

DGMK

Research Report

616-2

AdBlue as a Reducing Agent
for the Decrease of NO_x Emissions
from Diesel Engines of Commercial Vehicles

Part 2:
Laboratory and Field Testing of AdBlue
AdBlue Logistics



Deutsche Wissenschaftliche Gesellschaft
für Erdöl, Erdgas und Kohle e.V.

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DGМК-Research Report 616-2

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Part 2: Laboratory and Field Test Results of AdBlue AdBlue Logistics

Abstract:

In order to comply with stringent exhaust gas emission standards issued by the European Union for 2005 and 2008 for diesel engines of HD (heavy duty) commercial vehicles, the European automobile industry has chosen the „*selective catalytic reduction*“ (SCR) technology. By this method, nitrogen oxide (NO_x) emissions can be reduced by 60 % vis-à-vis current standards. In addition they agreed to use „*AdBlue*“, an aqueous solution of urea, as a reducing agent, which is needed to reduce the oxides of nitrogen into elementary nitrogen. The first vehicles equipped with this new technology will be launched in January 2005.

This report explains the mechanism of the SCR/urea technique and outlines production as well as physical, chemical and environmental properties of urea and AdBlue. It summarises the results of laboratory and field tests, which were run in order to investigate the technical and logistical requirements of this new technique, and it describes first experiences gained at public service stations, which have been equipped with an AdBlue filling station. Finally, the report reflects on what is needed for the build-up of an AdBlue distribution network.

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Deutsche Wissenschaftliche Gesellschaft
für Erdöl, Erdgas und Kohle e.V.

DGMK-Forschungsbericht 616-2

AdBlue als Reduktionsmittel für die Absenkung der NO_x-Emissionen aus Nutzfahrzeugen mit Dieselmotor

Teil 2: AdBlue-Erprobung in Labor- und Feldtesten AdBlue-Logistik

Kurzfassung:

Um die verschärften Abgasnormen für Dieselmotoren schwerer Nutzfahrzeuge zu erfüllen, die die Europäische Union für die Jahre 2005 und 2008 (Euro 4 bzw. 5) erlassen hat, hat die europäische Automobilindustrie sich auf das Verfahren der „*selektiven katalytischen Reduktion*“ (SCR-Verfahren) geeinigt. Mit dieser Methode ist es möglich, die Stickoxid-Emissionen um die erforderlichen 60 % gegenüber den heutigen Standards zu reduzieren. Außerdem hat sie sich darauf geeinigt, „*AdBlue*“, eine wässrige Harnstoff-Lösung, als Reduktionsmittel zu verwenden, das benötigt wird, um die Stickoxide in elementaren Stickstoff umzusetzen. Die ersten, mit der SCR-Technologie ausgerüsteten Nutzfahrzeuge werden ab 2005 auf den Markt gebracht.

Dieser Forschungsbericht erläutert die Wirkungsweise der SCR/Harnstoff-Technologie und beschreibt Produktion sowie physikalische, chemische und ökologische Eigenschaften von Harnstoff und AdBlue. Er fasst die Ergebnisse von Labor- und Feldtesten zusammen, die durchgeführt wurden, um die technischen und logistischen Anforderungen der neuen Technologie zu untersuchen, und schildert erste Erfahrungen, die an öffentlichen Tankstellen mit einer AdBlue-Zapfanlage gewonnen wurden. Schließlich schildert er, was für den Aufbau eines europaweiten, flächendeckenden Verteilungsnetzes für AdBlue erforderlich ist.

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Summary

The Parliament of the European Union has issued stricter emission standards (Euro 4 and 5) for HD diesel engines for the years 2005 and 2008. Compared to current limits, an emission reduction of 60 % for nitrogen oxides and of 80 % for particulate matter will be required.

After testing and comparing various technical options, the European manufacturers of HD commercial vehicles have agreed on **one** method for meeting these standards: the „*selective catalytic reduction*“ (SCR technology), which, in the presence of a reducing agent, converts nitrogen oxides into nitrogen and water, which are natural components of our atmosphere. The European automobile industry has also agreed on **one** reducing agent: an aqueous solution of urea, which will be distributed under the trade name „*AdBlue*“.

This report describes the mechanism and advantages of the SCR/urea technology. The main reason for the decision of the manufacturers of HD vehicles was the fact that this technique, according to current knowledge, appears to be the only method, which enables the necessary decrease of all exhaust gas emissions to the level of Euro 5, while maintaining engine optimization with regard to performance and fuel economy. Furthermore this method can be applied to all types of engines.

In addition this report describes properties and environmental impact of urea as well as its aqueous solution. According to the European product classification guideline, AdBlue poses no serious risk to humans, animals or the environment, if properly handled. Materials used in direct contact with AdBlue must be checked for compatibility with this product. For production, transportation and storage of AdBlue very stringent quality requirements have to be observed, in order to avoid impairment of the efficiency of the SCR catalyst.

Within the scope of this project, a number of pilot and laboratory tests has been conducted, to investigate storage and thermal stability as well as compatibility of AdBlue with other materials. Under correct storage conditions, AdBlue is a stable, easy to handle solution, which does not attack highly alloyed steel, various plastic materials or seals and which does not form stable emulsions with fuel.

In extended vehicle tests, the European automobile industry has investigated the suitability of the SCR technology for decreasing NO_x emissions and of AdBlue as a reducing agent. This report describes test results which were achieved under extreme climatic conditions thus confirming the suitability of the method.

In the course of 2003, the first public service stations have been equipped with AdBlue filling devices. The experiences, gained since then, have confirmed good storage stability and easy handling of the product.

