

ENVIRONMENTAL LOAD OF SULPHUR FROM STORAGE CATALYSTS

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Abstract

Under the development of quality standards for fuels was the theoretical basis of storage-reduction catalyse with sulphur also examined. Lowering the NO_x emission of motor vehicles powered by an Otto-motor with direct fuel injection can be done in the most effective way by the use of a NO_x storage catalyst. But sulphur-regeneration shows a strongly negative impact on NO_x conversion. In contrast to SCR catalysts sulphur content in exhaust gas can cause here strong detractions in normal operation of the catalysts.

An on-board analysator has been developed and built to recognize SO_2 , H_2S and CO_2 concentration of the exhaust gas simultaneously both at the test bench and while drive. SO_2 and H_2S are measured by electro-chemical cells while CO_2 concentration is detected optically based on infrared light adsorption. This device gives reliable data about the emission in one second timing during the regeneration phase of the storage catalyst. This system makes NO_x storage more manageable, secure and optimised with regard to the increasing notability of the ‘in-use compliance’.

Keywords: NO_x storage catalyst, sulphur-regeneration, NO_x conversion, on-board gas analysis SO_2 , H_2S and CO_2 concentration measurement.

1. Quality Requirements to Fuels

These days in the European Union approximately 60 million t of petrol and diesel fuel is used up annually in the motorized traffic.

This load caused by the traffic with substances originating from the sulphur containing exhaust gas components must be drastically reduced due to reasons for environment and health protection.

Current computations for sulphur concentration in the fuel are presented below, which have been codified in the guideline 98/70/EG.

The agreed fuel specifications can be found in *Table 1*.

The World-Wide Fuel Charter of the automobile industry calls for further improvement in petrol fuel with a sulphur content of max. 30 ppm for category 3 and 5–10 for category 4. It was primarily justified by the requirements of future technologies for lower consumption (lean-burn engine with NO_x storage catalyst or other system for NO_x reduction) as well as an olefins content of max. 10% v/v. Furthermore, there are additional requirements from a vehicle-technical’s view: the

Table 1. Fuel quality for spark-ignition engines

Parameter	Basis 1995	Comm. June 1996	OK 2000 98/70/EG	OK 2005 98/70/EG	RFG II	World-Wide Fuel Charta
sulphur in petrol engine ppm max.	240	200	150	50	40	30 5–10

Table 2. Fuel quality for compression-ignition engines

Parameter	Basis 1995	Comm. June 1996	OK 2000 98/70/EG	OK 2005 98/70/EG	RFG II	World-Wide Fuel Charta
sulphur in diesel engine ppm max.	450	350	350	50	10	30 5–10

Source: Basic fuel 1995/96 in Germany

Comm. June 1996: Proposal of the EU-Commission from June 1996

OK 2000: specification of the EU-guideline 98/70/EG starting from the year 2000

OK 2005: specification of the EU-guideline 98/70/EG starting from the year 2005

RFG II: petrol according to Californian Reformulated Gasoline II (adjusted steam pressure)

World-Wide Fuel Charta: Category 3 and 4 of the World-Wide Fuel Charta, draft from January 2000

most important measures are for diesel engines the SCR technology, the DeNO_x and NO_x storage catalyst and the CRT filter. To achieve the sulphur concentration needed the following measures for lowering the emission are planned.

The first stage of the guideline 98/70/EG does not contain any improvements in diesel fuel quality but in the second stage there are significant improvements concerning the sulphur content [1].

2. Theoretical Basis of the Storage-Reduction-Catalyst for sulphur

The exhaust gas from the oxygen-rich environment of the combustion engine contains beside nitrogen-oxides also sulphur-oxides, which appear according to the high temperatures existing there as nitrogen oxide (NO) and sulphur dioxide.

The highly soluted NO and SO₂ are sorbed at the (quasi two-dimensional) solid surface of the catalyst system and thus concentrated. The precious metal component (platinum) being in contact O₂ containing exhaust gas is completely covered with oxygen, see Fig. 1.

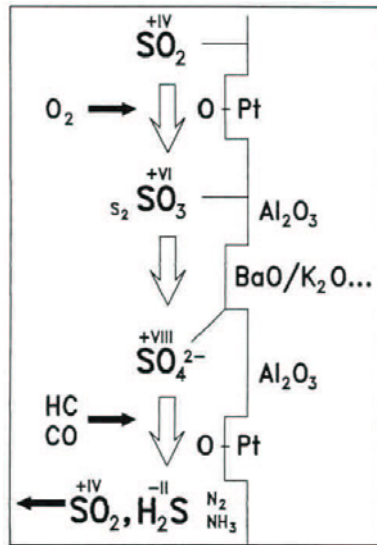


Fig. 1. Function of NO_x -storage-reduction-catalyst for sulphur

Sulphur behaves analogously to nitrogen by the unfortunately not avoidable sulphonisation (so-called sulphurisation) and by the following aimed desulfatisation (desulphurisation, regeneration) of the NO_x -storage-reduction catalysts. Due to fuels containing sulphur the exhaust gas contains sulphur dioxide (SO_2 , S formally +4 loaded), which will be oxidized to the sulphur trioxide (SO_3 , S formally +6 loaded) at the precious metal (Pt) and that reacts with the basic components mentioned stoichiometrically to sulfates. After change-over from the net oxidizing conditions to the net reducing conditions the sulfates should be reduced actually again to the SO_2 or even to the less desired hydrogen sulfide (H_2S , S formally -2 loaded).

3. NO_x Storage in the Motor Vehicle

In the year 1994 after a press release of the company Toyota the patent rights to a new concept to remove NO_x from exhaust gases became conscious to a broader interested party. By the so called NO_x -storage-reduction-principle devices for the preparation of reducing agents (as for instance with the HC-SCR) and also specially the carrying along of additional reducing agent (as with the urea- or carbamate-SCR) are disclaimed.

By the NSR-procedure the necessary reducing agent is made available on demand in active or passive engine-generated way (during short fat operating phases).

