) will be compared.

During the long-duration sampling for VPNSSET the ISO 8178 8-pts test is used,[15].

The dynamic testing was started with the European

Transient Cycle ETC and continued with the WHTC (worldwide heavy duty transient cycle), Fig. 5. Also two city-bus cycles: NYCC (New York City Cycle) and Braunschweig Bus Cycle were investigated. All these cycles were defined on the basis of the full (non-limited) engine map. They cause very different operating conditions of the engine, which is depicted with different profiles of exhaust gas temperatures at the tailpipe (Fig. 5, bottom).

The tests were performed after a warm-up phase, when the engine coolant temperature and lube oil temperature reached their stationary values (stationary points tests).

Before starting a dynamic cycle the same conditioning procedure was used to stabilize the thermal conditions of the exhaust gas aftertreatment system. This conditioning was: 5 min at point 1 (2200 rpm/FL) and 0.5 min of idling.

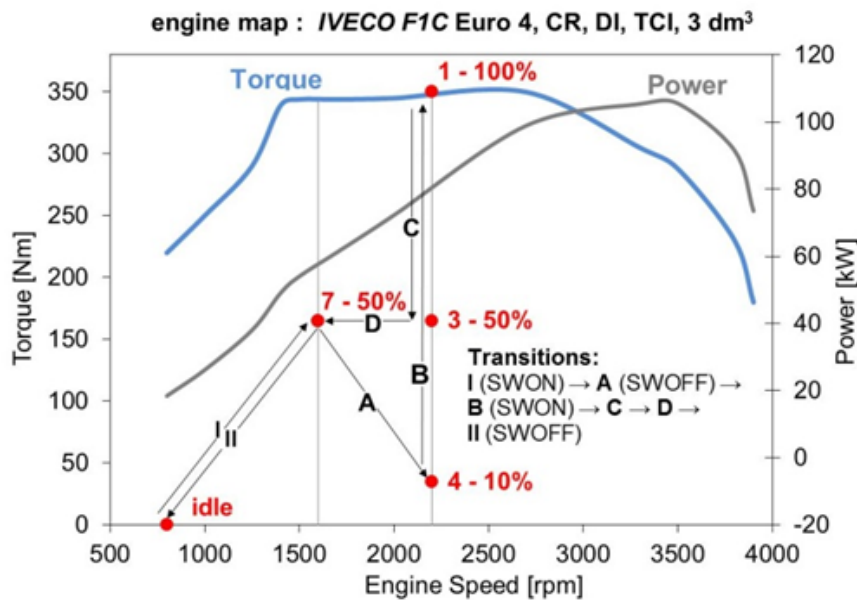


Figure 2. Engine map of the IVECO F1C engine and 4-points test for SCR-investigations

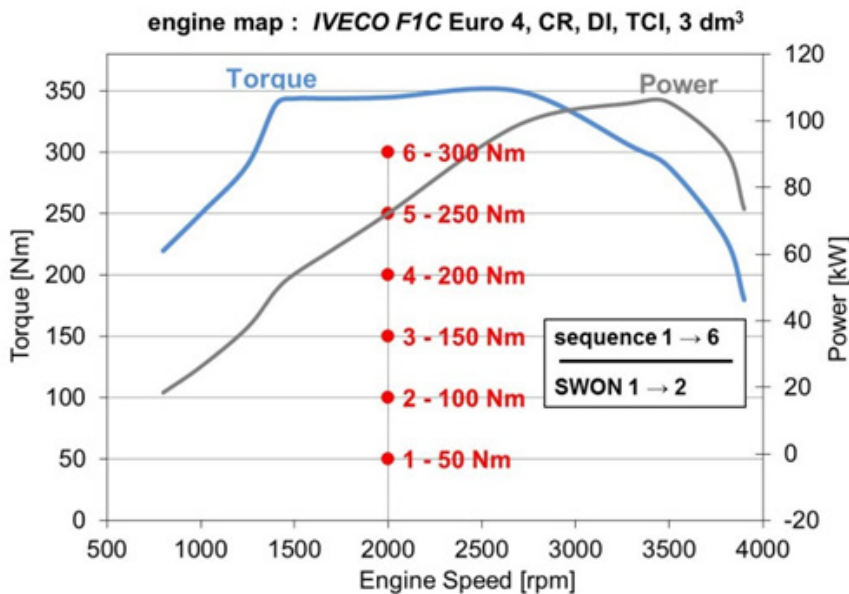


Figure 3. Engine map of the IVECO F1C engine and 6-points steptest for SCR-investigations

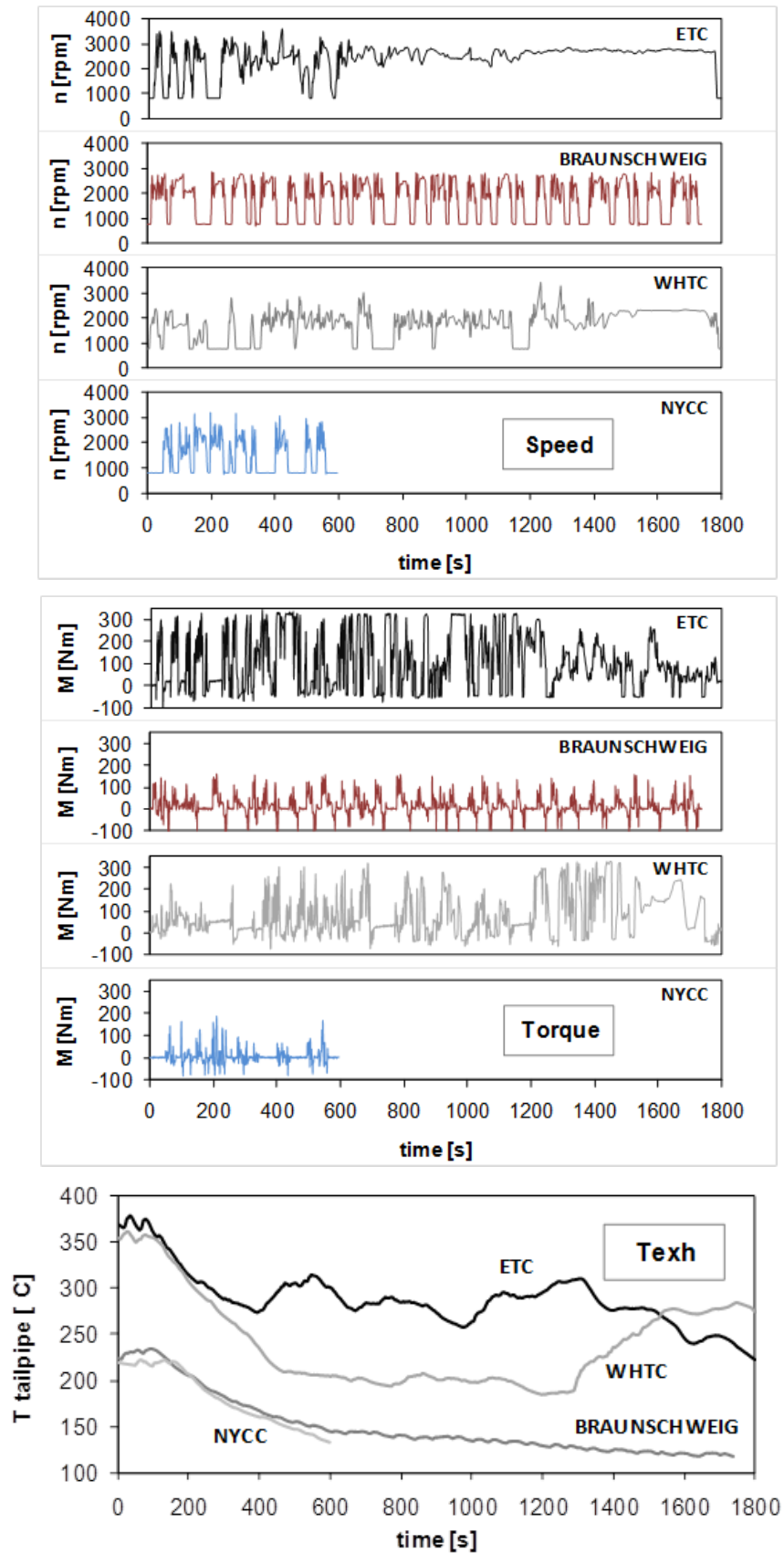


Figure 5. Speed, torque and exhaust temperature in the investigated dynamic test cycles

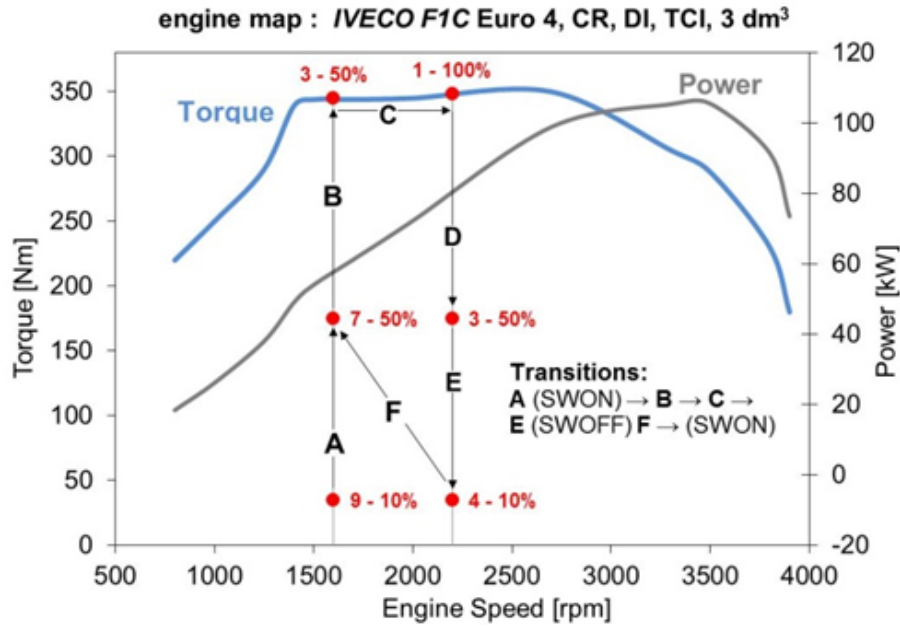


Figure 4. Engine map of the IVECO F1C engine and 6-points test for DPF and SCR-investigations