The important European manufacturers of commercial vehicles will offer HD trucks equipped with the SCR/urea technology starting in January 2005. With an AdBlue tank of 100 l capacity, such vehicles have a travelling range of about 6,000 km. In Germany, two thirds of the HD trucks are refuelled at private filling stations of the larger fleet owners, where they could also refuel AdBlue. For an area covering Europe-wide supply of the other vehicles, it would be sufficient, if the mineral oil trade would equip a number of their public service stations with AdBlue filling devices along the main expressways. Starting in 2006, it is planned to also offer lighter trucks and busses with SCR technique. At that time, private filling stations of larger fleet owners

and some public service stations along the main expressways alone may not be sufficient for an area covering AdBlue supply.

In the meantime AdBlue filling equipment is offered, ranging from smaller mobile filling systems to fully integrated filling stations with storage tank, heated pipes, pumps and standardized filling nozzles. The European producers of AdBlue have entered into contracts with distributors of chemicals to establish a Europe-wide distribution network for AdBlue.

Thus the area covering supply of HD vehicles with AdBlue should be secured – at least for the initial phase.

Zusammenfassung

Das Parlament der Europäischen Union hat für die Jahre 2005 und 2008 verschärfte Abgasnormen für Dieselmotoren von schweren Nutzfahrzeugen verabschiedet, die für die Emission von Stickoxiden eine Reduktion um insgesamt 60 % und die von Partikeln um 80 % gegenüber den zur Zeit geltenden Grenzwerten verlangen (Euro 4 und Euro 5).

Nach Erprobung und Vergleich verschiedener technischer Möglichkeiten haben sich die europäischen Hersteller der schweren Nutzfahrzeuge auf ein Verfahren zur Einhaltung dieser Grenzwerte geeinigt: die „*selektive katalytische Reduktion*“ (SCR-Verfahren), bei der die Stickoxide in Gegenwart eines Reduktionsmittels in Stickstoff und Wasserdampf, also natürliche Bestandteile unserer Atmosphäre, umgesetzt werden. Die europäische Automobilindustrie hat sich ferner darauf verständigt, einheitlich eine wässrige Lösung von Harnstoff als Reduktionsmittel einzusetzen, die unter der Bezeichnung „AdBlue“ vertrieben werden soll.

Im vorliegenden Bericht werden Wirkungsweise und Vorteile der SCR/Harnstoff-Technologie beschrieben. Maßgeblich für den Beschluss der Nutzfahrzeughersteller ist die Tatsache, dass diese Technik nach heutiger Kenntnis das einzige Verfahren darstellt, welches die erforderliche Absenkung aller Abgasemissionen auf das Niveau von Euro 5 bei gleichzeitiger Optimierung des Motors hinsichtlich Leistungsverhalten und Kraftstoffverbrauch ermöglicht. Außerdem ist dieses Verfahren bei allen Motoren anwendbar.

Der Bericht beschreibt außerdem Eigenschaften und Umweltverhalten sowohl des Harnstoffs als auch seiner wässrigen Lösung. AdBlue ist gemäß den europäischen Einstufungsrichtlinien bei ordnungsgemäßer Verwendung ungefährlich für Mensch, Tier und Umwelt. Beim Umgang mit AdBlue sind Werkstoffe mit entsprechender Chemikalienbeständigkeit zu verwenden. An Produktion, Transport und Lagerung von AdBlue werden außerdem besonders strenge Qualitätsanforderungen gestellt, damit der SCR-Katalysator durch das Reduktionsmittel nicht in seiner Wirksamkeit beeinträchtigt wird.

Im Rahmen dieses Projektes wurden einige Pilot- und Laborteste durchgeführt, mit denen die Lager- und thermische Stabilität sowie die Verträglichkeit von AdBlue mit anderen Materialien untersucht wurden. AdBlue ist unter normalen Lagerungsbedingungen eine stabile Lösung, die sich gut handhaben lässt, hochlegierte Stähle sowie eine Reihe von Kunststoffen und Dichtungsmaterialien nicht angreift und mit Kraftstoffen keine stabilen Emulsionen bildet.

Die europäische Automobilindustrie hat in ausgedehnten Fahrzeugtesten die Eignung der SCR-Technologie zur Herabsetzung der NO_x-Emissionen sowie der AdBlue-Lösung als Reduktionsmittel untersucht. Der Bericht beschreibt die Testergebnisse, die vor allem unter extremen klimatischen Bedingungen ermittelt wurden und die Eignung dieses Verfahrens bestätigten.

Im Laufe des Jahres 2003 wurden die ersten öffentlichen Tankstellen mit einer AdBlue-Zapfanlage ausgerüstet. Die hierbei gemachten Erfahrungen bestätigten die gute Lagerstabilität und leichte Handhabbarkeit des Produktes.

Mehrere große, europäische Hersteller von Nutzfahrzeugen werden ihre schweren LKWs ab Januar 2005 in einer SCR-Version anbieten. Mit einem AdBlue-Tank von 100 l Kapazität ausgerüstet, haben solche Fahrzeuge eine Reichweite von rund 6000 km. Zwei Drittel der schweren LKWs in Deutschland werden an betriebseigenen

Tankstellen betankt, wo sie dann auch mit AdBlue versorgt werden könnten. Für eine flächendeckende europaweite Versorgung der übrigen Fahrzeuge würde es ausreichen, wenn die Mineralölwirtschaft entlang der Hauptverkehrsrouten einige Tankstellen mit entsprechenden AdBlue-Zapfsäulen ausrüsten würde. Ab 2006 werden auch leichtere LKWs (ab ca. 6 t zGG) und Busse mit SCR-Technik auf den Markt kommen. Für diese Fahrzeuge reichen dann betriebseigene Tankstellen der Fuhrparks und wenige öffentliche Tankstellen entlang der Hauptverkehrswege allein nicht mehr für eine flächendeckende AdBlue-Versorgung aus.