The function is as follows: In the lean operating condition (air number of

$\lambda > 1$) the NSR- contact functions as oxidation catalyst is expected. Hereby unburned hydrocarbons and carbon monoxide (CO) are oxidized to CO_2 and H_2O at the precious metal components (Pt).

Sulphur dioxide SO_2 is oxidized at the platinum-contact to SO_3 and it is stored in the form of sulfates in the washcoat ($\gamma\text{-Al}_2\text{O}_3$) and in the available basicity alkaline carbonates/oxides that are stored in a massive way (alkali metals such as potassium, heavy alkaline earth metals such as barium, light seldom earth metals such as lanthanoides or cerium; transition metals), see Fig. 2.

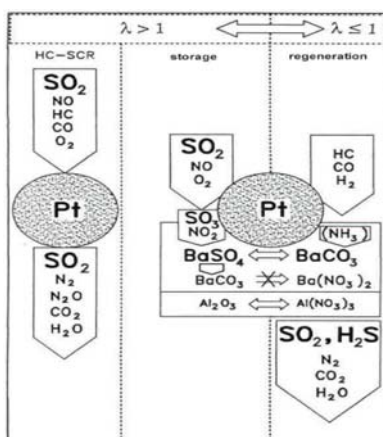


Fig. 2. Function of SO_2 -storage in the NO_x storage-reduction-catalyst

Operation diagramme of a NSR catalyst on the basis of the SO_2 - and NO_x storage and sulfate and nitrate regeneration by the example of platinum as active component, barium oxide/carbonate and aluminium oxide as storage components.

Latest after some minutes the NO_x storage capacity of the catalyst is exhausted by the stoichiometric chemical reaction and a regeneration (nitrate decomposition) must take place, which is introduced by brief grease of the exhaust gas on λ of about 0.98 (sometimes to λ around 0.8). The NO_x setting free in such a way (in the substantial NO_2) is in that way reduced by the other exhaust components HC and CO_2 , which exists in a surplus to the compounds H_2 and N_2 without arising considerable mass of N_2O . The grease of the composite is caused by the motor management system if the current operating does not require it anyway (warming-up phase, acceleration, full load).

Due to the higher thermodynamic stability of sulfates opposite to nitrate larger problems are to be expected with the desulfatization [2].

4. Functional Principle of NO_x-Catalysts in Motor Vehicle

After the test of the New European Driving Cycle (NEDC) with NO_x catalysts conversion-rates can be over 50% by diesel engines and appr. 90% by spark-ignition motors.

The emission reduction of NO_x by a spark-ignition engine with direct injecting though a starting catalyst and a fresh NO_x-storage catalyst in NEDC of over 90% shows *Fig. 3*.

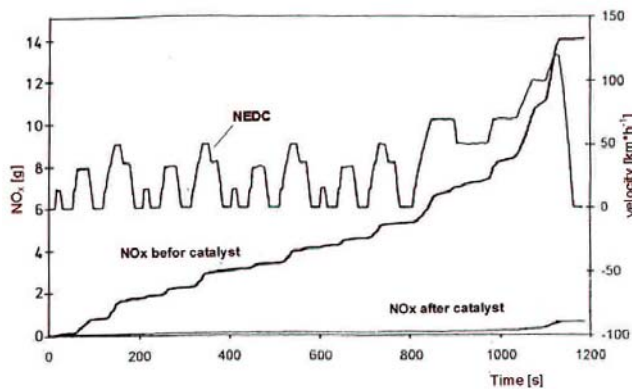


Fig. 3. Cumulated NO_x emission of a motor vehicle with direct injecting petrol engine and NO_x storage catalyst

The principle of the operation of an NO_x-storage catalyst can be seen in *Fig. 4*.

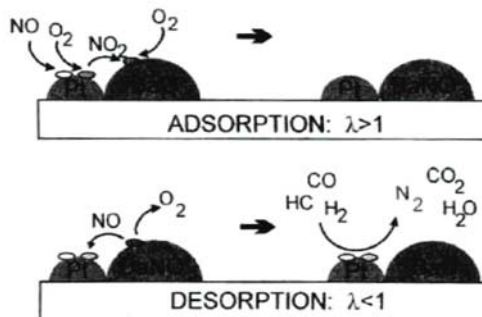


Fig. 4. Principle of operation of an NO_x-storage catalyst

The course of nitrogen oxide concentration by a diesel engine before and after the storage catalyst and the combustion air relationship can be seen in *Fig. 5*. NO_x conversion of 91% has been reached in the represented stationary operating point of the engine with a fresh catalyst and with the use of sulphur-free fuel.

4-cylinder Common-Rail-Diesel-Engine	
$n = 2000 \text{ min}^{-1}$, P_{me}	2 bar
T_{abg}	300 °C
time of adsorption	45 s
time of regeneration	2 s
NO_x -conversion	91%

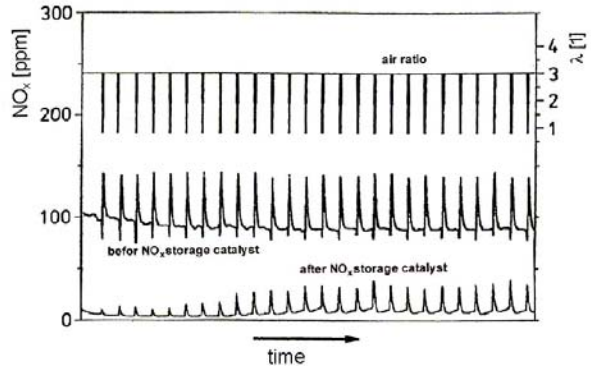


Fig. 5. Course of the NO_x concentration and the combustion air relation at the test motor

According to Chapter 3 the sensitivity of the NO_x adsorbents towards sulphur is principle-conditioned. The sulphur oxides SO_2/SO_3 in the +exhaust gas react with the alkaline storage elements to sulfate, thus leading to a sulphur poisoning. Although the nitrate is desorbed as nitrogen oxide during the regeneration-phase of NO_x but the sulfates remain in the storage, i.e. with the running time the NO_x adsorbent is more and more sulfatisated and can be ever less used for the adsorption of nitrogen oxides, see Fig. 6.