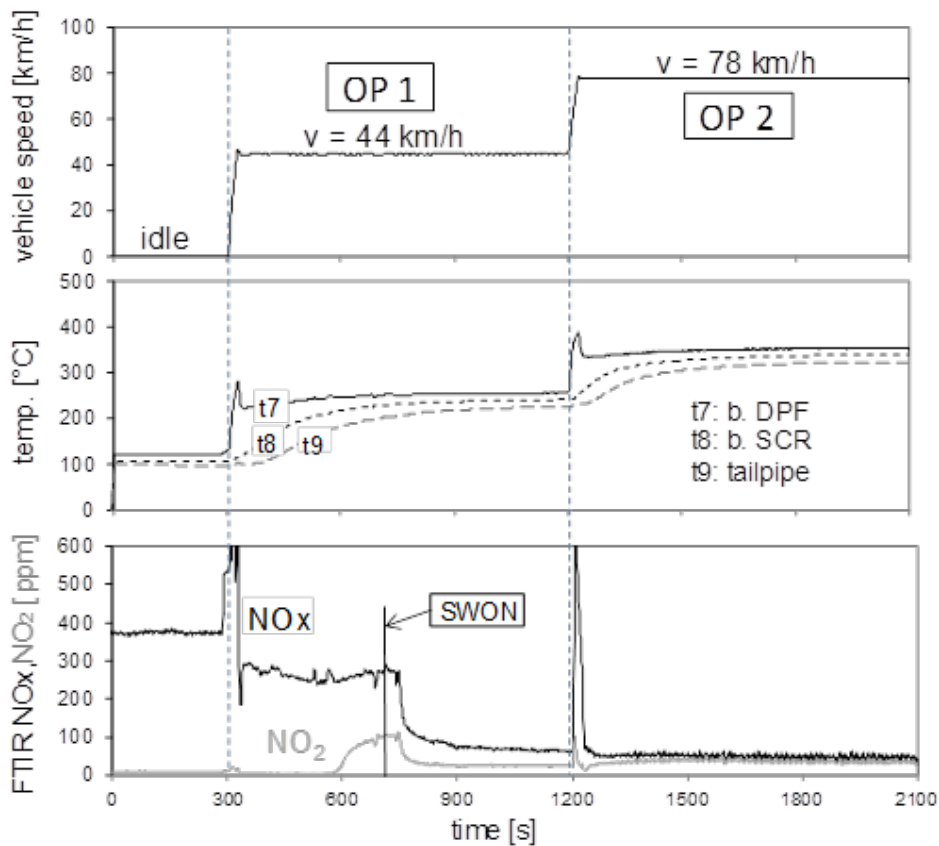


Figure 6. 2-points test for SCR quality control on HD chassis dynamometer

For the low-load cycles (NYCC and Braunschweig), the conditioning was: 5 min 1600 rpm / 165 Nm and 0,5 min idling.

For the dynamic testing on engine dynamometer the European Transient Cycle (ETC) is mostly used, Fig. 5.

All tests are performed in the warm state of the engine and of the exhaust system. As conditioning for a dynamic

test cycle the same cycle is used.

Different sampling positions (SP) were used to analyse exhaust compositions and temperatures in more detail (see Fig. 8):

- SP 0 sampling engine out w/o aftertreatment system
- SP 1 sampling engine out with aftertreatment



system

- SP 2 sampling after DPF (before urea dosing) with aftertreatment system
- SP 3 sampling at tailpipe with aftertreatment system.

This designation of sampling positions is used in the presented figures and in the discussion of results.

Another useful procedure is the urea switch-on test (SW). This test consists of a warm-up of the engine in a low load operation point, without any urea injection, and then increasing the engine load in a controlled way to an engine load where urea injection starts.

This quite simple procedure can be repeated during each warm-up of the engine and of the exhaust system. The SW-test can be joined with the OEM datalogging and it gives additional information on the extent of emissions and NO<sub>x</sub> reduction efficiency. The urea switch-on test is considered as a simple and reliable check of SCR quality on vehicles (chassis dynamometer).

For the preliminary control on vehicle (VPNT2) and for the final control (VPNT3) a simple 2-pts test is used, Fig. 6.

This test is easy to perform on a HD chassis dynamometer in a braking mode. The constant speeds in the range of 50 km/h and 80 km/h can be slightly adapted according to the best feasibility. The transition from idling to the OP1 provokes the increase of the exhaust temperatures and consequently urea switch-on (SWON), see Fig. 6. The transition from OP2 to idling can be used for retrofit systems as a repetitive switch-off (SWOFF).

During the tests of VERTdePN and TeVeNO<sub>x</sub> several road tests were performed. These results and experiences are reported separately.

### 2.3. Previous Results

Following most important findings from previous VERTdePN activities,[15] are:

- the investigated combined dePN systems (DPF+SCR) for dynamic engine application efficiently reduce the target emissions with deNO<sub>x</sub>-efficiencies up to 92% (if operated in the right temperature window) and particle number filtration efficiency up to 100%,
- the particle number filtration efficiency, which is verified at stationary engine operation, is valid also at the transient operation,
- the OEM NO<sub>x</sub>-sensors of the investigated systems are appropriate tools for the in-use control.

## 3. Tested Engine, Fuel, Lubricant

### 3.1. Test engine

Manufacturer: Iveco, Torino Italy  
 Type: F1C Euro 3 / Euro 4  
 Displacement: 3.00 Liters  
 RPM: max. 4200 rpm

Rated power: 100 kW @ 3500 rpm

Model: 4 cylinder in-line

Combustion process: direct injection

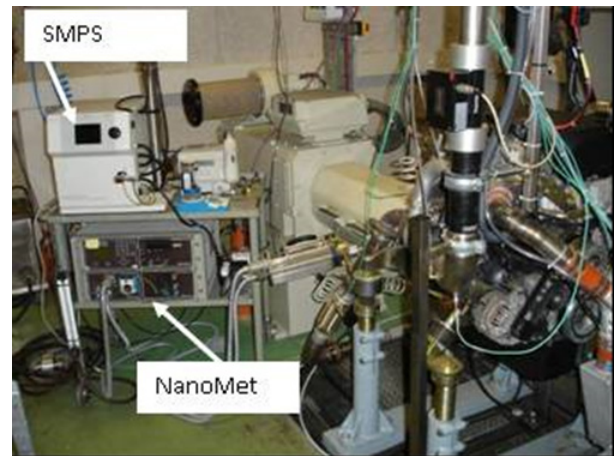
Injection system Bosch Common Rail 1600 bar

Supercharging: Turbocharger with intercooling

Emission control: none

Development period: until 2000 (Euro 3)

Fig. 7 shows the engine and the apparatus for nano-particle analytics SMPS & NanoMet in the laboratory for IC-engines, University of Applied Sciences, Biel-Bienne.



**Figure 7.** IVECO engine F1C and equipment for nanoparticle measurements in the engine room

### 3.2. Fuel

Following Diesel fuel was used for the research (Table 1):

**Table 1.** Fuel properties as per EU-standards

		<b>Diesel</b>
Density 15°C	g/ml	0.842
Viscosity at 40°C	mm <sup>2</sup> /s	2.0-4.5
Flash point		above 55°C
Cloud point		max -10°C
Filterability CFPP		max -20°C
Ash	%	max 0.010
Sulfur	ppm	<10
Cetane number		51
Calorific value	MJ/kg	42.7
C fraction	in %	86.7
H fraction	in %	13.3
O fraction	in %	0
Air <sub>min</sub>	kg/kg	14.52
Boiling range 10-90%°C		180-340

\*measured value

- Shell Formula Diesel fuel Swiss market summer quality (10 ppm S) according to SN EN 590

Table 1 represents the most important data of the fuel according to the standards.

### 3.3. Lubricant

For all tests a special lubeoil Mobil 1 ESP Formula

5W-30 was used.

Table 2 shows the available data of this oil,  
ACEA classes: C3, A3, B3/B4,  
API classes: SL / SM; CF

**Table 2.** Data of the utilized oil (\* analysis, others: specifications)

Property	Mobil oil	
Kinematic Viscosity 40°C	72.8	mm <sup>2</sup> /s
Kinematic Viscosity 100°C	12.1	mm <sup>2</sup> /s
Viscosity index	164	(--)
Density 15°C	0.85	kg/dm <sup>3</sup>
Pourpoint	-45	°C
Flamepoint	254	°C
Total Base Number TBN*	6	mg KOH/g
Sulfur ashes*	600	mg/kg
Sulfur*	2000	mg/kg
MG*	41	mg/kg
MO*	80	mg/kg
Zn*	900	mg/kg
Ca*	1100	mg/kg
P*	820	mg/kg

### 3.4. Engine Version Euro 4

In collaboration with the engine manufacturer the research engine version Euro 3 was upgraded to the version Euro 4. The new engine equipment consisted of:

- EGR valve (high pressure EGR), (see sketch Fig. 8)
- EGR cooler,
- throttle valve at intake,
- air mass flowmeter at intake,
- injectors,
- new engine calibration (ECU) for modifications of injection timing and injection mode (pre-/post- injections).

The principal influences on engine combustion and emissions are given through the:

- HP EGR regulated continuously in the engine map,
- further use of potentials of CR-injection system (pressure, timing, shaping, strategies).

The EGR is regulated by means of simultaneous positioning of the EGR-valve and of the throttle valve with air mass flow as guiding parameter. The total injected fuel quantity is adapted to the air mass flow.

The ECU-engine calibration is given in two versions: for HD- and for LD-application. In the present work only the HD-version was used.

The research laboratory received access to the ECU with the possibility of switching on/off EGR.

The engine version Euro 4 with EGR is shortly called E4 and the same version with EGR switched off is called E(4).

## 4. Measuring Set-up and Instrumentation

### 4.1. Engine Dynamometer and Standard Test Equipment

Fig. 8 represents the special systems installed on the engine, or in its periphery for analysis of the regulated and unregulated emissions.

Laboratory equipment employed:

- Dynamic test bench Kristl & Seibt with force transducer HBM T10F
- Tornado Software Kristl & Seibt
- Fuel flow measurement AIC 2022
- Air mass meter ABB Sensiflow P
- Pressure transducers Keller KAA-2/8235, PD-4/8236
- Thermo-couples Type K.