Inzwischen werden AdBlue-Zapfanlagen angeboten, die von einfachen mobilen Füllsystemen bis zu voll integrierten Tankanlagen mit Lagertank, beheizten Leitungen, Pumpen und standardisierten Zapfpistolen reichen. Die europäischen Hersteller von AdBlue haben internationale Kooperationsverträge mit Vertriebspartnern, die sich auf die Distribution von Chemikalien spezialisiert haben, abgeschlossen.

Damit sollte zumindest für die Anlaufphase die flächendeckende Versorgung der schweren Nutzfahrzeuge mit AdBlue europaweit gesichert sein.

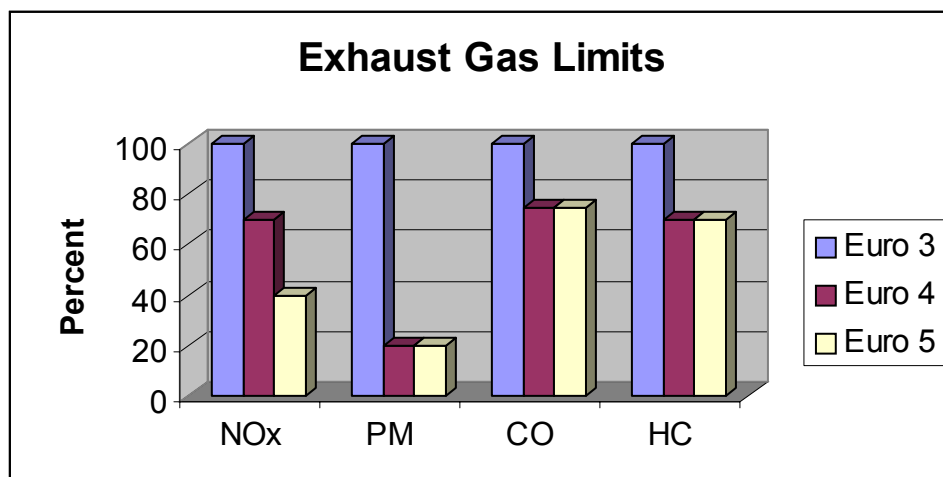
1 Introduction: Reason and Objective

1.1 Euro standards for the reduction of exhaust gas emissions

DGMK Research Report 616 – 1 of September 2003 dealt with the issue: „AdBlue as a reduction agent for the decrease of nitrogen oxide emissions (NO_x) from diesel engines of commercial vehicles“ [1]. As described there, at the end of 1999, the parliament of the EU has issued stricter exhaust gas limit values for diesel engines of heavy duty (HD) commercial vehicles. According to these new standards, NO_x emissions have to be reduced in two steps in 2005 (Euro 4) and 2008 (Euro 5), respectively, each time by 30 % vis-à-vis the current limits of Euro 3. Significantly tighter limits apply to the other pollutants, as well: for carbon monoxide (CO), unburned hydrocarbons (HC) and most importantly for particulate matter (PM) (table 1 and 2).

Pollutant	Euro 3 from 2001	Euro 4 from 2005	Euro 5 from 2008
Nitrogen oxides (NO _x)	5.0	3.5	2.0
Carbon monoxide (CO)	2.0	1.5	1.5
Unburned hydrocarbons (HC)	0.66	0.46	0.46
Particulate matter (PM)	0.1	0.02	0.02

Tab. 1: Exhaust gas limits of the EU standard 99/96/EG for HD commercial vehicles (> 3.5 t; > 85 kW) in g/kWh



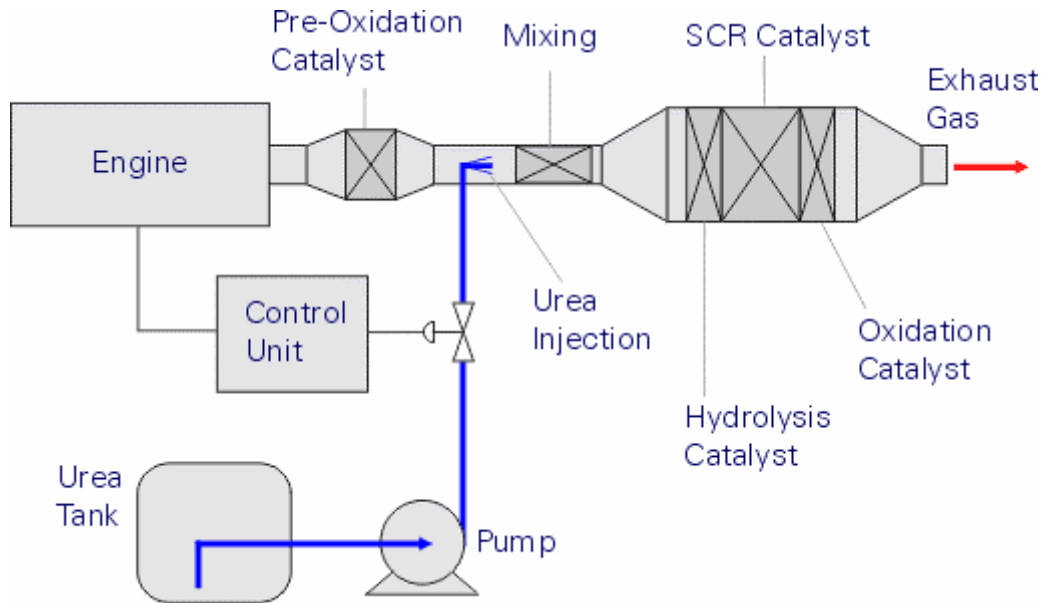
Tab. 2: Exhaust gas limits for HD commercial vehicles in percent vis-à-vis the current standard (Euro 3)

Whereas the limit values of Euro 4 can still be met with various methods, such as exhaust gas recirculation in combination with a particle filter, the simultaneous reduction of NO_x and particle emissions to the values of Euro 5 poses a technical problem, which, according to current knowledge, can only be solved with one specific method described in the following paragraph [3].