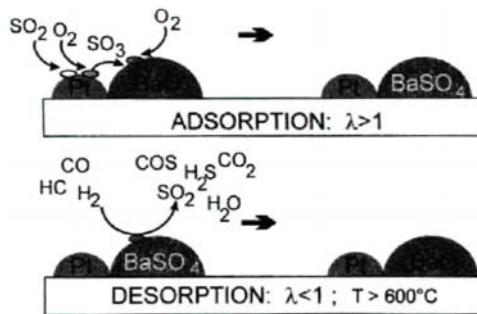


Fig. 6. Scheme for the poisoning of a NO_x storage catalyst caused by the sulphur oxide

4.1. Influence of the Sulphur Regeneration on NO_x -Conversion

Fig. 7 shows us that in order to an effective regeneration of NO_x storage catalysts poisoned with sulphur the exhaust gas temperatur must be above 600°C and understoichiometric combustion air conditions are also necessary.

The influence of the duration of the regeneration on the sulphur regeneration is

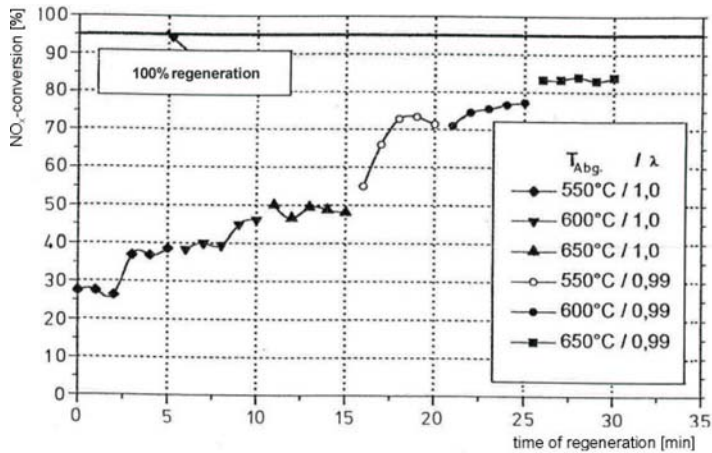


Fig. 7. Influence of the sulphur regeneration strategy on NO_x conversion in Partial-load operation

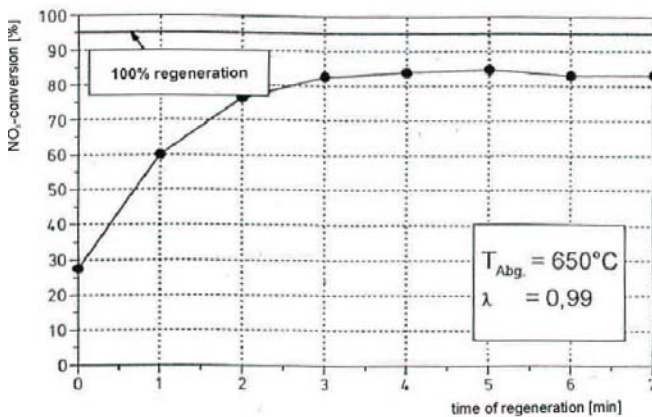


Fig. 8. Regeneration of sulphur in the partial load operation

shown in Fig. 8. The regeneration of the sulphur-poisoned catalyst with a remaining NO_x conversion of approximately 30% takes several minutes. So these operating conditions are not ensured long enough in the pure city or short-term drive.

By sulphur-regeneration the fuel consumption of the engine notably increases. Even a SO_2 -storage ('sulphur-trap'), which becomes upstream to the NO_x storage catalyst does not represent a full protection to the NO_x storage-catalyst against sulphur-poisoning at a lean engine use. Depending on the conditions of regeneration the adsorbed sulphur can be set free as sulphur-hydrogen H_2S , carbonyl-sulfide COS

or sulphur-dioxide SO_2 . Fig. 9 shows that a NO_x storage catalyst despite of sulphur regeneration loses its effectiveness relatively fast.

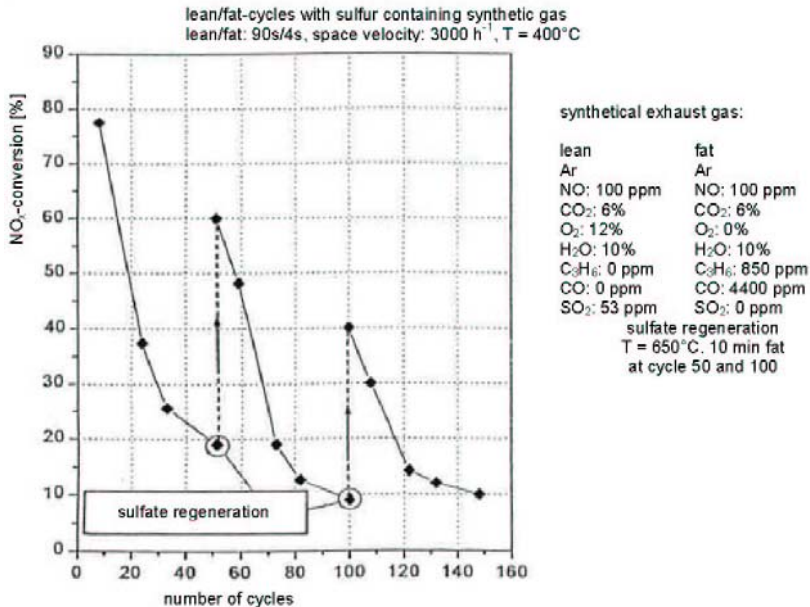


Fig. 9. Decrease of the NO_x conversion despite of sulphur-regeneration

An estimation for the fuel with a sulphur content of 100 ppm resulted in an additional fuel consumption of 1.4% for the sulphur-regeneration of the NO_x -storage catalyst in a spark-ignition motor. In a diesel engine still higher consumption disadvantages have to be taken into account due to smaller exhaust gas temperature [3].

5. Influence of SO_2 on the Activity of the SCR Catalyst

An important issue is the sensitivity on sulphur of the catalyst by the SCR-technique with vanadium-pentoxide.