### 4.2. Test Equipment for Exhaust Gas Emissions

Measurement is performed according to the exhaust gas emissions regulations for heavy duty vehicles which are also in force in Switzerland (Directive 2005 / 55 / CE & ISO 8178):

- Standard exhaust gas components:
  - Horiba exhaust gas measurement devices  
Type: VIA-510 for CO<sub>2</sub>, CO, HCIR, O<sub>2</sub>,  
Type: CLA-510 for NO, NO<sub>x</sub> (this standard hot analyser with one reactor is marked in this report as “1 CLD”)
  - Amluk exhaust gas measurement device Type: FID 2010 for HCFID,
- NH<sub>3</sub> and N<sub>2</sub>O:

With SCR several unregulated and secondary pollutants can be produced. The slip of gaseous components such as ammonia NH<sub>3</sub> and nitrous oxide N<sub>2</sub>O was measured by means of:

- Siemens LDS 6 Laser Analyzer 7MB 6021, NH<sub>3</sub>
- Siemens ULTRAMAT 6E 7MB2121, N<sub>2</sub>O
- Eco physics CLD 822 CM hr with hot line for NO, NO<sub>2</sub>, NO<sub>3</sub>, NH<sub>3</sub> (this analyzer with two reactors is marked in this report as “2 CLD”)
- FTIR (Fourier Transform Infrared) Spectrometer (AVL SESAM) with the possibility of simultaneous, time-resolved measurement of approximately 30 emission components – among those validated are: NO, NO<sub>2</sub>, NO<sub>x</sub>, NH<sub>3</sub>, N<sub>2</sub>O.

#### 4.2.1. Particle Size Analysis

The particle size and number distributions were analysed with following apparatus, Fig. 7:

- SMPS – Scanning Mobility Particle Sizer, TSI (DMA TSI 3071, CPC TSI 3025 A)
- NanoMet – System consisting of:
  - PAS – Photoelectric Aerosol Sensor (EcoChem PAS 2000) indicates the carbonaceous total surface of the aerosol
  - DC – Diffusion Charging Sensor (Matter Eng. LQ1-DC) indicates the totale surface of the aerosol independently of the chemical properties
  - MD19 tunable minidiluter (Matter Eng. MD19-2E)



The nanoparticle results represented in this paper are obtained with sampling at tail pipe with MD19 and with thermoconditioner (300°C). The nanoparticulate measurements were performed at constant engine speed (warm) with SMPS and NanoMet.

During the dynamic engine operation NanoMet and CPC were used.

## 5. (DPF+SCR) Systems

A combined system consisting of a DPF upstream of the urea dosing point and a vanadium-based SCR catalyst downstream of the filter was used (as in Fig. 8).

A mixing tube of 1.0m, without mixer was used between the DPF and SCR.

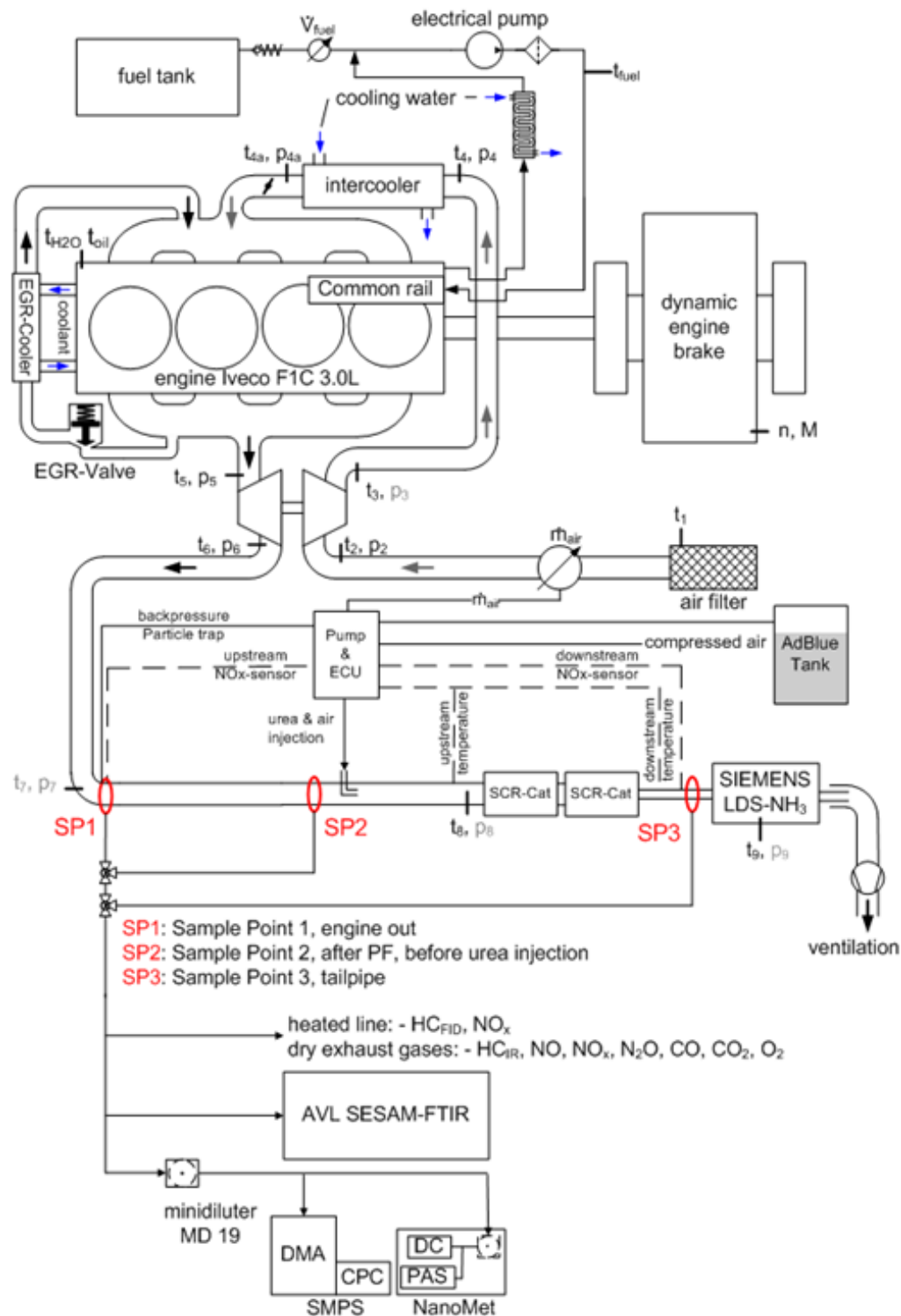


Figure 8. Engine dynamometer and test equipment

In certain experiments, an ammonia slip catalyst was used at the tail pipe. The combined DPF+SCR system is designed for transient application with an electronic control unit, using the signals of: air flow, NO<sub>x</sub> before/after system, and temperatures before/after the SCR catalyst.

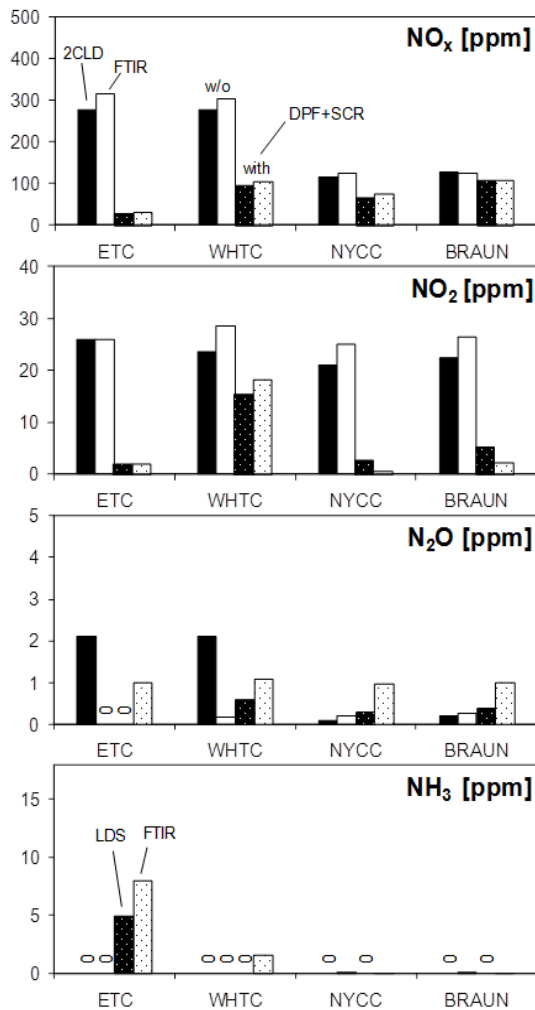
Following three variants were investigated:

- PF1 + SCR
- PF1 + SCR + slip cat.
- PF3 + SCR

PF1 is a catalytically coated Cordierite DPF, PF3 is a uncoated SiC DPF with FBC (40 ppm Fe, double dosing for testing purposes).

Both DPF's are VERT-approved with average NP filtration efficiencies FE > 99%.

A feed factor of  $\alpha = 0.9$  was generally used (during investigations on secondary nanoparticles in Fig. 11 & 12 a feed factor  $\alpha = 1.0$  was applied).



**Figure 9.** Integral average emission values in dynamic cycles, PF1+SCR,  $\alpha=0.9$

## 6. Results

Following the results of some specific research subjects,

which may complete or enlarge the present state of knowledge, will be shortly presented.

### 6.1. Different Test Cycles

Tests were performed with different driving cycles with non limited engine map (NEM).

The exhaust temperatures at the tailpipe over cycle time for all investigated test cycles are given in Fig. 5 together with engine speed and torque.

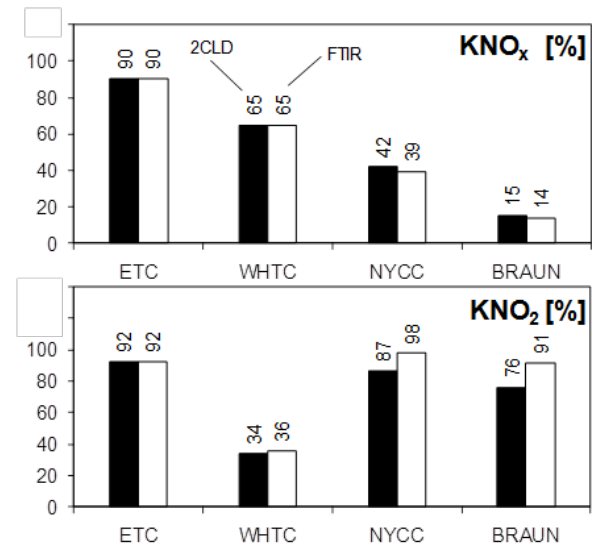
Fig. 9 summarizes average exhaust concentrations for the different test cycles and Fig. 10 shows the conversion efficiencies in different driving cycles.