1.2 The technology of selective catalytic reduction

After testing and comparing different technical options, the European manufacturers of HD commercial vehicles have agreed on one method for compliance with stricter NO_x limit values: the “*selective catalytic reduction*” (SCR method) [4]. Ammonia (NH₃) is used as a reducing agent; however, due to its aggressiveness and toxicity, it is not applied as such, but in the form of an innocuous substance, which releases NH₃ in the exhaust gas stream, only. The European automobile industry has agreed to use a 32.5 % by weight aqueous solution of urea as a reducing agent, which is distributed under the trade name “*AdBlue*”.

When using the SCR technology, the engine can be operated under optimal conditions, thus minimising fuel consumption and therefore CO₂ emissions as well as the discharge of all pollutants except NO_x. After the combustion gases have left the engine, they first run through a pre-oxidation catalyst where unburned hydrocarbons, carbon monoxide and particulate matter are oxidised as completely as possible. NO is oxidized partly to NO₂, because the subsequent reduction proceeds fastest at a mixing ratio of NO:NO₂ of 1:1. Then a pump, which is controlled by a monitoring unit, injects AdBlue from a separate tank into the hot exhaust gas stream, where it hydrolyses to NH₃ and CO₂. In the actual selective reduction, NH₃ reacts with the NO/NO₂ mixture to form nitrogen and water (steam), which are regular components of our breathing air. This chemical reaction takes place on a catalytic converter consisting of transition metal compounds on a ceramic carrier. Small unused amounts of NH₃ can be oxidised in a subsequent oxidation catalyst (figure 1):



Chemical reactions:

- Partial oxidation: $2 \text{NO} + \text{O}_2 \rightarrow 2 \text{NO}_2$
- Urea hydrolysis: $(\text{NH}_2)_2\text{CO} + \text{H}_2\text{O} \rightarrow 2 \text{NH}_3 + \text{CO}_2$
- Selective reduction: $\text{NO} + \text{NO}_2 + 2\text{NH}_3 \rightarrow 2\text{N}_2 + 3\text{H}_2\text{O}$

Fig. 1: Scheme of a combined deNO_x system for exhaust gas treatment on the basis of the SCR method [4]

The European manufacturers of commercial vehicles have agreed to use SCR/urea technology, because this method offers a number of advantages, which no other method known at this time can provide [5]:

- The engine can be operated at optimal conditions, because the nitrogen oxides formed in the process are converted into elementary and therefore innocuous nitrogen in a second step behind the engine.
- The optimal engine adjustment results in a better efficiency (about 5 % less fuel consumption) and therefore in less CO₂ emissions.
- The discharge of all pollutants (NO_x, PM, CO and CH) is minimised, thereby complying with the stringent requirements of Euro 4 and 5.
- The SCR/urea technology is applicable to all types of engines and functions with diesel fuels of varying qualities.
- The SCR method is maintenance-free and designed for the entire lifespan of the vehicle.
- SCR has no influence on service and oil change intervals of the vehicles.
- In some European countries, SCR equipped vehicles are eligible for certain financial incentives, such as reduced expressway fees (10 instead of 12 cent/km in Germany, starting in 2005, planned in Switzerland and Austria), lower taxes or more favourable depreciation rates.

In the medium- and long-term, a technical and economical alternative to the SCR technology for compliance with the Euro 5 standard is not in sight. This method offers the most cost-efficient solution with regard to environmental protection and efficiency.

1.3 Technical and logistic requirements

After the European car manufacturers had committed themselves to the SCR/urea technology, they then expected that mineral oil industry and trade will provide the required AdBlue - possibly in the same way like fuels – through the public service station net in a Europe-wide and area-covering manner. This expectation resulted in a number of unresolved issues with regard to technical and logistic requirements:

- Standardisation and quality assurance of AdBlue (from product delivery by the producer to sale at the dispense pistol)
- Supply and storage of AdBlue at the service station
- Temperature effects (crystallisation, hydrolysis) and storage stability
- Compatibility with other materials (metals, concrete pavement, seals)
- Behaviour in an oil separator
- Interface filling nozzle/filler neck for the AdBlue fuelling

The automobile and mineral oil industry have run various laboratory, service station and vehicle tests, in order to resolve these questions. The results are described in this research report 616-2 (chapter 3 to 5). In addition, this report contains some considerations about the Europe-wide supply of HD commercial vehicles with AdBlue (chapter 6).

2 Properties of Urea and AdBlue

2.1 Urea

In view of the importance of urea for the application of the SCR method, this chapter summarises the most important properties of this substance and its aqueous solution (see also DGMK Research Report 616-1 and the literature quoted there).