Several basic experiments were made for answering this question. The concentration of SO_2 in the experiment amounted to 50 ppm. By comparison of the conversion curves from the Fig. 11 it becomes clear that the NO_x conversion in presence of sulphur oxide lowers itself only slightly. After an admission of the catalyst with SO_2 -containing exhaust gas over a time of 20 h no further decrease can be observed in the activity [4].

The attempts in presence of SO_2 have shown that 50 ppm sulphur dioxide hardly affect the activity of the catalyst. Because of using diesel fuel with a sulphur content of 500 ppm the SO_2 -content lies in the exhaust gas only at approximately

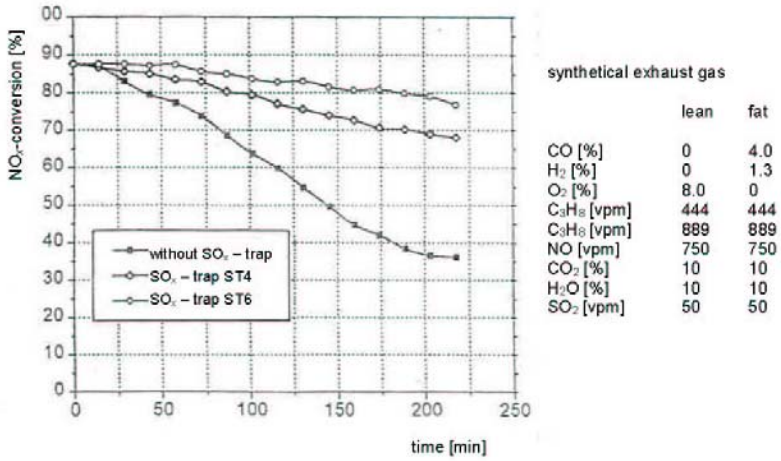


Fig. 10. Decrease of the NO_x conversion with a catalyst combination of SO_x-storage and NO_x storage-catalyst at 450 °C by 120 s/6 s lean/fat cycles

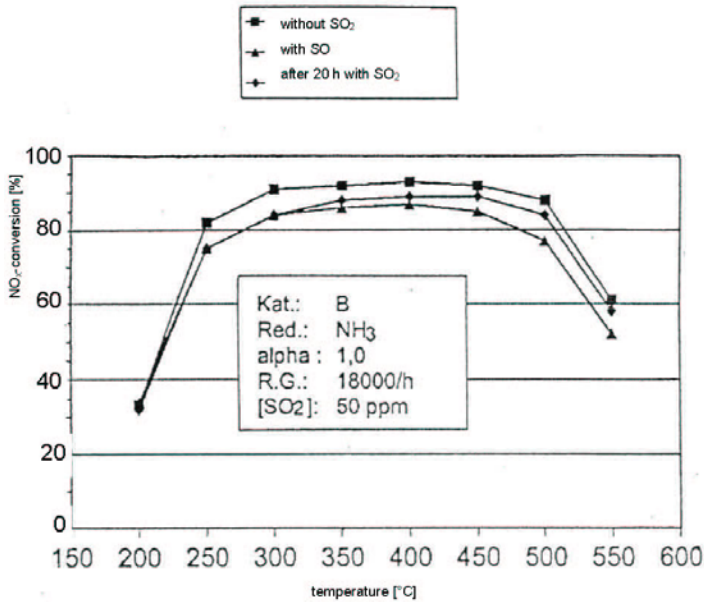


Fig. 11. Influence of SO₂ on the NO_x-conversion at the SCR-catalyst

20 ppm so the influence of SO_2 on the NO_x conversion by the SCR-technique is to be classified under real conditions as negligible.

5.1. Evaluation of the Results

As a summary one can note that with fresh NO_x -storage catalysts high NO_x conversion rates are possible. The storage materials known today require extremely low sulphur contents in the fuel in order to guarantee their effectiveness by a sufficiently long life of use as well. The regeneration of sulphur is already by small sulphur contents necessary but it's only limited feasible and leads to a deterioration of the fuel consumption and to the intensified release of secondary pollutants. For this reason a sulphur-sensitive mobile analyser has been developed for the determination of the SO_2 , H_2S and CO_2 concentration.

6. The OBM-S-System

The OBM-O-system means a multi component gas analysator, which is bound to the motor management system and can record 3 gaseous substances and further physical-chemical characteristics in the exhaust gas stream and in the motor during drive.

6.1. Description of the Mechanics

Fig. 12 shows the gas flow sheet of the measurement device. The elements are integrated in an aluminium case and they can be divided into five building groups:

- gas preparation with filter, valve, cooler and flow rate counter
- optical cell with emitter and detector for CO_2
- the electrochemical cells for SO_2 and H_2S
- the board with a micro controller and
- the internal control and calculation software.

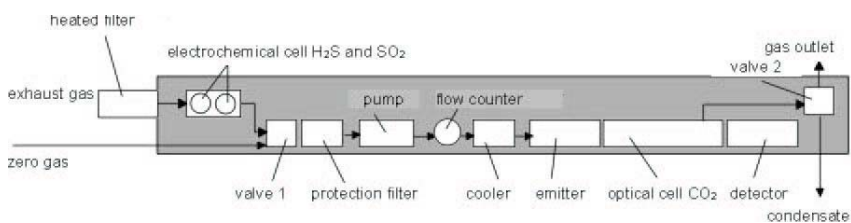


Fig. 12. Schematic gas flow sheet of the OBM-S-System

Due to the 3-way-valve 1 the gas way before the actual absorption cell can be switched into two directions.

- on one way gas comes from the measuring gas. In this case the exhaust gas to be measured flows into the measure unit
- on the other way ambient air (zero-gas) is sucked into the measuring system. In case of possibly dirty ambient air with the use of an adsorption filter with activated charcoal is recommended.

The first phase is the so called measuring phase and the second phase is the zero-phase. In the 'measuring phase' the optical cell is flowed through by measuring gas but in the 'zero-phase' ambient air is led through. The device can do corrections based on 'null gas' particularly to equalize the influences of temperature.

