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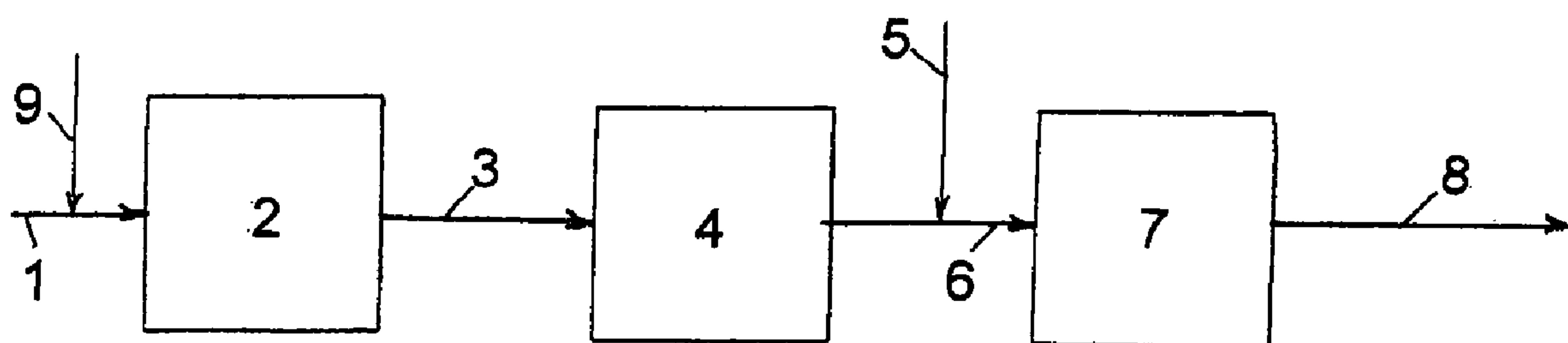
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(54) Titre : PROCEDE DE PURIFICATION DE GAZ D'ECHAPPEMENT D'UN MOTEUR DIESEL  
(54) Title: METHOD FOR PURIFICATION OF EXHAUST GAS FROM A DIESEL ENGINE



(57) Abrégé/Abstract:

The invention provides a method for purification of exhaust gas from a diesel engine in a system, which comprises a device for selective catalytic reduction and a diesel particulate filter preferably at least partially covered by a catalytic layer installed downstream of the device for selective catalytic reduction. A device for catalytic oxidation is installed upstream of the device for selective catalytic reduction and/or between the device for selective catalytic reduction and the diesel particulate filter. A device for injection of a controlled amount of reductant is installed inlet of the device for selective catalytic reduction, and a device for injection of a controlled amount of hydrocarbon is installed inlet of the catalytic oxidation.

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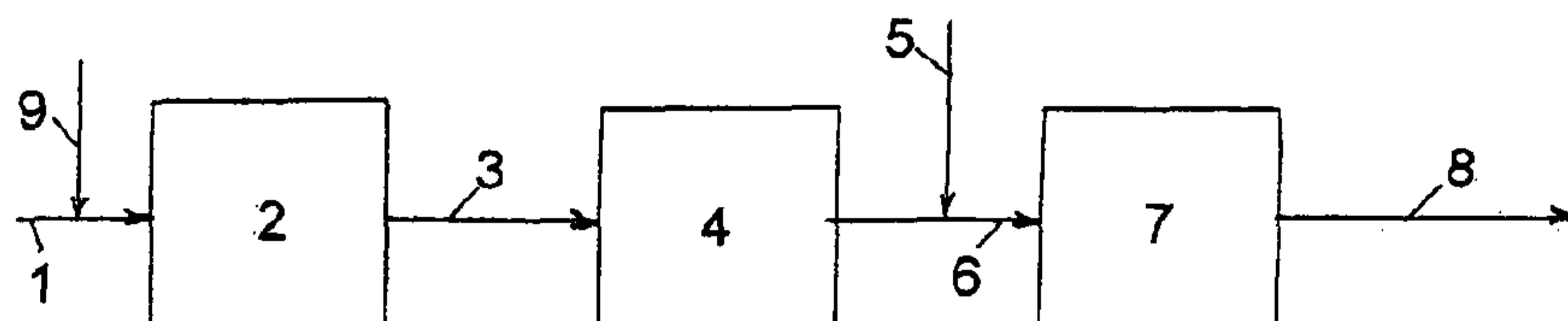


Fig 1

(57) **Abstract:** The invention provides a method for purification of exhaust gas from a diesel engine in a system, which comprises a device for selective catalytic reduction and a diesel particulate filter preferably at least partially covered by a catalytic layer installed downstream of the device for selective catalytic reduction. A device for catalytic oxidation is installed upstream of the device for selective catalytic reduction and/or between the device for selective catalytic reduction and the diesel particulate filter. A device for injection of a controlled amount of reductant is installed inlet of the device for selective catalytic reduction, and a device for injection of a controlled amount of hydrocarbon is installed inlet of the catalytic oxidation.

WO 2010/094313 A1

**Method for purification of exhaust gas from a diesel engine**

The invention relates to purification of exhaust gas from a diesel engine. Particles, incompletely combusted hydrocarbons, carbon oxide, CO, and nitrogen oxides, NO<sub>x</sub> are removed from the exhaust gas.