In Table 3 the fuel consumption and the average exhaust temperatures in all driving cycles are represented.

The New York City Cycle (NYCC) and the Braunschweig cycle (BRAUN) are low-load cycles, which were developed in those cities and represent the city bus driving.

Exhaust temperatures in these low load cycles are too low (< 200°C) to enable the full working potential of the SCR-system. The urea injection is usually cut off at  $t_{exh}$  190-200°C to prevent the deposition of solid matter in the system.

Average NO<sub>x</sub> emissions in the low load cycles are lower too, but there is also a poor NO<sub>x</sub>-conversion due to the urea dosing strategy.



**Figure 10.** Comparison of reduction efficiencies in dynamic cycles, PF1+SCR,  $\alpha=0.9$

**Table 3.** Fuel consumption and average exhaust temperatures in different driving cycles, PF1+SCR,  $\alpha=0.9$

	[l/test]	ETC		WHTC		NYCC		Braunschweig	
		1800 s		1800 s		598 s		1740 s	
		OEM	DPF+SCR	OEM	DPF+SCR	OEM	DPF+SCR	OEM	DPF+SCR
Fuel		4.15	4.30	2.58	2.64	0.25	0.26	1.02	1.05
T Engine Out	[°C]	282	310	247	261	144	151	149	156
T Tailpipe	[°C]	248	291	210	241	139	182	127	150

N<sub>2</sub>O emissions are generally very low (< 2 ppm) and NH<sub>3</sub> was found only in ETC with SCR NH<sub>3</sub> but its concentrations are typically < 8 ppm at urea feed factors of 0.9.

Comparing different measuring techniques as shown in Figures 9 & 10

one can conclude:

- good correlation of NO<sub>x</sub> & NO<sub>2</sub> measured with FTIR and with two CLD,
- good correlation of NH<sub>3</sub> measured with FTIR and with LDS,
- poor correlation of N<sub>2</sub>O measured with FTIR and with ULTRAMAT; This is the case, because N<sub>2</sub>O concentrations are low – mostly at the detection limits of the analytical devices,
- the relative values, like conversion efficiencies K<sub>x</sub> are well correlated with all measuring methods.

These conclusions are supported from several other internal investigations not presented in this paper.

Finally we conclude that the investigated SCR-system has low NO<sub>x</sub> conversion efficiencies of 40 and 15% in both low-load city driving cycles. This has been observed before, when comparing the NO<sub>x</sub> emissions of the high-load-ETC (non-limited engine map NEM) with those in the low-load-ETC (limited engine map LEM[15]).

### 6.2. Secondary Nanoparticles

The production of secondary nanoparticles downstream of the urea dosing point was noticed earlier[15]. Such nanoparticles are possibly formed during the deNO<sub>x</sub> process. The chemical composition of these particles is yet unknown and further investigations are on the way. They may consist of unreacted urea or derivatives thereof such as cyanuric acid, or salts from acidic and basic compounds such as ammonium nitrite, nitrate, and sulfates and others.

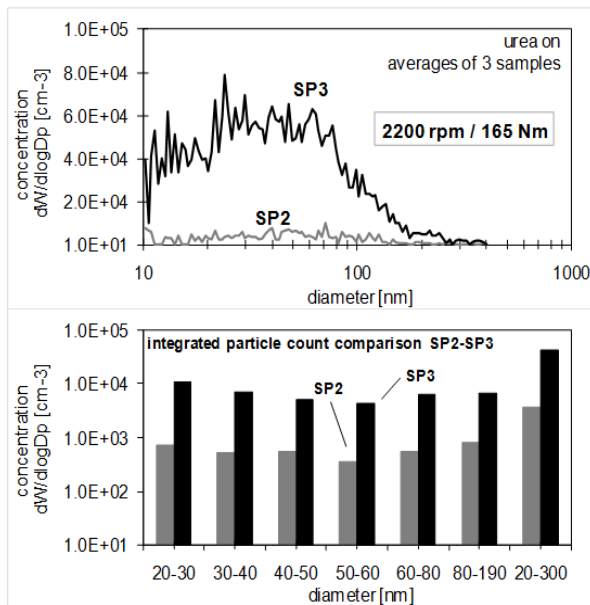


Figure 11. SMPS-size spectra at SP2 & SP3, PF1+SCR,  $\alpha=1.0$

In the present status of these investigations, the objective was to systematically study the number and particle size of these secondary NP by means of PM, SMPS and NanoMet at sampling positions SP2 & SP3, (SP2 ... after DPF & before urea dosing, SP3 ... after SCR).

SMPS measurements after the DPF (SP2) and after the SCR (SP3) confirmed the presence of secondary nanoparticles in all load stages of the 4-points test.

Fig. 11 shows an example of the SMPS size distributions and the integrated particle number in different parts of the size spectra at 2200 rpm / 50%. The increase of particle mass emissions between SP2 and SP3 as a result of a secondary particles formation was noticed as well.

Fig. 12 represents the particle mass PM, the mass-related filtration-efficiency PMFE and the particle number related filtration efficiency PCFE in all operating points and at all sampling positions SP0, SP2 & SP3. At two OP's, there is a little, but consistent increase of PM between SP2 and SP3. This causes a significant reduction of PMFE, but no significant reduction of PCFE. This indicates that these secondary particles mainly contribute to particle mass but not much to particle numbers.

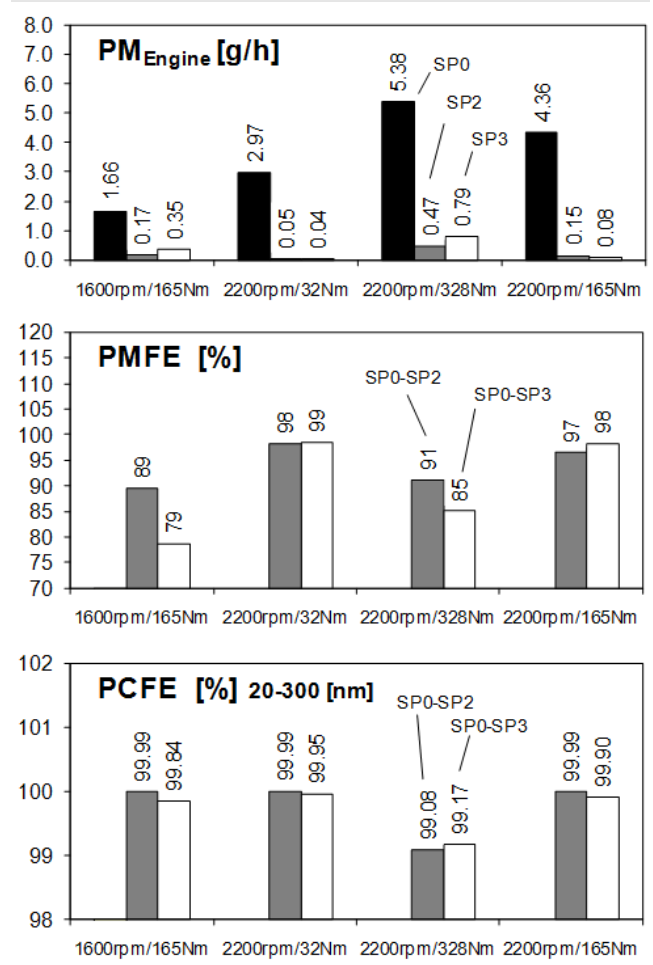
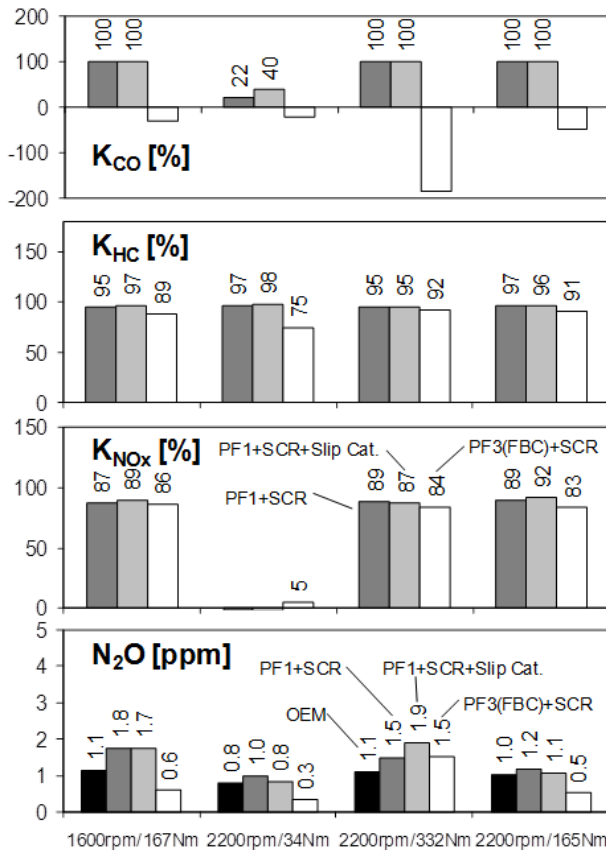


Figure 12. Particle mass and counts reduction in the 4 points test, PF1+SCR,  $\alpha=1.00$

It can be summarized that the presence of secondary nanoparticles after the SCR-system was confirmed. In some cases, secondary NP contribute to an increase of the particle mass.

### 6.3. Comparisons of Different Aftertreatment Systems

Fig. 13 represents the conversion efficiencies of regulated gaseous components with the investigated aftertreatment systems in the 4- points test.



**Figure 13.** Conversion efficiencies of regulated pollutants and N<sub>2</sub>O in the 4-points test with different DPF+SCR systems

With uncatylsed PF3, negative CO conversion efficiencies were noticed, mostly at the highest OP, with the highest backpressure. HC is also converted in a vanadium-based SCR-catalyst, and there are as high conversion efficiencies K<sub>HC</sub> in combination with the PF3 system (FBC).