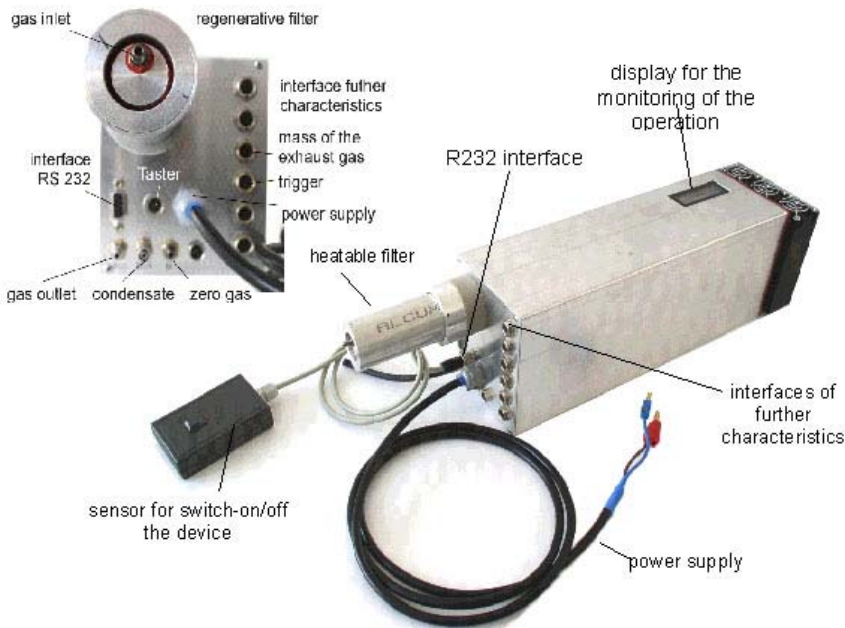


Fig. 13. The outer appearance of the OBM-S-System

In order to avoid the acid dew point the gas to be measured is taken away from the exhaust gas stream by means of a heated line with a temperature of 180°C and it is treated. With a pump a partial volume is continuously sucked off from the exhaust gas stream.

6.2. Regenerative Filter Directly at the Measurement System

For analysing the emission of the pollutants it is unavoidable to prepare the exhaust gas, e.g. it has to be cleaned, dried and perhaps tempered. For this it has been developed a heatable filter that can be installed directly in the front of the OBM-S-System. In such a way contamination and as a result possible damages within the optics are prevented.

The system consist of three main modules:

- the regenerative filter candle
- the pneumatic – consisting of a pump for backwash and valves and
- the flow rate counter as a sensor for blockage.

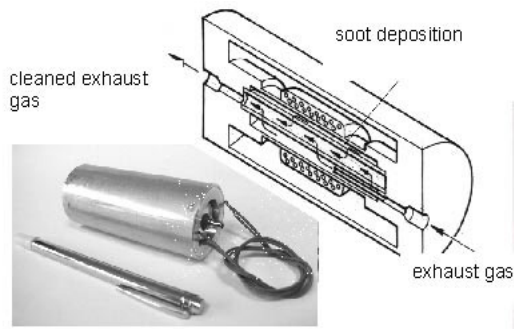


Fig. 14. Filter tube



Fig. 15. Filter at the front panel of the OBM-S-System

The regenerative filter will be installed in front of the actual measurement system. The measurement gas will be directed through the filter. If the downstream flow sensor indicates that the amount of gas is no longer sufficient a regeneration

must be accomplished. The stuffy filter will be heated up to 800 °C electrically and regenerated in about 10 minutes. During the regeneration phase the residues will be blown out by means of an external pump for backwash on a reversed air stream. The need for regeneration can be indicated by a flow sensor integrated into the device or it can also be performed periodically in a predefined time (e.g. every hour). The adjustment of the regeneration phase and the measurement phase can be set by the customer with the delivered software.

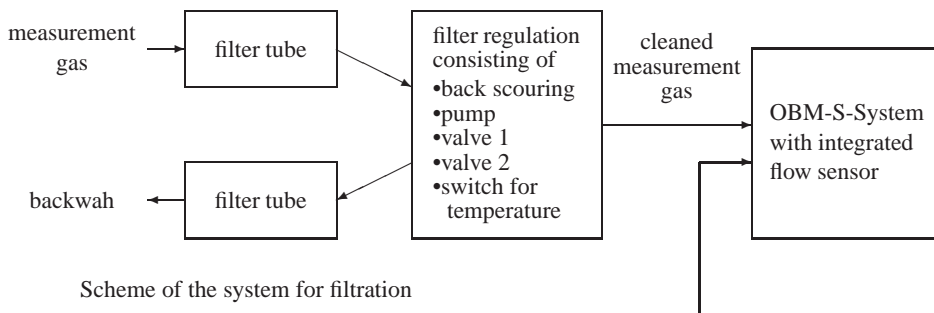


Fig. 16. Function of regenerativ filters

6.3. Long-Term-Stability

Little electromagnetic switches are used as valves to switch the way of the gas in certain little time units from the exhaust gas stream to ambient air. By appropriate stacking of these so called zero phases the signal shift (that is caused by changes of the temperature in the neighbourhood) is reduced to a minimum. Thereof the system is extensive independently of the influence of temperature. The zero calibration is made automatically for the IR optical cell (CO₂) and it is made manual for the electrochemical sensors (for H₂S and SO₂).

In substantially larger time intervals in relation to the zero point adjustment the sensitivity of the system is adjusted manually towards CO₂, H₂S and SO₂ with a test gas. Without these measures the sensitivity of the measuring system could decrease with time.