The invention is specifically directed to a purification method including an effective, but simple method for filter 10 regeneration.

Methods for exhaust gas purification are already known. In US 6,863,874 a method is described, where impurities in an exhaust gas are removed by oxidation followed by a filter, 15 where soot is oxidised by nitrogen dioxide and oxygen. Further downstream, reductant is injected in a NO<sub>x</sub> absorber and subsequently a three-way catalyst or a catalyst for selective reduction is installed.

20 Another process is disclosed in US 6,696,031, where impurities are removed by oxidation, filtering, and selective catalytic reduction (SCR). Ammonia is injected upstream of the oxidation or SCR, and further upstream of this, a pre-oxidation catalyst is installed, to which hydrocarbon may 25 be introduced. A computer must control the two ammonia streams.

30 Impurities from an exhaust gas are by the method of US 6,871,489 removed by passing the gas through an oxidation catalyst, a cooler, equipped with a by-pass, a SCR section including urea injection, through a heater and finally a diesel particulate filter. In this way, the filter must be

regenerated by elevating the temperature of the filter by increasing the heat output from the heater.

These processes are either complicated, energy demanding or 5 having slow urea decomposition simultaneously with slow catalytic reduction of NO<sub>x</sub> at start of cold engine.

It is the general object of this invention to provide a purification process, which effectively removes particles, 10 incompletely combusted hydrocarbons, carbon oxide, CO, and nitrogen oxides, NO<sub>x</sub>, from an exhaust gas and regenerates the filter and at the same time is very simple.

The invention provides a method for purification of exhaust 15 gas from a diesel engine in a system, which comprises a device for selective catalytic reduction and a diesel particulate filter preferably at least partially covered by a catalytic layer and installed downstream of the device for selective catalytic reduction. A device for catalytic oxidation is installed upstream of the device for selective catalytic reduction and/or between the device for selective catalytic reduction and the catalysed diesel particulate filter. A device for injection of a controlled amount of reductant is installed inlet of the device for selective 20 catalytic reduction, and a device for injection of a controlled amount of hydrocarbon is installed inlet of the catalytic oxidation.

The invention provides that the catalysed diesel particulate filter is passively regenerated by closing the injection of reductant and the catalysed diesel particulate filter is actively regenerated by opening for the injection of 25

hydrocarbon inlet of at least one device for catalytic oxidation and optionally closing for the injection of reductant. The filter is passively regenerated by NO<sub>2</sub> at a temperature up to 500°C and actively regenerated at a temperature from 500°C to 700°C.

The reductant is ammonia, aqueous solution of ammonia, urea, an aqueous solution of urea, cyanuric acid. Other potential nitrogen based reagents include ammelide, ammelinge, ammonium cyanate, biuret, ammonium carbamate, ammonium carbonate, ammonium formate, melamine, and tricyanourea. Ammonia, aqueous solution of ammonia, urea, an aqueous solution of urea, cyanuric acid are preferred.

The hydrocarbon is fuel, preferably fuel to the diesel engine. The selective catalytic reduction takes place in presence of a vanadium based catalyst, or a zeolite based catalyst, or a functionalized acidic base metal oxide mixture. The vanadium based catalyst is vanadium oxide on titantium oxide with possible addition of tungsten- or molybdenum oxides. The zeolite based catalyst comprises copper and/or iron modified beta zeolite, ZSM-5 or chabasite and the functionalized acidic base metal oxide mixture comprises acidic cerium-zirconium oxide mixtures and zirconium-titanium oxide mixtures. The vanadium based catalyst is used from 150 °C to 550°C and the zeolite based catalyst or the acidic base metal mixture catalyst is used from 150°C to 800°C.

When a first oxidation catalyst is installed upstream of the selective catalytic reduction device, and a second oxidation catalyst is installed between the selective cata-

lytic reduction device and the diesel particulate filter, then ammonia can be injected inlet the first oxidation catalyst. The first oxidation catalyst comprises platinum and palladium on lanthanum oxide promoted aluminium oxide, 5 or platinum and palladium on silicon oxide promoted titanium oxide, or platinum and palladium on zirconium oxide promoted cerium oxide. The second oxidation catalyst comprises palladium on lanthanum oxide promoted aluminium oxide, or palladium oxide on silicon promoted titanium oxide, 10 or palladium on zirconium oxide promoted cerium oxide, or mixture of copper and manganese oxides or palladium on a mixture of copper and manganese oxides.

The catalytic layer on the diesel particulate filter comprises a mixture of base metal oxides in possible combination with precious metals as palladium and platinum. A special advantageous example with palladium on cerium oxide zirconium oxide mixture is described in EP 1 916 029.

20 The pressure drop across the filter is measured and a created signal is used for controlling addition of reducing agent and addition fuel.

25 The invention further comprises a system for performing the above mentioned method.

This system secures fast cold start of the selective reduction catalyst and very high NO<sub>x</sub> conversions are obtained. By active on/off management of the reducing agent and hydrocarbon injection both passive and active regeneration of 30 the catalyzed filter is secured.