NO<sub>x</sub> is not reduced at the lowest OP due to urea switch-off. At higher OP's, with active SCR catalyst, there is a tendency of slightly lower K<sub>NOx</sub>-values with the fuel-borne catalyzed PF3. The principal reason is the lower NO<sub>2</sub> concentration after this filter.

Emissions of nitrous oxide N<sub>2</sub>O are very low (< 2 ppm) and deviate less than 1 ppm from values without aftertreatment (OEM).

Emissions of ammonia NH<sub>3</sub> are not represented, since they are generally at zero level, except of one value (10 ppm) for the PF1 (cat.) + SCR at the highest OP (2200 rpm / 332 Nm).

Fig. 14 compares the different exhaust aftertreatment systems in the steps-test at 2200 rpm. Following effects were observed:

- reduction of NO<sub>x</sub> at  $t_{\text{exh}} \geq 190^\circ\text{C}$ ,
- emission of NO<sub>2</sub> increase with PF1 (cat) in the  $t_{\text{exh}}$  range 240-340°C
- emission of NH<sub>3</sub> increase up to 8 ppm with PF3 in the  $t_{\text{exh}}$  range 240-340°C
- emission of NH<sub>3</sub> increase up to 10 ppm with PF1 in the  $t_{\text{exh}}$  range < 390°C
- NO<sub>x</sub> conversion starts at slightly lower  $t_{\text{exh}}$  with PF1 than with the fuel-borne catalyzed PF3 ( $\Delta t \approx 10\text{-}15^\circ\text{C}$ ),
- similar maximum conversion efficiencies K<sub>NOx</sub> were found for both systems.

### 6.4. Demonstration of NH<sub>3</sub>-storage

For the electronic control of AdBlue dosing of SCR systems several physico-chemical processes have to be considered, like: Ad-Blue mixture preparation, transformation of AdBlue in Ammonia (thermolysis & hydrolysis) and store /release of Ammonia in the SCR-catalyst. The best AdBlue dosing is reached, when with the variable engine-out NO<sub>x</sub> emissions in the engine map and also in transient engine operation the maximum NO<sub>x</sub> conversion rates with possible no NH<sub>3</sub>-slip are reached.

The effects of NH<sub>3</sub>-storage were studied and an example of the most significant results is represented here. Fig. 15 shows urea switching on and off at constant low-load OP 2200 rpm / 80 Nm. The objective was to leave enough time to stabilize the emissions after urea switch on (SWON). The diagram represents 1 hour run.

In the short time (approximate 20s) after SW on there is a “quick” reduction of approximate  $\frac{3}{4}$  of initial NO<sub>x</sub> and totality of NO<sub>2</sub>. NO<sub>2</sub> supports the quicker deNO<sub>x</sub>-reactions and from the instant, when NO<sub>2</sub> is consumed the further NO<sub>x</sub>-reduction slows down – the last quarter of NO<sub>x</sub>-reduction takes the time “t<sub>2</sub>” (approximate 13.3 min).

After the time “t<sub>1</sub>” (approximate 7.5 min) NH<sub>3</sub> starts to increase. It is no more totally needed for deNO<sub>x</sub>-reactions, part of it is stored in the SCR-catalyst, part of NH<sub>3</sub> is available for the negligible production of Formic Acid and the rest is emitted. It can be assumed that after time “t<sub>3</sub>” (approximate 32 min), when the NH<sub>3</sub>-emission level stabilizes, the storage capacity is filled out.

The increase of NO<sub>x</sub>- and NO<sub>2</sub>- concentrations (CLD) after time “t<sub>2</sub>” is due to the presence of NH<sub>3</sub> and to the crossensitivity of CLD against NH<sub>3</sub>.

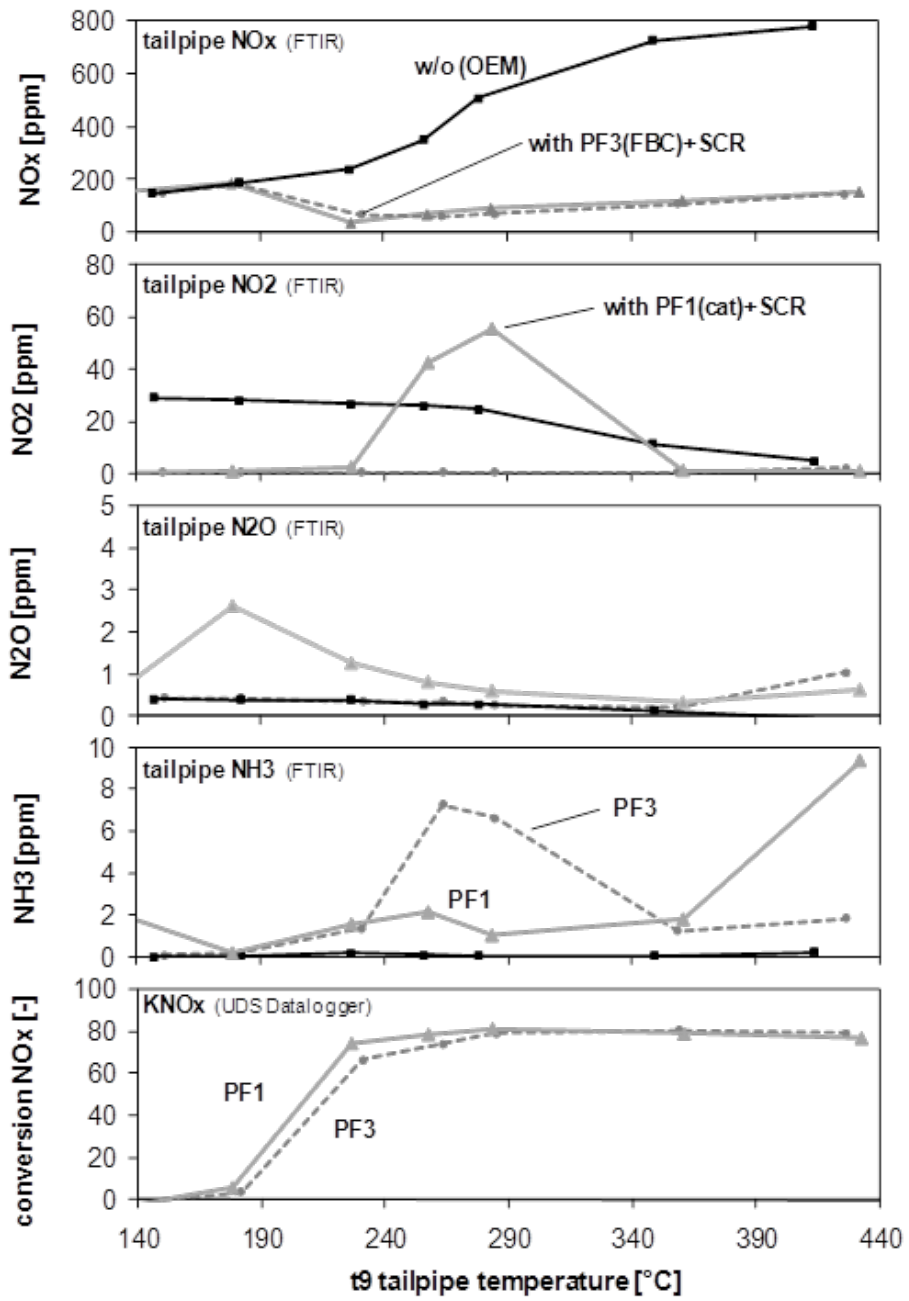
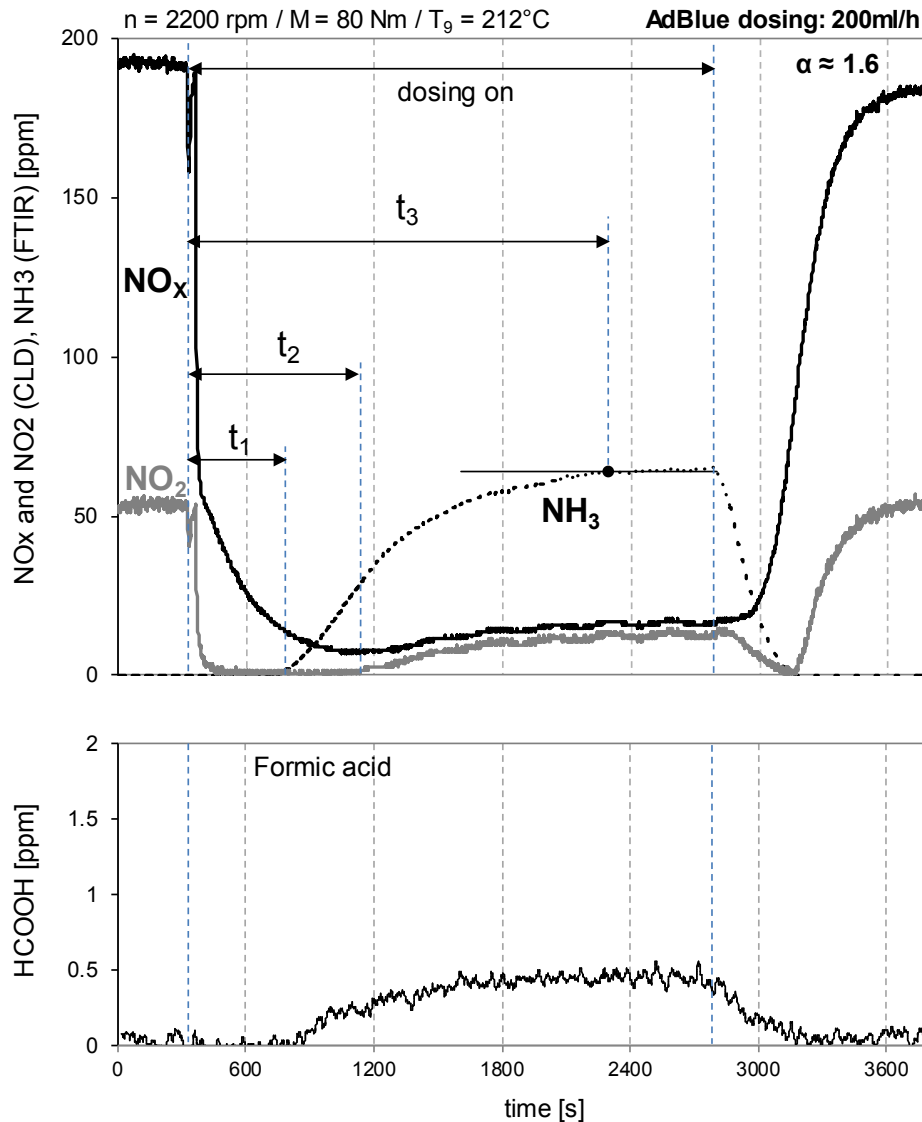