Urea is a colour- and odourless, crystalline substance, which is produced synthetically in large amounts from ammonia and carbon dioxide. It is used mainly in agriculture as a fertiliser and feed additive, but also in the plastic, textile, pharmaceutical and particle board industry. Urea as well as its aqueous solutions (e. g. AdBlue) are of low acute toxicity and do not act as a skin or eye irritant; sensitising, carcinogenic or mutagenic effects are unknown. Therefore, urea is not listed as a dangerous substance according to the Hazardous Substances Ordinance and does not require any labelling according to the Chemicals Act. Urea poses only a small risk to surface waters and is easily biodegradable. According to transport regulations, the aqueous solution of urea is classified as a non-hazardous material and not subject to the Hazardous Incident Ordinance. Urea is classified by the Consulting Committee for environmentally relevant, existing substances of the Society of German Chemists in group III, comprising products which represent a low endangering potential for the general population and the environment according to current knowledge [6].

2.2 AdBlue

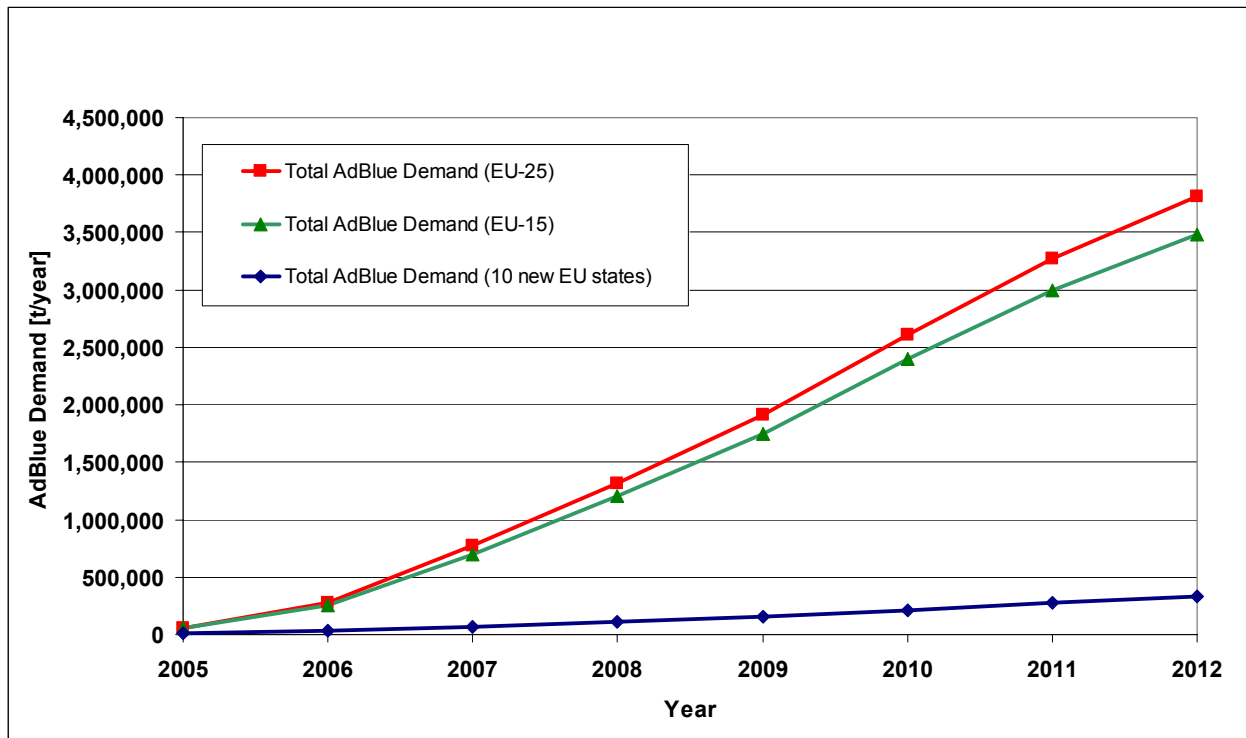
AdBlue is a 32.5 % by weight solution of technically pure urea in demineralized water. This concentration was chosen, because at this urea content, the crystallisation point reaches a minimum of minus 11 °C (eutectic mixing ratio). High demands are made on the purity of the solution, not only during production, but also during handling, transportation and storage. AdBlue must be kept free of any metal ions (e.g. Na, K, Mg, Ca, Al and especially heavy metals), because their salts can clog the pores of the ceramic carrier material or poison the active centres of the catalyst thereby rendering it ineffective [7]. AdBlue is not inflammable, but can release ammonia under heat exposure. According to the European product classification guideline 67/548/EU, AdBlue poses no serious risk to humans, animals or the environment [8]. However, it should be stored separately from nitrates and nitrites.

Urea solution corrodes certain materials, such as copper, zinc and unalloyed steels. Therefore containers, pipes and pumps must be made of stable materials, such as highly alloyed austenitic steels, different plastic materials (polyethylene, -propylene, -isobutylene and -fluoroethylene, Viton etc.) and plastic coated metal containers. Unalloyed and galvanised steels as well as copper and its alloys are not suited. In view of the crystallisation of AdBlue, which starts at minus 11 °C, storage tanks, filling stations and valves must be insulated and heated during the winter, if necessary.

Within Cefic, the European producers of urea have prepared a “*quality assurance guidance document*”, which describes recommended practices for handling, transport and storage of the aqueous urea solution to ensure that the AdBlue quality is maintained from production to the sale to the end-user. This document is shown in

attachment 9.1 of this report, dated 15.07.2004. The newest version can be obtained from the internet [8].

As the European urea producers guarantee, their production capacities are sufficient to cover the AdBlue demand in Europe, which, according to the latest forecast of ACEA, will increase from about 280,000 t in 2006 to almost 4 million t in 2012 (information of ACEA from 2. Nov. 2004).