6.4. Cooling of the Gas for Measurement of CO₂

A micro cooler was developed, see Fig. 17, in order to be able to temper and thus dry the water vapour containing measurement gas for determine the CO₂ content, which

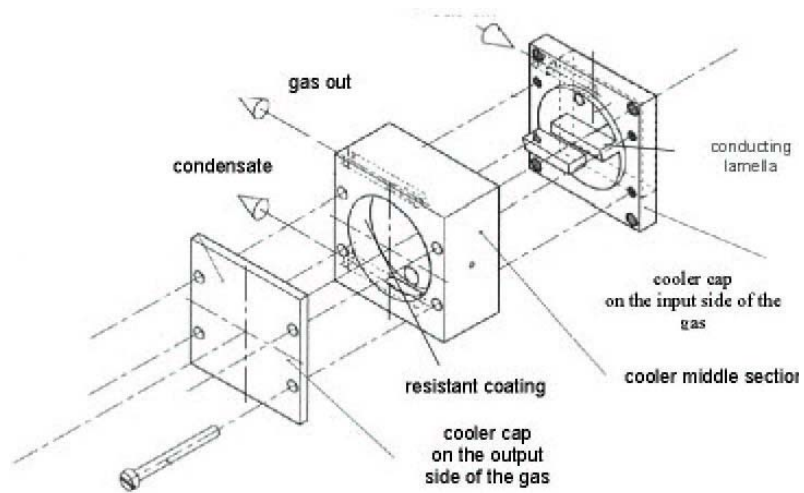


Fig. 17. Construction of the micro cooler

shall be recorded unheated. This guarantees independence for the measurement signal from the water vapour content of the measuring gas.

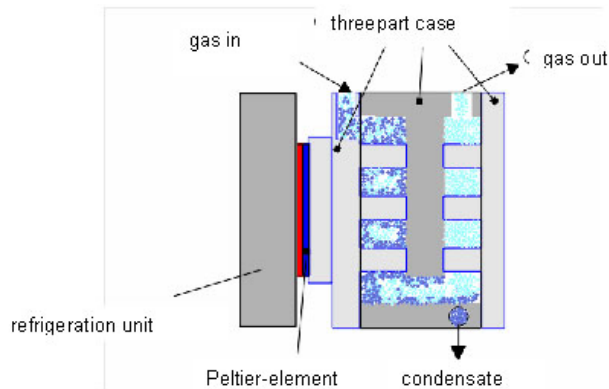


Fig. 18. Function of the micro cooler

6.5. CO_2 Measurement with Optics

The gas to be measured is led through an optical cell. There it is beamed by infrared light and on the opposite side a detector measures the intensity of this light. The respective molecules absorb the light at definite wave lengths. Measuring the light

intensity definitely at this wave lengths the extent of absorption gives a measure for the CO₂ concentration of the gas.

The infrared system consists a module of up to four optical cells with high optical reflection grade. At the entry of the optical cell is an optical emitter and at the end is the pyro-electrical detector with two active surfaces in order to make the compensation of the two signals for the optimal correction, see *Fig. 19*.

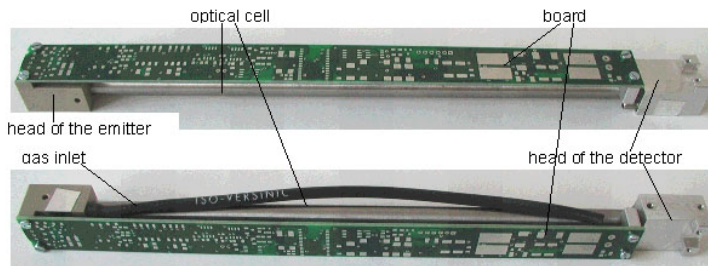


Fig. 19. Optical cell

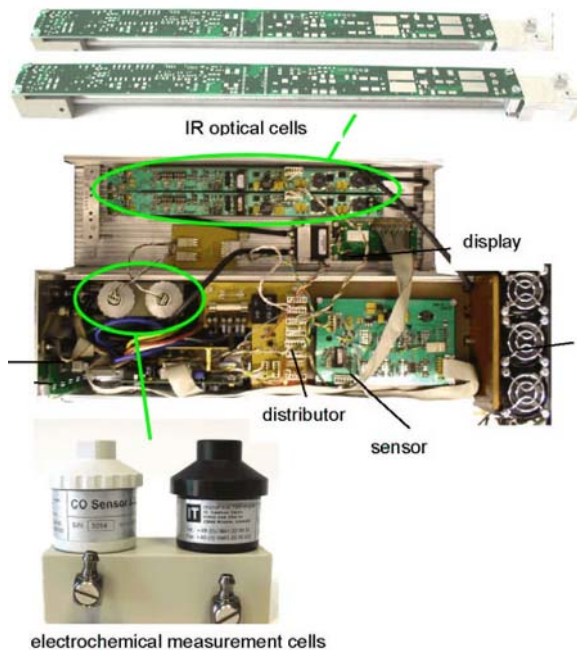


Fig. 20. Measurement system with infrared and electrochemical technique

Base technique for the measurement of H₂S and SO₂.

Electrochemical cells are measuring transformers based on the measurement

of potential difference caused by an electro chemical reaction of the corresponding gas under atmospheric conditions, see *Fig. 21*.

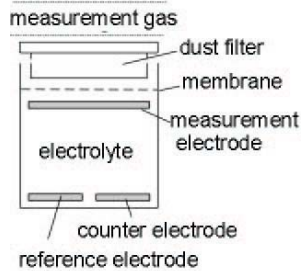


Fig. 21. Functional principle of the electrochemical cell

Advantages of the Electrochemical Cells are:

- the small dimensional expansion
- the small current consumption
- the high resolution by small concentrations from 1–10 ppm.

Disadvantages of the Electrochemical Cells are:

- limited operation runtime
- strong drift behaviour in a function of temperature.

6.6. Description of the Electronics

Fig. 22 shows the schematic representation of the system. The section, which is marked grey shows the measurement device. It divides into the following hardware-components:

- gas preparation
- optical cell module 1-4
- master-module
- slave module (up to 16 parts)
- data record.