Figure 14. Reference - PF1(cat.)+SCR - PF3(FBC)+SCR, 60s average values of stationary operating



**Figure 15.** Urea dosing SWON & SWOFF at low-load OP. DPF & SCR-catalyst, AdBlue ; IVECO FIC, E(4), ULSD

Some tests were performed, when after  $\text{NH}_3$ -storage period and after SWOFF at lower OP1 (80 Nm) time was left to stabilize the emission and after that a load jump to the higher OP2 (250 Nm) was performed, still without RAI (see Fig. 16). This provoked a sudden increase of  $\text{NO}_x$ -emissions, but due to the increasing temperature and some release of  $\text{NH}_3$  there is a break-down of  $\text{NO}_x$ -emissions during certain time period after the maximum of  $\text{NO}_x$ , Fig. 16. This was a clear and repetitive demonstration of  $\text{NH}_3$ -release. Similar tests were performed without previous urea dosing and consequently without  $\text{NH}_3$ -storage.

Fig. 16 shows  $\text{NO}_x$ -emission at load-jump with and without  $\text{NH}_3$ -release. The lower  $\text{NO}_x$ -values are an effect of  $\text{NH}_3$ -release. This is particularly visible, when there is significant increase of exhaust temperature. When the temperature starts to stabilize, the  $\text{NH}_3$ -release stops.

### 6.5. Cleaning of the System

Dependent on temperature different solid residues can be created from the injected urea. According to [16] these are:

- 130-230°C crystals of Urea & Biuret
- 200-350°C Cyanuric Acid
- 200-450°C Ammelid, Ammelin, Melanin

Urea crystallization typically takes place at low temperatures. If there is insufficient mixture preparation and wall wetting, than it is very likely that crystallization happens. The activation of RAI at 200°C is first of all to prevent the crystallization at lower temperature.

At higher temperatures other residues can be created. Some of them can be melted and evaporated and take part on the chemical reactions. Some others are difficult to remove even at high temperatures.

In a research work on one of the investigated SCR-systems different dosings (and also overdosings) of AdBlue happened. In the following 4-pts test exceptionally high peak values of some unregulated emissions resulted (Formaldehyde, Hydrocyanic Acid and Isocyanic Acid).



After “cleaning” of the system – heating up at full load during ½ hour, without urea dosing – the test was repeated.

Fig. 17 shows the results: after cleaning there are no significant emissions of the critical components.

The cleaning is recommended at the beginning of every basic test on the engine dynamometer.

**6.6. Results at Different Steps Tests**

The validation of  $K_{NOx}$  in 4-pts or in 6-pts test is represented in Fig. 18.

At low-load OP's with deactivated RAI the  $NO_x$  reduction rate is near to zero. At higher OP's the full efficiency is attained.

Principally there is no difference for the validation to

apply any of those steps tests. For reasons of comparability it is recommended to always use the same test schedule for the same tested system.

In this schedule the same duration of “cleaning” and the same sequence and duration of the load steps should be applied.

**6.7. Secondary Emissions with Cu- & Fe-zeolites**

Secondary emissions and traces are investigated as a part of VERT / LRV quality verification procedure for DPF- and SCR-systems. The sampling and analytics are performed by a specialized team of EMPA Analytical Laboratory for Organic Chemistry. There are 3 groups of secondary emissions, which are known as very carcinogen and toxic:

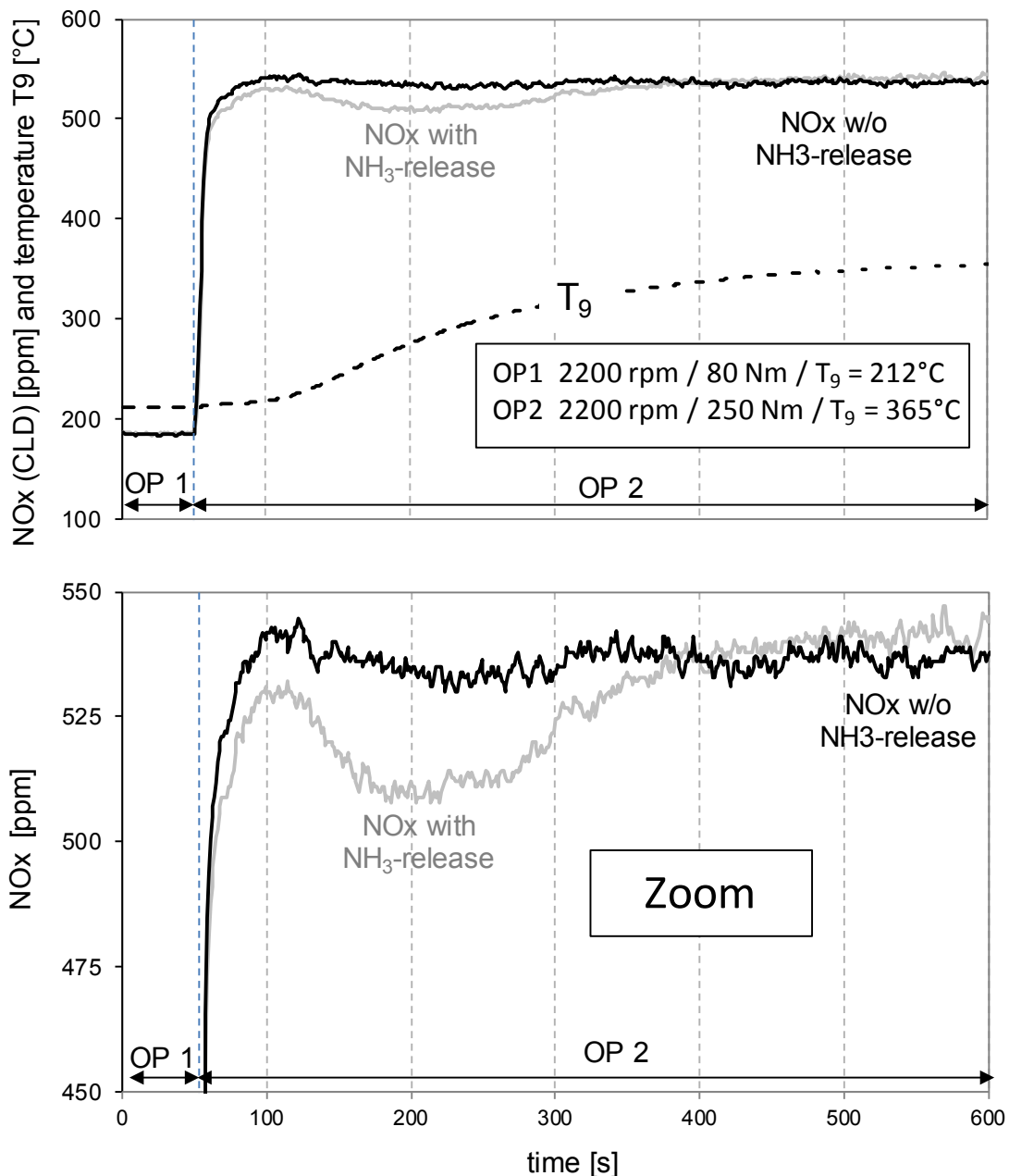


Figure 16. Ammonia storage: NO<sub>x</sub> at load-jump with / without NH<sub>3</sub>-release. DPF & SCR-catalyst, AdBlue dosing off; IVECO F1C, E(4), ULSD