This invention is very useful in cleaning an exhaust gas from a diesel engine, which is installed in motor vehicles, typically cars, vans, trucks, and also vessels. Also in power plants driven by diesel engines the exhaust gas can 5 advantageously be cleaned by the process of the invention.

FIG 1 is a schematic drawing of a process for purification of exhaust gas according to known technology.

10 FIG 2 is a schematic drawing of a process for purification of exhaust gas according to one preferred embodiment of the process of the invention.

15 FIG 3 is a schematic drawing of a process for purification of exhaust gas according to another preferred embodiment of the process of the invention.

20 FIG 4 is a schematic drawing of a process for purification of exhaust gas according to yet another preferred embodiment of the process of the invention.

25 Diesel engines operate with excess air and their exhaust gasses comprise nitrogen oxides,  $\text{NO}_x$ , carbon monoxide,  $\text{CO}$ , particulate matter and incompletely combusted hydrocarbons, which all implement health risk.

The invention basically comprises three processes during normal purification of exhaust gas:

30 Catalytic oxidation, where

CO, NO and HC react with O<sub>2</sub> to form CO<sub>2</sub>, NO<sub>2</sub> and H<sub>2</sub>O and heat

Selective catalytic reduction, where

5

NO and NO<sub>2</sub> are typically reduced by NH<sub>3</sub> to form N<sub>2</sub> and H<sub>2</sub>O

And catching particles in a catalytically coated filter,

10 where

CO, C, HC, and possible slip of NH<sub>3</sub> are oxidised to N<sub>2</sub>, CO<sub>2</sub> and H<sub>2</sub>O

15 C refers here to particulate matter or soot deposited on the diesel particulate filter

When the particles however are accumulated on the filter, the filter can be regenerated by oxidising with nitrogen dioxide, base metal oxide of the filter coating or at 20 higher temperatures with oxygen in the exhaust gas:

C and HC are oxidised by NO, NO<sub>2</sub> and/or O<sub>2</sub> to form N<sub>2</sub>, CO<sub>2</sub> and H<sub>2</sub>O

25

The presently used diesel exhaust system technology (known technology) is characterized in having a SCR catalyst downstream catalyzed filter as shown in Fig. 1.

30 Exhaust gas 1 flows from an engine to an oxidation catalyst 2, the oxidized exhaust gas 3 is then passed to diesel particulate filter 4. After this reductant 5 is injected into

the exhaust gas, and the mixed exhaust gas 6 is then introduced into a catalyst 7 for selective catalytic reduction, SCR, from where the exhaust gas 8 leaves the catalyst 7 cleaned. When increased temperatures are needed, hydrocarbon 9 is post injected in the engine or injected into exhaust gas 1 between the engine and oxidation catalyst 2. This is further described in below process (1).

10 (1) Engine → DOC → cDPF → Urea<sub>inj</sub> → zSCR

where DOC is a diesel oxidation catalyst containing Pt for NO<sub>2</sub> formation, and cDPF is a catalyzed soot filter that also can contain Pt for NO<sub>2</sub> formation .

15 Urea<sub>inj</sub> is injected reductant, typically an aqueous solution of urea, zSCR is a zeolithic based SCR catalyst or a functionalized acidic base metal oxide mixture

20 During forced regeneration of the filter with filter inlet temperatures around 600°C the system will be as

(2) Engine → HC<sub>inj</sub> → DOC → cDPF → zSCR

25 where HC<sub>inj</sub> is hydrocarbon, which is injected into exhaust gas from the engine and upstream of DOC.

Passive regeneration of soot with NO<sub>2</sub> takes place in the configuration as shown in process (1).

30 This system has several drawbacks. The heat up and the start of the SCR reaction from cold start are slow, and the total NO<sub>x</sub> emission, also measured in a test cycle, will be high. The start is also slow, as injection of urea requires

minimum 200°C. Further, the zSCR catalyst shall be able to withstand filter regeneration temperatures of at least 750 °C, and also operation with zSCR normally requires managed NO<sub>2</sub>/NO ratio, which is disturbed by the cDPF as particulate matter also reacts with NO<sub>2</sub> in filter. Finally, the system 5 is very expensive.

A preferred embodiment of present invention is shown in Fig. 2. Exhaust gas 1 from an engine is passed through an 10 oxidation catalyst 2, a catalyst 7 for selective catalytic reduction and as last step through a diesel particulate filter 4. Reductant 5 is injected between oxidation catalyst 2 and the SCR catalyst 7. Hydrocarbon 9 can be injected upstream of oxidation catalyst 2.

15

The new diesel exhaust system with the reverse configuration, SCR upfront cDPF, is further described in below process (3). It further comprises a new, special management.

20 (3) Engine → DOC→Urea<sub>inj</sub> →zSCR→ cDPF

where zSCR is a zeolith based SCR catalyst or a functionalized acidic base metal oxide mixture catalyst, which tolerates minimum 750 °C.