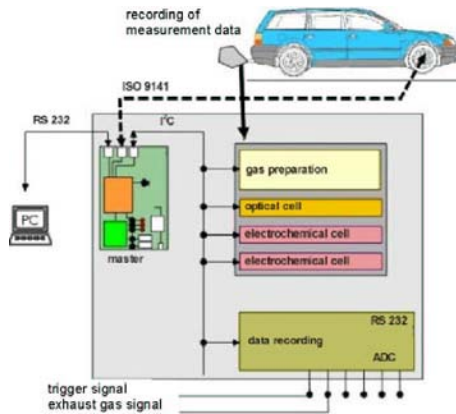


Fig. 22. Schematic representation of the OBM-S-System

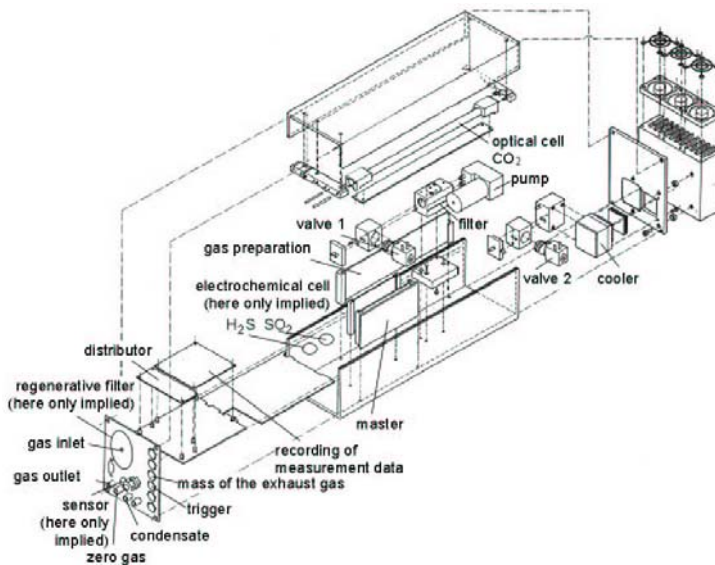


Fig. 23. The OBM-S-System from the inside

The master with I²C-bus enables a flexible and expandable configuration, i.e. the number of the optical cell modules is variable. The compilation of H₂S, SO₂ and CO₂ can be realised in one device and at the same time. The data record can be expanded to 16 measurement receipts and GPS-data may be captured as well. In certain motor vehicles data request is possible from the motor management via the K-line interface. All data is sent to the PC by a serial interface where it gets visualized and evaluated.

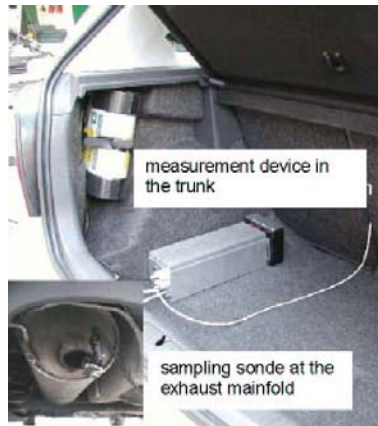


Fig. 24. The OBM-S-System in the trunk

The OBM-System from the inside can be seen in the Fig. 23.

6.7. The Use of OBM-S-System in the Motor Vehicle

If the use of the motor vehicle's own generator and battery is not possible (because they are inefficient) an external accumulator must be carried along without a connection to the generator in the vehicle.

The OBM-S-System can be installed

- in the motor room near to the motor block
- below the chassis near to the catalyst
- in the luggage trunk of the motor vehicle, see Fig. 24.

The OBM-S-System is designed so that the user does not need to make any handles while driving because each distraction is meant an enhanced accident hazard, which should be avoided. The system consists of the actual measurement system and of a regenerative filter unit (REFi). A laptop computer controls all functions and stores all data during the drive. Before starting the drive the system is started via a main switch. An LED (blinking) indicates whether the system is operational and the measurement drive can start. The main switch can also serve as a 'breaker'. Via an LED (blinking) it will be signalled to the user that the switch was pressed. Now the measurement is initiated with the zero-gas phase and afterwards the device is powered off.

The measurement system is controlled by a laptop or a PC. The measurement can be monitored directly in the laptop. The power supply is an external power unit. The connection of a compatible industrial PC-module of the type iPC is also possible.

The determination of the mass flow of H_2S , SO_2 and CO_2 in the unit of $\text{kg} \cdot \text{h}^{-1}$ takes place in the micro controller of the device. The signal must be attached in the analogue form of 0–10 V at an implemented analogue input (third from above).

The use of a laptop is recommended in order to visualize the data on board. This laptop can be installed firmly, possibly at the dashboard. The results of the measurement can be pursued and judged ‘real time’ on the screen.

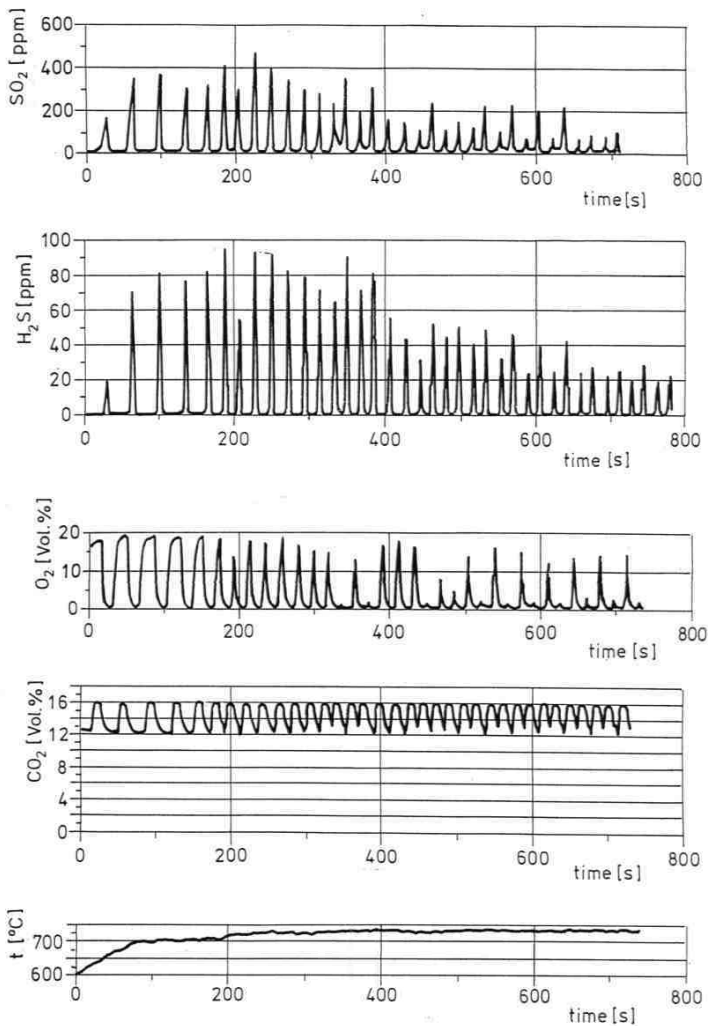


Fig. 25. Distribution of the concentration of SO_2 , H_2S , O_2 , CO_2 and the temperature of the catalyst during a test with the storage catalyst

7. Experimental Results

Fig. 25 shows the tests results of regeneration on an average loaded motor-vehicle with a storage-catalyst at a test-stand.