Tab. 3: AdBlue demand in the European Union according to an estimate of ACEA, November 2004

3 Laboratory and Field Testing of AdBlue

In 2003, the Research Department of Aral, Bochum, has carried out the following tests within the scope of DGMK Research Project 616:

1. Stability tests
 - Storage stability
 - Thermal stability
2. Field tests
 - Handling: filler nozzle/spills/ drippings/cleaning
 - Winter operation (temperatures between -4 and -8 °C)
 - Summer operation (temperatures up to 32 °C)
3. Compatibility with other materials
 - Concrete
 - Metals and plastic material
 - Seals
4. Emulsion test
 - Separation of fuel/AdBlue mixtures

These tests have shown the following results:

3.1 Stability tests

AdBlue was stored at room temperature; after half a year, the solution showed no significant changes of the quality characteristics defined by DIN V 70 070. At the end of the test, all specified values were within the limits of the DIN standard.

In storage stability tests at higher temperatures, slightly different results were achieved: At a storage temperature of 50 °C, only very small amounts of ammonia were developed; after about 100 hours at 70 °C, the NH_3 concentration of the AdBlue solution increased beyond the limit value of 0.2 % by weight as specified by the DIN standard, and it reached a value of 1.4 % by weight NH_3 after 350 hours (attachment 9.2). Based on these tests of Aral, AdBlue is relatively stable at storage temperatures of up to 50 °C; at higher temperatures, however, an increasing hydrolysis of urea takes place.

3.2 Field tests

For these tests, Aral installed an 'intermediate bulk container' (IBC), which was filled with AdBlue. Handling of the urea solution turned out to be without any problems. Stains of smaller drops, which resulted from filling AdBlue with a filler nozzle, could be removed easily. Lasting stains developed only, if these drops were not removed from the concrete floor (B35) within a few days.

Summer operations at temperatures of up to 30 °C confirmed the lab tests about the thermal stability of AdBlue. Apart from very minor smells, no significant changes or nuisance factors could be attributed to AdBlue (the odour threshold value of NH₃ is between 10 and 20 ppm [9]).

Winter operations could not be run, since ambient temperatures did not fall below –8 °C and therefore did not reach the crystallisation point of AdBlue of –11 °C. No problems were observed at a temperature of –8 °C, which prevailed for a few days,.

3.3 Compatibility with other materials

B 35 concrete cubes and plates were exposed to AdBlue and water for six weeks. Subsequently, the solutions were analysed for their calcium content. It was demonstrated that AdBlue does not extract more Ca than water from the concrete, which is used as a pavement at service stations.

Tests about the compatibility of AdBlue with various metals and plastic materials confirm the data as shown in DIN V 70 070. Polysulfide was investigated as an example for seals, and it was found to be stable.

3.4 Emulsion test

Mixtures of AdBlue and diesel fuel were investigated according to an Aral in-house test method, in order to check, if AdBlue forms stable emulsions with petrol or diesel fuel, which could impair the function of the oil separator of a service station. For this purpose, an AdBlue/diesel fuel mixture was stirred in a Turax for 30 seconds at 11,000 rpm. The emulsion formed in the Turax was then centrifuged for 5 minutes at 1,600 rpm. Thereafter, the emulsion had completely separated into an aqueous and a hydrocarbon layer. According to Aral's experiences, these test results show, there are no problems to be expected with emulsion formation in an oil separator.

3.5 Result

The results of the above mentioned pilot and lab tests of the Aral Research Department can be summarized as follows: AdBlue is a stable solution, even under extended time periods, if stored properly. It can be handled without difficulties, does not attack concrete, highly alloyed steels, plastic materials and seals, and it does not form stable emulsions with fuel oils.

4 Field Tests with SCR Vehicles

4.1 Functional tests of DaimlerChrysler

Already prior the decision for the SCR method, DaimlerChrysler had tested ten Mercedes-Benz commercial vehicles over a distance totalling 3.2 million kilometres [10]. The specific purpose of this report is to describe more recent field tests, which were mainly carried out to test the suitability of the method under extreme climatic conditions.

4.1.1 Winter tests in Rovaniemi, Finland, early 2003

The results of the first winter tests with regard to the vehicles can be summarised as follows:

- The components have proven their suitability for winter conditions.
- The heating of the AdBlue system with cooling water was successful.
- The total system of the vehicles needs further optimisation; especially the heating system under extreme conditions requires improvement.
- For the assessment of the total system, additional investigations in a cooling cell and further tests are planned.

The results of these winter tests with regard to the AdBlue filling stations yielded the following:

- In the cooling cell, vehicles could be refuelled at temperatures down to $-20\text{ }^{\circ}\text{C}$ without any problems.
- During subsequent field tests in Finland, the content of the tank was heated to more than $10\text{ }^{\circ}\text{C}$. Nevertheless, filling nozzle and pump occasionally failed.
- The outdoor version of the AdBlue filling station worked satisfactorily at temperatures to $-20\text{ }^{\circ}\text{C}$. At temperatures between -30 and $-34\text{ }^{\circ}\text{C}$, pipes and pumps froze up, resulting in a rupture of an impeller gear tooth. The filling station worked satisfactorily again, after exchange of the impeller gear and raising the temperature over $24\text{ }^{\circ}\text{C}$ by intensified heating.

4.1.2 Winter tests in Rovaniemi, Finland, winter of 2003/04

After initial tests of four vehicles and two filling stations, the vehicles were parked from Dec. 15, 2003 to Jan. 22, 2004. Four freezing cycles with complete freezing and thawing were carried out during this time. No problems were experienced during the subsequent start and operation, but the heating by cooling water required about two hours to thaw the content of a large, completely frozen AdBlue tank. However, by appropriate design of the heating pipes, it was possible, to pump AdBlue out of the tank already after a heating period of about 15 minutes. Since it takes two to three days, until an AdBlue tank is completely frozen – even at very low temperatures – these heating times should not pose any problem in practise, because commercial vehicles are usually operated without any breaks, and therefore, the tank filling is kept at sufficiently high temperatures by the heated cooling water of the engine. Successful driving tests with twelve Euro 4 vehicles at temperatures down to $-20\text{ }^{\circ}\text{C}$ were carried out from Jan. 23 to Feb. 16, 2004.