25

cDPF is a diesel particulate filter preferably catalytically coated. In this embodiment it is especially advantageous to use a palladium base metal oxide coating as the commercially available BMC-211 catalyst from Haldor Topsøe 30 A/S and described in patent application EP 1 916 029 . This catalyst limits possible NO<sub>2</sub> emission and facilitates soot combustion in the temperature range 300 - 600 °C.

DOC is a diesel oxidation catalyst, which can have different compositions. This catalyst is precious metal(s) on promoted metal oxide carriers that typically is applied on monolithic skeleton substrates as cordierite, silicon carbide mullite or Fecralloy. It contains typically platinum. It comprises platinum and palladium on lanthanum oxide promoted aluminium oxide, or platinum and palladium on silicon oxide promoted titanium oxide, or platinum and palladium on zirconium oxide promoted cerium oxide.

10

This invention will secure filter soot regeneration effectively both for passive and active conditions in diesel exhaust systems, where the SCR catalyst is placed upstream of cDPF. It further secures high NO<sub>x</sub> conversion at cold start. 15 Soot combustion with O<sub>2</sub> in the exhaust gas takes place in the temperature range above 575°C during active regeneration. Passive NO<sub>2</sub> regeneration, which is soot combustion using NO<sub>2</sub>, can take place in the temperature range of 250 - ca 450 °C. Finally, passive catalyst soot combustion can 20 also take place in a catalysed filter. In the temperature interval of 300 - 600 °C a base metal catalyst BMC-211 can facilitate soot combustion.

An active regeneration in system (3) takes place at ca 620 25 °C filter inlet temperature with diesel post injection in the engine by simultaneously stopping the urea injection as shown in below process (4). In this system it is required that the SCR catalyst for instance zeolithic SCR catalyst can withstand the temperature

30

(4) Engine → HC<sub>inj</sub> → DOC → zSCR → cDPF T<sub>filter in</sub> = 620°C for 10 minutes

Process (3) has many advantages compared to present technology, but seems to have one drawback during passive filter regeneration by nitrogen dioxide. Present invention solves this seeming drawback of system (3) of very low NO<sub>2</sub> concentration downstream the SCR compared to system with a SCR catalyst downstream catalyzed filter as shown in known technology process (1). The present invention secures passive regeneration of the soot filter with NO<sub>2</sub>.

10 The present invention also involves active management of system (3) or (4). The invention comprises that the NO<sub>2</sub> concentration is temporary increased in short time intervals by stopping the urea injection, so NO<sub>2</sub> is formed for the passive regeneration and diesel fuel can be saved. This  
15 temporary NO<sub>2</sub> increase is started, when the pressure drop across the catalyzed filter has increased to a preset value, and the filter inlet temperature has reached a preset value, which should be at least 250 °C. Then system (3) will function like shown in system process (5)

20 (5) Engine → DOC→zSCR→cDPF

In the temperature range ca 300 – 400 °C, the NO<sub>2</sub>/NO will be optimal for a high NO<sub>2</sub> content. The NO<sub>2</sub> concentration and the effect of passive soot combustion can be further  
25 increased by temporary closing for the engine Exhaust Gas Recirculation (EGR) as shown below by (6) concurrently with urea injection is stopped as shown in (5).

(6) Engine (noEGR) → DOC→zSCR→cDPF

30 A further improvement of system (3) can be achieved by injection of ammonia (NH<sub>3</sub>) in stead of urea, which requires

heat both for water evaporation and for urea decomposition. Then the SCR reaction can start from about 150 °C and the SCR catalyst can be placed much closer to the diesel oxidation catalyst DOC as NH<sub>3</sub> mixing can take place within a few 5 centimetre exhaust pipe length f. ex 5 cm.

Lower NO<sub>x</sub> out of the exhaust system is then achieved on the road and when tested by any governmental standard test cycle.

10 The system with enhanced passive regeneration by stopped NH<sub>3</sub> injection is shown by processes (5) and (6). A further possible improvement of system (5) is to inject ammonia (NH<sub>3</sub>) upstream the DOC. By doing this extra NO<sub>2</sub> is formed in the DOC and is used for passive regeneration. At 15 the same time, the temperature in the system is increased. This is shown below for with and without exhaust gas recycle.

(7) Engine → NH<sub>3</sub><sub>inj</sub> → DOC → SCR → cDPF

20 (8) Engine (noEGR) → NH<sub>3</sub><sub>inj</sub> → DOC → SCR → cDPF

System (3) has special interest for passenger cars with diesel post injection options.

25 Another preferred embodiment of the invention is shown in Fig. 3, where it is shown that exhaust gas 1 from the engine passes successively a first oxidation catalyst 2, selective reduction catalyst 7, a second oxidation catalyst 10 and finally the diesel particulate filter 4. Hydrocarbon 30 9 and 11 can be added to the exhaust gas stream upstream of the first 2 and the second 10 oxidation catalyst, while reductant 5 is injected into the selective reduction cata-

lyst 7. Out of the filter 4 flows the cleaned exhaust gas stream 8.

5 The amount of reductant 5 is controlled by valve 12. This will receive a signal from f. inst. a pressure difference measurement across the filter 4, and the valve 12 will close in order to start the passive regeneration. Similarly, when a need to increase the temperature in or outlet one of the oxidation catalysts 2, 10, valve 16 and/or 14 10 will open. Optionally ammonia might be added here. This embodiment is further described below. Normal operation is described by

(9) Engine → DOC(1) → Urea<sub>inj</sub> → SCR → HC<sub>inj</sub> → DOC(2) → cDPF.