As characteristics concentration of SO_2 , H_2S , O_2 and CO_2 were represented in volume % as a function of the time. In addition the temperature of the catalyst was also measured during the experiment. An increased cutout from the test-graph is presented in *Fig. 26*.

The results point on a low but existing H_2S -level. For this reason it is very important to keep the sulphur concentration of the fuel as low as possible.

8. Summary

For a successful practical use of NSR catalysts including management the following partially substantial requirements must be fulfilled by the storage systems:

- sufficient NO_x storage in the relevant temperature range
- to increase the storage capacity an oxidation from NO to NO_2 is necessary by the use of the known storage materials
- fast, but also reversible storage of NO/NO_2
- regeneration of the storage under moderate conditions, which doesn't damage the catalyst itself
- general system stability against too high temperatures
- possible neutral behaviour against further exhaust components as for example H_2O , CO_2 and particularly SO_2/SO_3 .

However, lacking stability against SO_2 is unfortunately common to the storage components used now. SO_2 existing in the exhaust gas is stored as sulfate. In particular the SO_2 after oxidation to SO_3 develop the corresponding sulfates, which are thermodynamically far more stable than the nitrates. By moderately adjusted conditions the sulfatisation reveals itself unfavourable to a large extent irreversible.

In principle at higher temperatures ($T > 650^\circ\text{C}$) with fat and thus by reducing conditions over a longer period (> 10 min) a partial regeneration of sulfate can be achieved whereby emission of SO_2 under avoidance of H_2S is desired.

In *Table 3* the two most important possibilities for the NO_x reduction are compared in focus of their most important characteristics. While the SCR technology offers itself mainly for the use in the diesel-commercial vehicles in the future in the modern diesel – passenger car is also foreseen. The storage technology will unfold its advantages especially at the diesel direct injection motor in the motor vehicle. In this case even at diesel motor vehicles large saving potential is available. Due

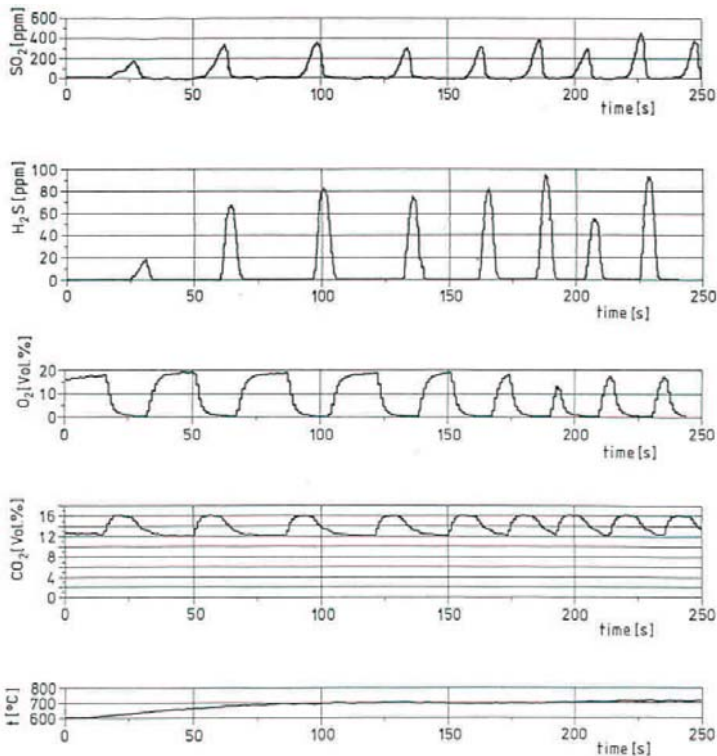


Fig. 26. Increased cutout from Fig. 25

to the drastically lowered limit values for particles in the European Union special interest will have to arise for the decrease of particles emission in research in the future. Measurements for simultaneous reduction of nitrogen oxides and particles (among others under the use of trade-off effect) gain importance in the view that nitrogen oxides are degraded to nitrogen and the particles contain carbon, sulphur (sulfates) and if necessary nitrogen (nitrates) but should only degrade to carbon dioxide, sulphur dioxide and air own nitrogen.

By the storage technology an expressed strong impairment of the forces as a function of the sulphur concentration can be attained, however, by the SCR-technology this influence is smaller.

The recording of the concentration of sulphur with a temporal resolution of 1s is an important measure for the controll of working and regulation phases. A mobile measurement system has been developed to determine the most important physical-chemical parameters and this device can be used long-term and stable both at the test stand and during the drive.

Table 3. Summary of the advantages and disadvantages of the SCR-and NSR-technology

	procedure	catalyst adsorbens	reducing agent	advantages	disadvantages problems
reduction	NH ₃ -SCR selective reduction	TiO ₂ -based vanadium oxide tungsten oxide	ammonia urea or ammonia-carbamate	known technologies high NO _x -conversions	additional tank reducing agent-marketing low temperature problem
adsorption reduction	NSR sorption reduction	precious metals (Pt, Rh) earth alkali oxide (Ba) rare earth oxide (u.a. Cer) wash-coat (Al ₂ O ₃) zeolites	hydro carbon monoxide hydrogen	Analogue 3-way-catalyst Reducing agent Engine generated	sulphur-content of the fuel temporal operation of the motor at $\lambda < 1$ complex motor management

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