Heated mobile filling stations of Bott (1000 l IBC) and Ucon (1000 l container) were used for refuelling. The Elaflex filling nozzle was equipped with a magnetic lock; its final cut-off system needs further improvement (too much dripping).

4.1.3 Summer tests in Spain in 2004

Fifteen Euro 4 trucks (Actros, Axor and Atego as well as two city buses, one Unimog and one Econic) were tested in Granada from July 5 to August 2, 2004 at ambient temperatures between $+30\text{ }^{\circ}\text{C}$ and $+40\text{ }^{\circ}\text{C}$. The test vehicles covered a distance of almost 100,000 km.

No functional failures and no breakdown of the exhaust gas after-treatment occurred. Again, filling stations of Bott and Ucon with filling nozzles of Elaflex were used. AdBlue had been produced by Yara.

A total of 1,400 l AdBlue were consumed during these tests. The heavier vehicles consumed between 1.4 and 2.0 l/100 km, the smaller ones 0.8 to 1.4 l/100 km; this corresponds to 4 – 5 % of diesel consumption.

The investigation was focussed on the following crucial points:

- Function of the dosage unit: The control of the dosage unit worked satisfactorily. There were no leaks. Smaller leaks at other locations could be eliminated.
- Testing of the AdBlue tank unit on board the vehicles under extreme heat and road conditions: The vehicles were equipped with aluminium tanks (AlMg₃ alloy); their capacity ranged from 25 to 110 l. There were no malfunctions of the tank unit, even when the tanks on the vehicle were exposed to direct sun light, thus reaching temperatures of 40 to 50 $^{\circ}\text{C}$. There were no leaks at the tanks and no nuisances by odours during the venting of tanks.

- Measurements at the SCR unit at high ambient temperatures: The unit functioned troublefree. All temperatures measured were at target value. Neither the catalytic converter nor the adjacent components reached inadmissible temperatures.
- Testing the refuelling process: The refuelling unit had a pump delivery rate of 30 l/min. After optimisation of the filling nozzle by Elaflex, the cut-off system of the pump worked correctly, when the tank was full. However, the discharge of remaining amounts of liquid after cut-off were too high despite the magnetic cut-off. There were no nuisance factors caused by odours during the refuelling process.

4.1.4 Result

In summary, it can be stated that the SCR technology is a reliable method for the reduction of NO_x emissions; it functions satisfactorily – even under the extreme conditions of a winter in Finland or a summer in southern Spain.

4.2 Functional tests by MAN

MAN conducted the following vehicle tests with AdBlue:

4.2.1 Summer tests in 2003 and 2004

A truck equipped with SCR technology was driven through Spain for three weeks during the summer of 2003. A plastic tank filled with AdBlue was installed on the vehicle. Refuelling did not pose any problems despite high temperatures. There were no noticeable odours. The test was repeated in southern Spain, starting July 7, 2004. Again there were no technical problems or any nuisance factors by odour.

4.2.2 Winter tests in 2003/04

An SCR vehicle was driven through Sweden for three weeks during the winter of 2003/04. It was refuelled at a container service station without any problems – even at temperatures of –32 °C. The heating of the AdBlue tank by the cooling water of the engine was sufficient to keep the AdBlue solution liquid, also at ambient temperatures below –30 °C.

Remark: MAN has decided to comply with the requirements of Euro 4 by measures alternative to the SCR technology. For this purpose MAN uses a further developed system of cooled exhaust gas recirculation in combination with a particle filter in the muffler made of high alloyed steel.

5 AdBlue Testing at Public Service Stations

The first three public service stations were equipped with an AdBlue filling station in Germany during the year 2003:

- OMV service station in Dingolfing, Bavaria. Opening March 3, 2003
- Total service station in Berlin. Opening Oct.10, 2003
- Total service station in Stuttgart. Opening on Nov. 19, 2003

In 2004, OMV, Austria, opened the first Austrian service station with AdBlue at the Inn autobahn in Vomp, Tyrol. In the meantime, OMV has opened further public service stations at an autobahn: Schnelldorf in Germany and Prostejov in the Czech Republic. Additionally, OMV has announced plans to equip 67 service stations with AdBlue filling units throughout Europe by the end of 2007. The company also plans to offer AdBlue in 10 litre cans at 81 autobahn service stations in ten countries, starting in January 2005.

The following is a summary of the experiences and test results gained while handling AdBlue.

5.1 OMV service station at Dingolfing, Bavaria

In early 2003, OMV, Vienna, had agreed to equip the service station in Dingolfing, Bavaria, with an AdBlue filling unit for a field test within the scope of the DGMK Research Project 616-2. On March 3, 2003, the AdBlue test station of Bott was commissioned, which had already been used during the field tests of DaimlerChrysler in Finland (chapter 4.1). The AdBlue unit consists of an above ground IBC plastic container, which was filled with 1000 l AdBlue produced by AMI Agrolinz Melamine GmbH, and a filling unit with a filling nozzle made by Elaflex. In total, eleven trucks were refuelled until Oct. 7, 2003. On Feb. 16, 2004, the IBC was replaced by another one filled with fresh AdBlue.