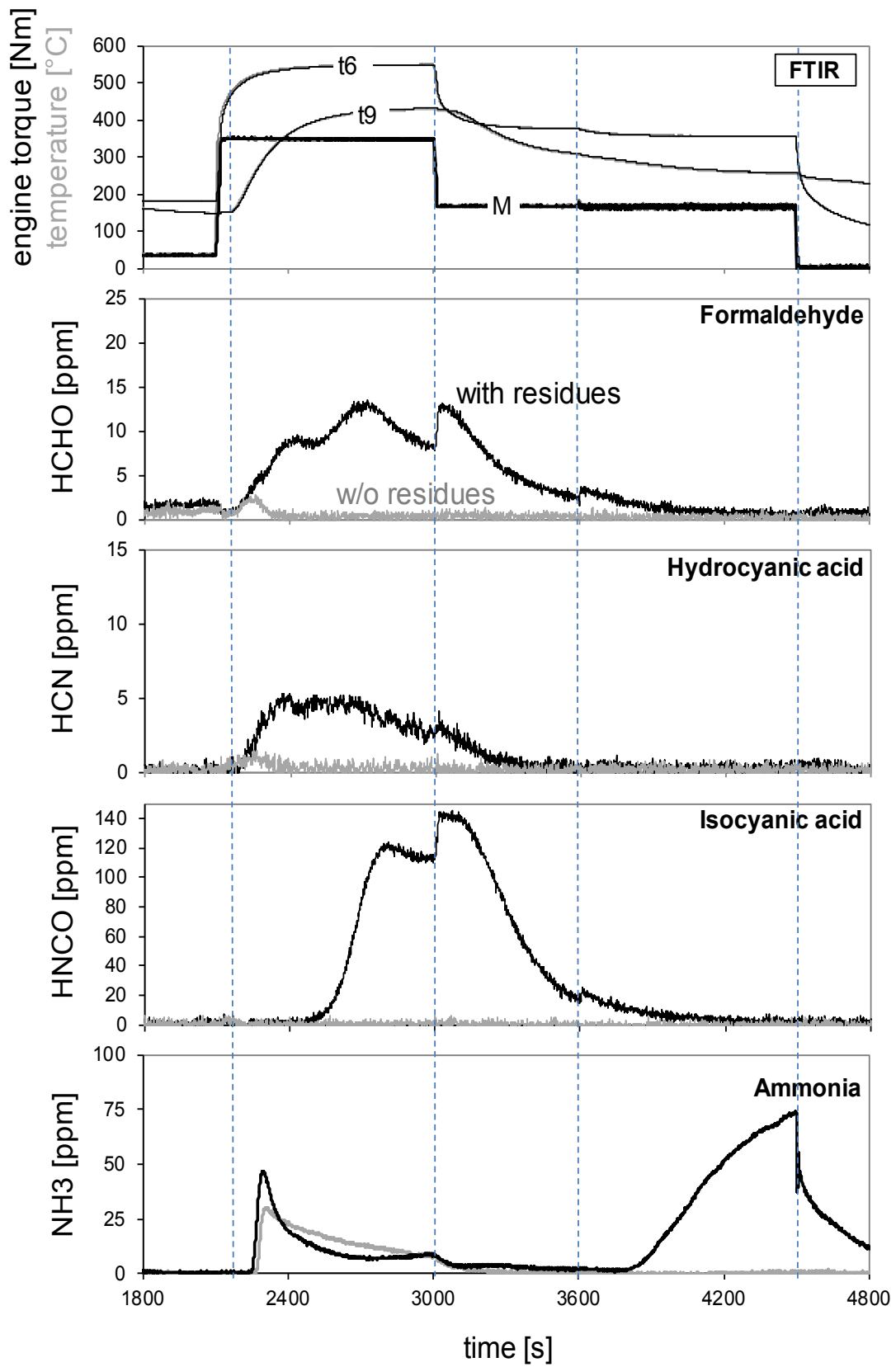
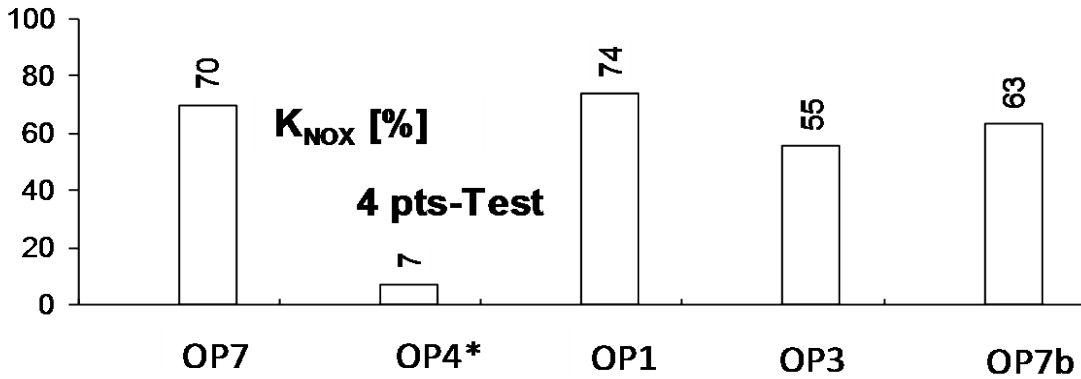
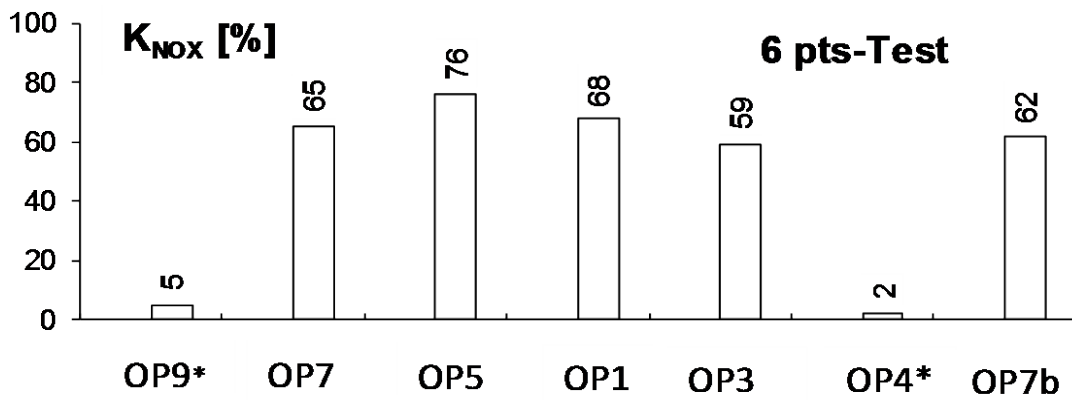


Figure 17. Comparison of Unregulated Emissions in 4 Pts-TEST with & without residues, SCR with AdBlue Injection, Iveco FIC E(4); ULSD



OP4\*: SCR not active ( $T < 200^{\circ}\text{C}$ )



OP9\*; OP4\*: SCR not active ( $T < 200^{\circ}\text{C}$ )

Figure 18. Validation of the NO<sub>x</sub>-reduction rate in 4-points- and in 6-points test

### 2,3,7,8-PCDD/Fs (TEQ-Sum)

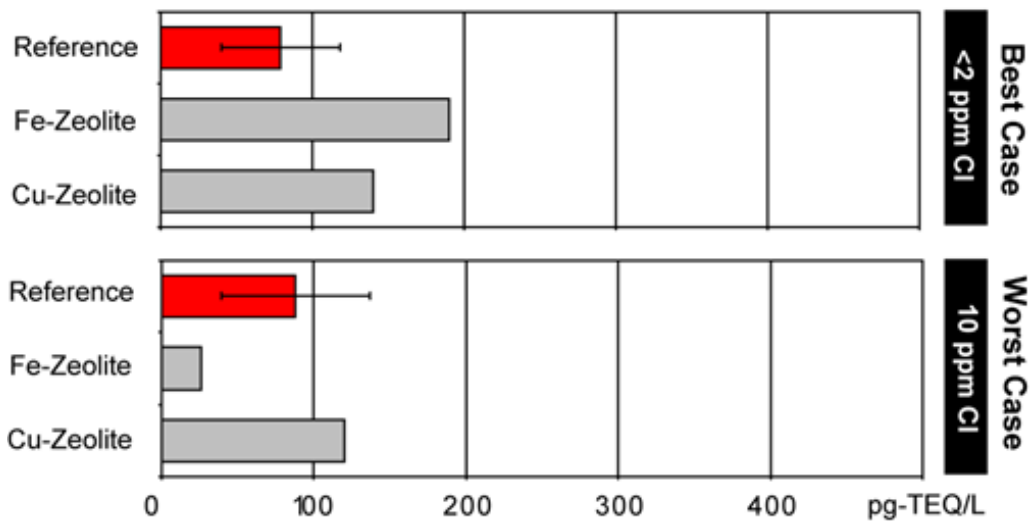
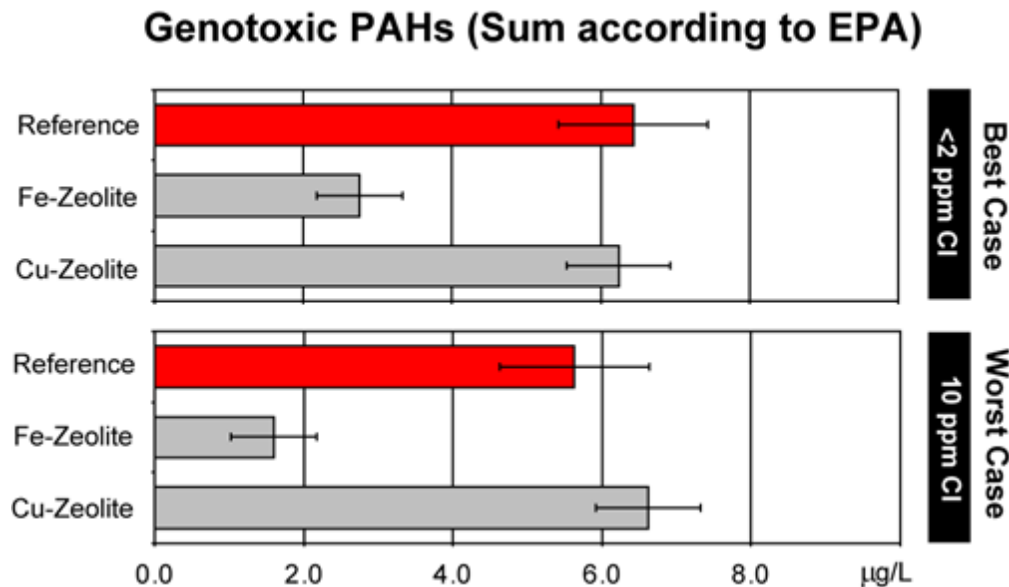


Figure 19. Emission factors for the weighted sum of 2,3,7,8-substituted PCDD/Fs (TEQ sum) in pg/L. Emissions of iron- and copper-zeolites are compared with engine-out emissions (reference) for fuels with chlorine levels of <2 and 10  $\mu\text{g/g}$



**Figure 20.** Emission factors for the sum of carcinogenic PAHs (EPA) in  $\mu\text{g/L}$ . Emissions of iron- and copper-exchanged zeolites are compared with engine-out emissions (reference) for fuels with chlorine levels of <2 and 10  $\mu\text{g/g}$

- Polycyclic Aromatic Hydrocarbons PAH, 12 substances \*), [ $\mu\text{g/L}$ ],
- Nitro-PAH, 22 substances \*), [ $\mu\text{g/L}$ ],
- Polychlorinated Dibenzodioxins PCDD and Polychlorinated Dibenzofurans PCDF, 33 substances \*), [ $\text{pg/L}$ ],

VERT Secondary Emissions Tests were performed with urea based SCR systems (without DPF).

One of the systems used a Cu-zeolithe and the other one a Fe-zeolithe as SCR-catalyst.

Samples were taken continuously proportional to the actual gas flow (aliquot) from the undiluted hot exhaust gas. A volume of typically 5-8  $\text{Nm}^3$  was collected through a validated sampling arrangement over 200 Minutes driving cycle time (2 full ISO 8178/4 C1-cycles).

The sampling device consists of a sampling sonde, cooler, condensate separator, filter stage and two-stage adsorber unit (XAD-2). The sampling apparatus, made from glass is extensively cleaned and heated to high temperatures prior to each sampling (heating in steps up to  $450^\circ\text{C}$ ). Using marked PCDD-standards in this investigation the recovery was found to be  $65 + 10\%$ .