15

This is an important example and called Haldor Topsøe reverse standard system Euro VI system. SCR can either be vanadium based SCR or zeolithic SCR or a functionalized acidic base metal oxide mixture. During active regeneration 20 the process is

(10) Engine → DOC (1) → NH<sub>3inj</sub> → zSCR → HC<sub>inj</sub> → DOC(2) → cDPF

And at cold start and with simultaneous active regeneration

25

(11) Engine → NH<sub>3inj</sub> → DOC (1) → NH<sub>3inj</sub> → zSCR → HC<sub>inj</sub> → DOC(2) → cDPF.

30 By NH<sub>3</sub> injection to the first upstream DOC (1) cold start performance can be improved by the generated heat, and the NO<sub>2</sub>/NO ratio will be more optimal for the SCR reaction on zSCR as well.

The DOC (1) is the DOC described for system (3).

The DOC (2) catalyst is precious metals on promoted metal oxide carriers that typically is applied on monolithic skeleton substrates as cordierite, silicon carbide mullite or Fecralloy. This catalyst can also be a mixture of base metal oxides with or without precious metals that typically is applied on monolithic skeleton substrates as cordierite, silicon carbide mullite or Fecralloy. It contains typically not platinum. It comprises palladium on lanthanum oxide promoted aluminium oxide, or palladium oxide on silicon promoted titanium oxide, or palladium on zirconium oxide promoted cerium oxide, or mixture of copper and manganese oxides or palladium on a mixture of copper and manganese oxides.

System (9) is advantageous for both trucks and passenger car applications. A third preferred embodiment is shown in Fig. 4. Here the exhaust gas 1 from the engine is passed through selective catalytic reduction 7, catalytic oxidation 10 and finally through filter 4. Reductant 5 is added to the exhaust gas 1 and hydrocarbon 11 can be added between selective catalytic reduction 7 and catalytic oxidation 10.

The advantage with this exhaust system is that the maximum inlet temperature to the SCR catalyst can be the same as engine outlet temperature, which is approximately 550 °C. This system will facilitate that a standard vanadium based SCR catalyst (V-SCR) can be selected. By use of V-SCR the process will be

(12) Engine → Urea<sub>inj</sub> → V-SCR → HC<sub>inj</sub> → DOC → cDPF

Copper based zeolite SCR catalysts, Cu-zSCR, may be used as the selected SCR catalyst as it is less dependant on the NO<sub>2</sub>/NO ratio, but it requires a Cu-zSCR type, which tolerates HC emission from the engine.

5

The system with NH<sub>3</sub> injection instead of urea for SCR reaction and with simultaneous active regeneration will be as

(13) Engine → NH<sub>3</sub><sub>inj</sub> → V-SCR → HC<sub>inj</sub> → DOC → cDPF

10

In system (13) the distance from engine manifold to the V-SCR catalyst can be decreased by a factor 10, from 50 cm to 5cm. So at cold start engine heat is not used for heating superfluous pipe lengths but for start of the chemical reactions. System (13) will during managed passive regeneration, where NH<sub>3</sub> injection is stopped, temporarily be

15

(14) Engine → V-SCR → DOC → cDPF

20

and equivalent without EGR

(15) Engine (noEGR) → V-SCR → DOC → cDPF

System (12) has special interest for truck applications

25

### **Example 1.**

Engine test bench experiments on a Scania 12 liter engine with a SCR+DOC+cDPF system gives unchanged soot load pressure drop after 7 world harmonized test cycles with intermittent urea dosing and temporary high NO<sub>2</sub> content.

30

Measurement of pressure drop over cDPF for the 1st and 7th repeated world harmonized test cycles (WHTC) measured in one day where the system was managed with both low and high dosing of urea.

5

In Fig. 5 the two curves are identical, consequently they are written in top of each other. This means that no pressure drop increase from additional soot loading is seen. This result shall be compared to the reference example below.

10

In this reference example the urea dosing is continuous and high for high SCR reaction. This gives low NO<sub>2</sub> and NOx (approximately 1-2 g NOx/kWh) exit the SCR catalyst - inlet cDPF). In Fig. 6, we see in contrast to the Fig. 5 above the pressure drop increases from first WHTC to 7<sup>th</sup> cycle. This means that soot is building up on the filter.

**CLAIMS**

1. A method for purification of exhaust gas from a diesel engine in a system comprising:
  - a device for selective catalytic reduction;
  - a diesel particulate filter preferably at least partially covered by a catalytic layer installed downstream of the device for selective catalytic reduction;
  - a device for catalytic oxidation installed at one or more of upstream of the device for selective catalytic reduction and between the device for selective catalytic reduction and the diesel particulate filter;
  - a device for injection of a controlled amount of reductant injected at an inlet of the device for selective catalytic reduction; and
  - a device for injection of a controlled amount of hydrocarbon injected at an inlet of the device for catalytic oxidation;

characterised in that the diesel particulate filter is passively regenerated by closing for the injection of reductant; and the diesel particulate filter is actively regenerated by opening for the injection of the hydrocarbon inlet of at least one device for catalytic oxidation, and optionally closing for the injection of the reductant.
2. The method according to claim 1, wherein the filter is passively regenerated by NO<sub>2</sub> at a temperature up to 500°C.
3. The method according to claim 1, wherein the filter is actively regenerated at a temperature from 500°C to 700°C.

4. The method according to claim 1, wherein the reductant is ammonia, aqueous solution of ammonia, urea, an aqueous solution of urea, cyanuric acid, ammelide, ammelinge, ammonium cyanate, biuret, ammonium carbamate, ammonium carbonate, ammonium formate, melamine, or tricyanourea.
5. The method according to claim 4, wherein the reductant is ammonia, aqueous solution of ammonia, urea, an aqueous solution of urea or cyanuric acid.
6. The method according to claim 1, wherein the hydrocarbon is fuel, preferably same fuel as to the diesel engine.
7. The method according to claim 1, wherein the selective catalytic reduction takes place in presence of a vanadium based catalyst or a zeolite based catalyst or a functionalized acidic base metal mixed oxide catalyst.
8. The method according to claim 7, wherein the vanadium based catalyst is vanadium oxide on titanium oxide, the zeolite based catalyst is copper modified beta zeolite, iron modified beta zeolite, copper and iron modified beta zeolite, ZSM-5 or chabasite, and the functionalized acidic base metal mixed oxide catalyst comprises acidic cerium-zirconium oxide mixtures and zirconium-titanium oxide mixtures.
9. The method according to claim 8, wherein the vanadium oxide on titanium oxide is vanadium oxide on titanium oxide with addition of tungsten- or molybdenum oxides.

10. The method according to claim 7, wherein the vanadium based catalyst is used at temperatures from 150°C to 550°C and the zeolite based catalyst is used at temperatures from 150°C to 800°C.

11. The method according to claim 1, wherein the catalytic layer on the diesel particulate filter comprises a mixture of base metal oxides.

12. The method according to claim 11, wherein the base metal oxides are in combination with precious metals.

13. The method according to claim 12, wherein the precious metals are one or more of palladium and platinum.

14. The method according to claim 1, wherein a first oxidation catalyst is installed upstream of the selective catalytic reduction device and a second oxidation catalyst is installed between the selective catalytic reduction device and the diesel particulate filter and ammonia is injected into the first oxidation catalyst.

15. The method according to claim 1, wherein the device for catalytic oxidation installed upstream of the device for selective catalytic reduction comprises a catalyst comprising platinum and palladium on lanthanum oxide promoted aluminium oxide, or platinum and palladium on silicon oxide promoted titanium oxide, or platinum and palladium on zirconium oxide promoted cerium oxide; and the device for catalytic oxidation installed downstream of the device for selective catalytic

reduction comprises a catalyst comprising palladium on lanthanum oxide promoted aluminium oxide, or palladium on silicon oxide promoted titanium oxide, or palladium on zirconium oxide promoted cerium oxide, or mixture of copper and manganese oxides or palladium on a mixture of copper and manganese oxides.

16. The method according to claim 1, wherein pressure drop across the filter is measured and a created signal is used for controlling addition of reducing agent and addition of hydrocarbon.