The same sample was used to determine the PCDD/F as well as the PAH.

PCDD/F-analysis was performed by separating the toxicologically relevant PCDD/F isomers using gas chromatography followed by high resolution mass spectrometry.

After chromatographic work-up quantitative analysis of the PAH and Nitro-PAH were also performed using capillary gas chromatography high resolution mass spectrometry.

### 6.8. PCDD/F-Analysis

The following graph, Figure 19, shows the overall results.

PCDD/F emissions (TEQ sum) of the engine accounted for  $80 \pm 40$   $\text{pg-TEQ/L}$  with reference fuel (best case) and  $90 \pm 50$   $\text{pg-TEQ/L}$  with chlorine-doped fuel (worst case). As shown in Fig. 19, PCDD/F emissions of configurations with iron- and copper-zeolites are at or below respective engine-out emissions. Even under worst case conditions, when using chlorine-doped fuels, no substantial increase of the PCDD/F emissions was noticed.

### 6.9. PAH-Analysis

PAH were analysed from the same sample as the PCDD/F.

The following graph, Fig. 20, summarizing the data for the carcinogenic PAH according to the IARC-definition shows the overall results.

The analysis of the broad class of polycyclic aromatic hydrocarbons (PAHs) was limited to those 4- to 7-ring PAHs, which are rated to be carcinogenic to humans by the US EPA. Among these compounds, we quantified emissions of benzo(a)an-thracene, benzo(b)fluoranthene, benzo(k)fluoran-thene, benzo(a)pyrene, di-benzo(a,h)anthracene, indeno (1,2,3-c,d)pyrene, and chrysene.

Fig. 20 illustrates the effects on genotoxic PAH emissions when applying both deNO<sub>x</sub> systems. Compared to the reference configurations, the iron-zeolite reduced the emissions of carcinogenic PAHs by about 70%, whereas the copper-zeolite did not convert these PAHs. The chlorine-doped fuel had no substantial effects on emissions of these PAHs.

### 6.10. Quality of a Retrofit System after 1000h

DPF+SCR) system retrofitted on bus was measured on a HD chassis dynamometer before and after the durability test of 1000h road application. Table 4 summarizes the most important results: there is a negligible reduction of the deNO<sub>x</sub>-efficiency and there are no significant secondary

emissions of NH<sub>3</sub> & N<sub>2</sub>O. It can be stated, that there is no deterioration of the SCR-system after 1000h. The used DPF was VERT/OAPC approved and always had the excellent particle counts filtration efficiency of 99.6%.

**Table 4.** Efficiency and non-regulated emissions of a retrofit VSCR system before and after 1000h field test; overall average PCFE = 99.6%

VERT	$\alpha$	TEST		SCR	NH <sub>3</sub>	N <sub>2</sub> O
VPNT2 Vehicle chassis- dynamo meter.	0.75	44 km/h	stat.	82	0	2
	0.75	78 km/h	stat.	92	8-26	2
	0.75	SWON	stat.	79	-	-
	0.85	SWON	stat.	97	-	-
	0.75	FIGE	dyn	-	5	2
<b>Averages (before)</b>				<b>87.50</b>	<b>11.00</b>	<b>2.00</b>
VPNT3 1000h Vehicle chassis- dynamo meter.	0.75	44 km/h	stat.	81	0	2
	0.75	78 km/h	stat.	90	5-21	2
	0.75	SWON	stat.	76	-	-
	0.85	SWON	stat.	85	-	-
	0.75	FIGE	dyn	63-9 2	5.2	1.7
<b>Averages (after)</b>				<b>81.9</b>	<b>9.10</b>	<b>1.90</b>

## 7. Conclusions

Following conclusions can be pointed out:

VERTdePN

- the procedures for the quality verification of SCR-, or (DPF+SCR) - systems are developed and confirmed,
- these test procedures on HD-chassis dynamometer and on-road are useful for OEM- and for retrofit systems,
- engine dynamometer testing enables the deepest insight in the investigated system concerning: secondary - and non-legislated emissions, variations of feed factor, analysis on different sampling positions and at specific engine operating conditions (like legal test procedures),
- testing on HD-chassis dynamometer can partially replace the engine dynamometer depending on the possibilities of the installation,
- testing of SCR-systems on vehicle is important, because of urea dosing, urea mixing and electronic control,
- the filtration efficiency of a DPF is independent of the operating condition (except of regeneration period, or passing over the maximum space velocity),
- the NO<sub>x</sub> reduction efficiency of SCR-systems is dependent on the operating conditions, because of the optimal temperature window of the SCR-catalysis; at the conditions with exhaust temperature below 200°C the urea dosing is stopped.

### 7.1. Different test cycles

- in the low-load transient cycles (NYCC & Braunschweig), there are rather low NO<sub>x</sub> conversion efficiencies due to low exhaust gas temperatures and limited urea dosing,
- the SCR-systems in the tested configuration has

moderate deNO<sub>x</sub> efficiencies at low-load city driving, but substantial deNO<sub>x</sub> efficiencies were found at higher engine loads e.g. at highway conditions,

- the average NO<sub>x</sub> conversion rate at transient operation strongly depends on the operation load profile, on the exhaust gas temperature and the resulting urea dosing control.

### 7.2. Secondary Nanoparticles

- secondary nanoparticles are produced due to urea injection, they nevertheless do not impact significantly the overall filtration efficiency of the system (here: DPF upstream & SCR downstream, differences of PCFE in the range of 0.1%).

### 7.3. Different Aftertreatment Systems

- the system with catalyzed DPF (upstream) attains slightly higher overall deNO<sub>x</sub>-efficiencies due to NO<sub>2</sub>-production in the DPF,
- for the investigated systems there are no critical emissions of unregulated components, NH<sub>3</sub> & N<sub>2</sub>O.

### 7.4. NH<sub>3</sub>-storage

- at low-load OP1 with RAI there is a storage of NH<sub>3</sub> in the SCR catalyst,
- after load increase to OP2 there is NH<sub>3</sub> release due to the increasing exhaust temperature; this release is indicated by transitory NO<sub>x</sub>-reduction effect without RAI.

### 7.5. Testing

- cleaning of the SCR-system from the possible residues (FL w/o RAI) is recommended at the beginning of test on the engine dynamometer,
- it is possible to use different stationary steps tests – for reasons of comparability it is recommended to always use the same test schedule for the same tested system,
- there were no significant secondary emissions of traces (PCDD/F), or genotoxic PAHs with all investigated SCR-catalysts (V, Cu & Fe),
- an excellent quality of a retrofitted VSCR-system after 1000h durability test was confirmed.

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## Abbreviations

		LRV	Luftreinhalteverordnung (see OAPC)
		ME	Matter Engineering
		MD19	heated minidiluter
AEEDA	Association Européenne d'Experts en Dépollution des Automobiles, Belgium		
AFHB	Abgasprüfstelle FH Biel, CH		
Air min	stoichiometric air requirement		
ASTRA	Amt für Strassen, CH, Swiss Federal Office of Roads	NanoMet	NanoMetnanoparticle summary surface analyser (PAS + DC + MD19) PAS + DC + sampling & dilution unit
BAFU	Bundesamt für Umwelt, CH (Swiss EPA, see FOEN)	NP	nanoparticles < 999 nm (SMPS range)
BAT	best available technology	NYCC	New York City Cycle
BRAUN	Braunschweig test cycle	OAPC	Swiss Ordinance on Air Pollution Control (LRV)
CDI	Common Rail Diesel Injection	OBD	on-board diagnostics
CFPP	cold filter plugging point	OEM	original equipment manufacturer
CLD	chemoluminescence detector	OP	operating point
CNC	condensation nuclei counter	PAH	polycyclic aromatic hydrocarbons
CPC	condensation particle counter	PAS	Photoelectric Aerosol Sensor
CPK	supplier of datalogging equipment	PC	particle counts
CR	common rail	PCDD/F	polychlorinated dibenzodioxines & furanes
DC	Diffusion Charging Sensor	PCFE	particle counts filtration efficiency
dePN	de Particles + deNO <sub>x</sub>	PM	particulate matter, particle mass
DI	Direct Injection	PMFE	particle mass filtration efficiency
DMA	differential mobility analyzer	PSD	particle size distribution
DPF	Diesel Particle Filter	RAI	reduction agent injection
ECU	electronic control unit	SCR	selective catalytic reduction
EGR	exhaust gas recirculation	SMPS	Scanning Mobility Particle Sizer
EMPA	Eidgenössische Material Prüf- und Forschungsanstalt (Swiss Federal Laboratories for Testing & Research)	SP	sampling position
EPA	Environmental Protection Agency	SUVA	Schweiz. Unfallversicherungs-Anstalt (Swiss Occupational Insurance)
ETC	European Transient Cycle	SW	urea switch on/of
FBC	Fuel born catalyst	SWON	urea switch on
FE	filtration efficiency	SWOFF	urea switch off
FID	flame ionization detector	TC	thermoconditioner.
		TCI	turbocharging & intercooling
FIGE	a non-standardized vehicle version of ETC, created by Forschungsinstitut für Geräusche und Erschütterungen	TeVNOx	Testing of Vehicles with NOx reduction systems
FL	full load	TEQ	toxicity equivalence
FOEN	Federal Office of Environment (BAFU)	TTM	Technik Thermische Maschinen
FTIR	Fourrier Transform Infrared Spectrometer	ULSD	ultra low sulfur Diesel
HD	heavy duty		
HP	high pressure	VERT	Verminderung der Emissionen von Realmaschinen in Tunnelbau Verification of Emission Reduction Technology
IARC	International Association of Research on Cancer		
		VERTdePN	VERT DPF + VERT deNO <sub>x</sub>
KNOx	NOx reduction rate	VPNT1	VERTdePN Test 1
LD	low duty	VPNT2	VERTdePN Test 2 - field durability 1000h
LDS	Laser Diode Spectrometer (for NH <sub>3</sub> )	VPNT3	VERTdePN Test 3 - check after field test chassis dynamometer
LEZ	low emission zones		



VPNSET	VERTdePN secondary emissions test - engine dynamometer
VSCR	Vanadium-based SCR
VSET	VERT Secondary Emissions Test
WHTC	worldwide HD transient cycle
$\alpha$	feed factor of urea dosing; ratio: urea injected / urea stoichiometric; calculated by the ECU.

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