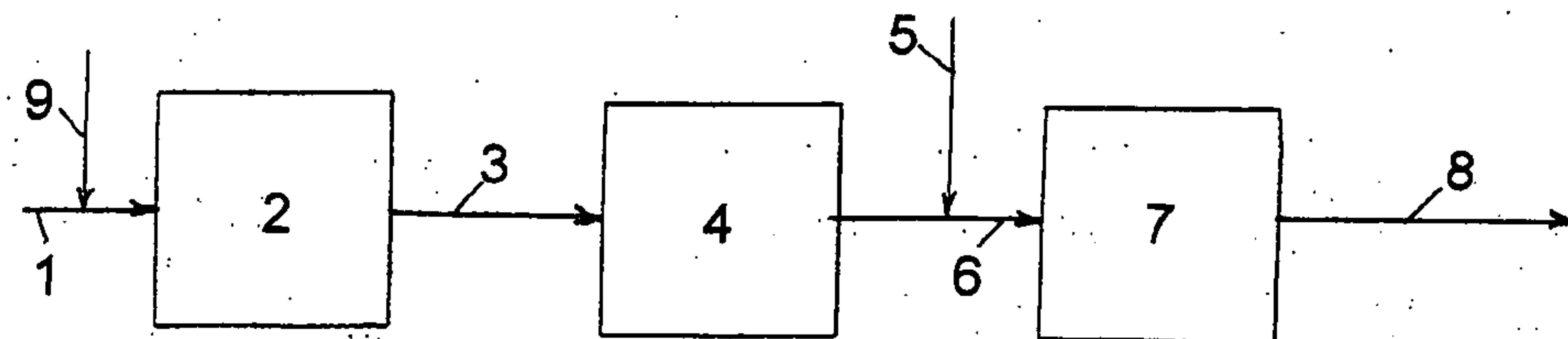


Fig 1

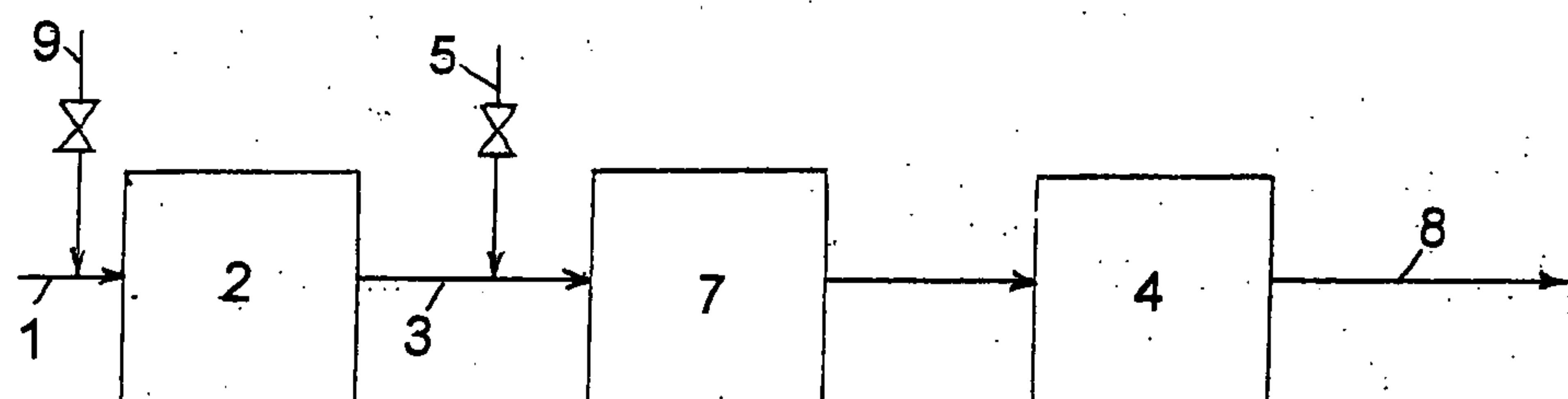


Fig 2

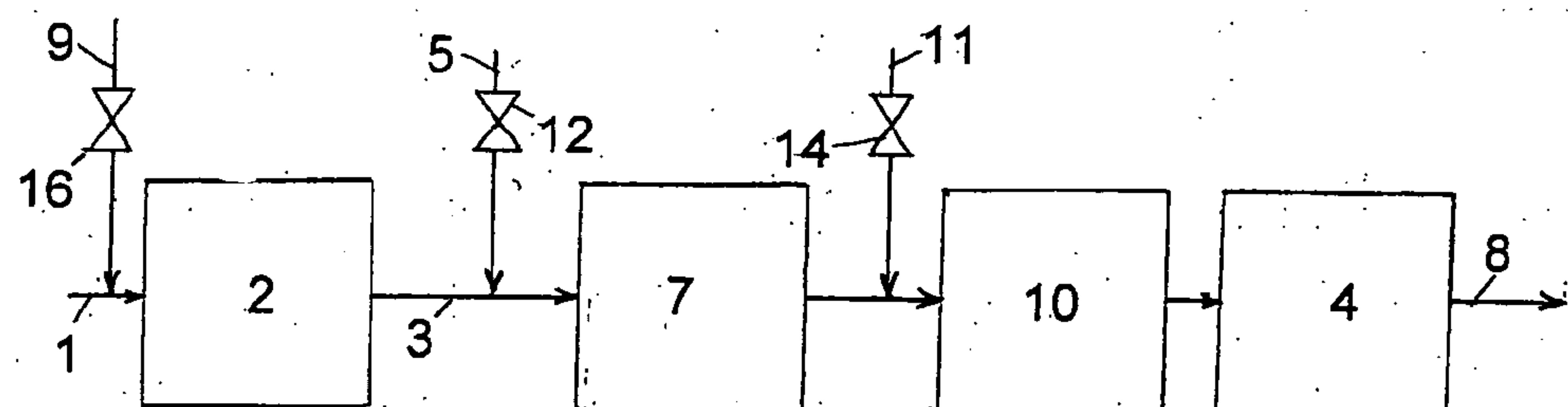


Fig 3

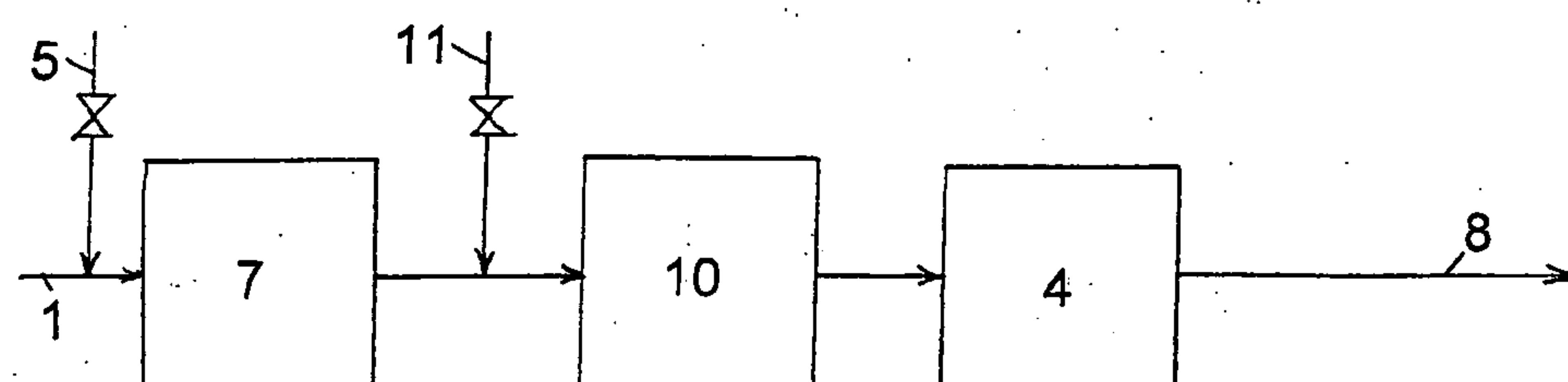
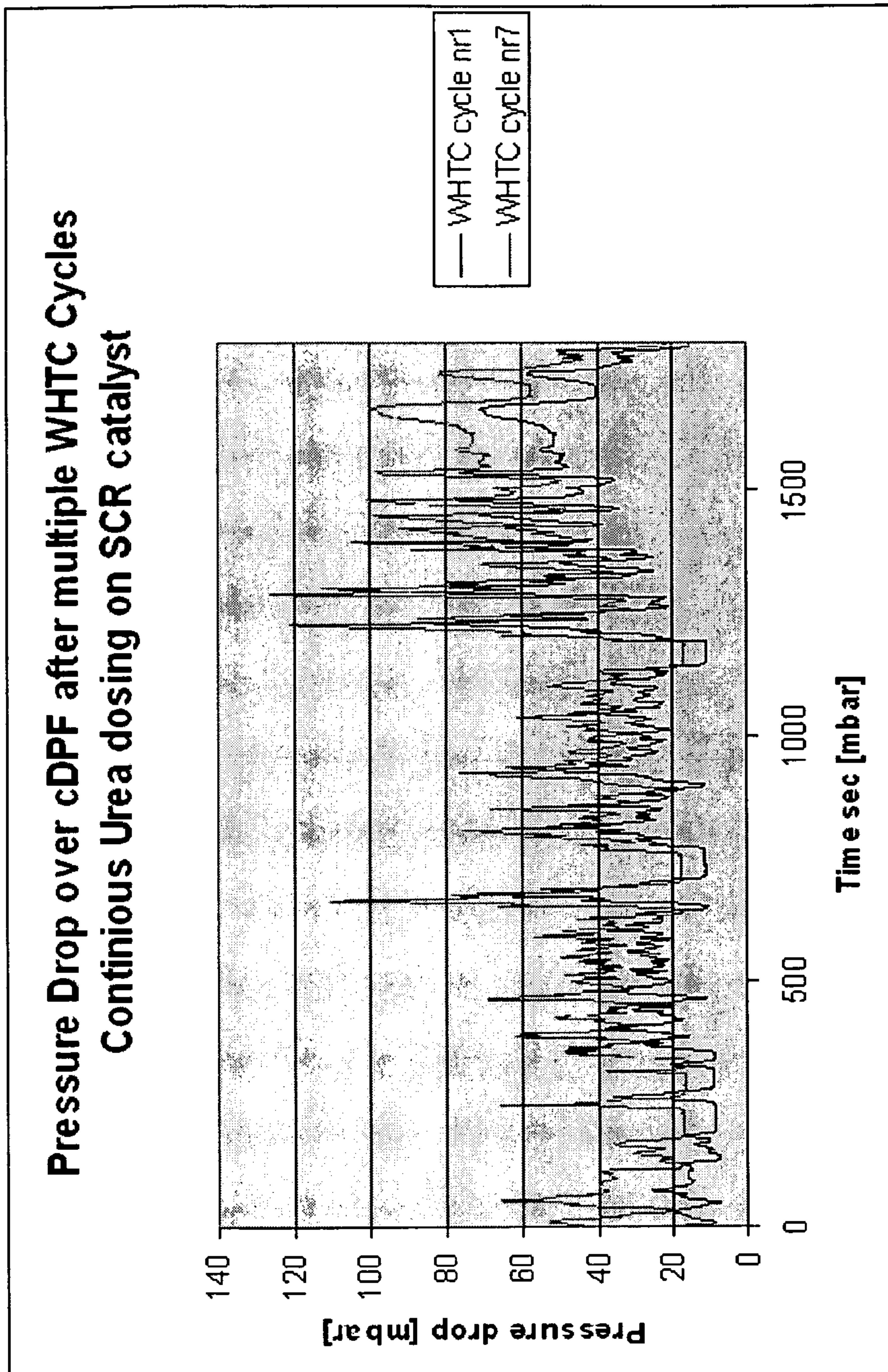


Fig 4

**Fig. 5**

**Fig. 6**