Figure 2: OMV Service Station in Dingolfing

For quality control of the AdBlue solution, samples were taken as required: directly at the producer and with the filling nozzle from the IBC of the service station at Dingolfing. The results of these samples show that even after one year of storage, all analytical characteristics of the AdBlue solution remained essentially unchanged and within the limits of the DIN standard (attachment 9.3).

These trials demonstrated the suitability of the Bott refuelling unit and the stability of the AdBlue solution. No smell of ammonia was observed, but some minor deposits of crystals showed up at locations, where water could evaporate from the solution, such as the filling nozzle and the filler neck.

5.2 Total service stations at Berlin and Stuttgart

Total opened the first fully integrated AdBlue filling system at a service station in Berlin in October 2003, and one month later another one in Stuttgart (figure 3 and 4). Construction work was done by Union Technik. AdBlue is stored in coated under ground tanks. Pipes leading to the dispensing unit are heated. The integrated filling system was developed by Dresser-Wayne. According to the results of the monthly investigations of the filling units by Dresser and Union Technik, both units are working satisfactorily.



Figure 3: Service Station TOTAL, Berlin Alt-Mahlsdorf



Figure 4: Service Station TOTAL, Stuttgart Ulmenstraße

SKW Piesteritz provided the AdBlue solution and took monthly AdBlue samples from both tanks for analysis (attachment 9.4). As the results show, all analytical characteristics remained practically constant and within the limits of the DIN V 70 070 standard. There was no hydrolytic degradation; the content of heavy metals was below detection limit. This demonstrates that AdBlue is stable for more than one year, if stored appropriately.

6 Logistics

Some of the truck manufacturers will start to launch the SCR/urea technology for their HD diesel vehicles at the beginning of 2005. For a successful market introduction, the automobile industry expects the mineral oil business to provide the required AdBlue in sufficient amounts and of adequate quality – eventually in an area covering Europe-wide manner. They expect a stepwise introduction: first mainly at privately owned filling stations of larger fleets, later at filling stations along expressways in Europe, and finally through the existing network of public filling stations for truck fuelling. The set-up of such an infrastructure, especially in its last phase, would require considerable investments by the mineral oil business for installation of additional dedicated tanks, pipes, pumps and filling units at the service stations. Therefore, this chapter describes a few considerations on what is needed to supply AdBlue for HD commercial vehicles throughout Europe.

Here are some facts about the situation in Germany: In 2003, diesel fuel consumption amounted to 27.9 million t, 25.1 million t of that in road traffic. About 70 % of the total diesel fuel (corresponding to 20 million t) is consumed by commercial vehicles. There are 3.6 million commercial vehicles in Germany (10 % thereof have a load capacity of over 6 t). Approximately two thirds of the operators of these cars refuel at 3,500 privately owned service stations. About 20 % of the commercial vehicles are refuelled at truck stops and only 10 % at public service stations [11].

The normal HD commercial vehicle has a diesel fuel tank size of 1,000 l and an average fuel consumption of 34 l/100 km. It can cover a distance of almost 3,000 km, corresponding to a trip from Copenhagen to Palermo or from Paris to Moscow. The AdBlue consumption amounts to ca. 5 % of the diesel fuel consumption. With an AdBlue tank of 100 l capacity, this vehicle could travel the route Copenhagen – Palermo both ways, without the need to refuel AdBlue. Even at smaller tank capacities, the routes of trucks are planned such that refuelling outside the own refuelling station is unnecessary.

In addition, DaimlerChrysler have announced that they will equip their long-distance trucks with extra large AdBlue tanks with a capacity of 145 l for the interim period, in which AdBlue is not offered everywhere; this would allow a travel distance of almost 10,000 km [12].

These considerations show that about two thirds of all HD trucks in Germany can cover their AdBlue demand at their private service stations – even for trips across Europe, provided the fleet owner has made the necessary investments for AdBlue fuelling.

For this purpose, a number of companies (such as Bott and Ucon) are offering AdBlue dispensing equipment comprising:

- Containers with pump systems, heating and filling nozzles and
- Filling units with heating, flow meters and accounting systems..

This equipment was used successfully in the field tests of the car manufacturers (chapter 4).

The remaining third of commercial vehicles, which refuel at truck stops and public service stations, will also require AdBlue at these locations. However, since these vehicles have the same driving range as the other ones, their AdBlue supply should be sufficiently guaranteed by service stations along the main expressways. This puts the request of the automobile industry into perspective for an AdBlue supply at practically all public service stations in the third and last phase. The mineral oil business could considerably reduce the investment cost by focussing on the service stations along the main traffic routes.

In June 2004, four important European urea producers (AMI, BASF, SKW Piesteritz and Yara Intern.), five manufacturers of commercial vehicles (DAF, DaimlerChrysler, Iveco, Renault and Volvo) and three mineral oil companies (CEPSA, OMV and Total) have committed themselves in a joint press release to the concept of the SCR/urea technology. According to their statement it ideally combines economic and ecological advantages [13].

AdBlue producers (AMI, BASF, SKW Piesteritz and Yara) have announced co-operations with distributor companies (OMV, Penta Chemicals and ERC Technology, Kruse Chemie and Brenntag AG) for the set-up of a German or European AdBlue infrastructure, which could guarantee the supply of the private as well as public service stations. The following supply alternatives are offered:

- in a 10 litre can, especially as a reserve tank or emergency supply,
- in a 1000 litre IBC, which can be equipped with pump, heating, filling nozzle or complete dispensing unit as well as flow meter and accounting system and
- in bulk by tank car for filling 1,000 to 30,000 litre tanks, which can be equipped with heating, insulation, liquid level indicator, complete encasement, filling nozzle or complete dispensing unit, flow meter and accounting system.

The AdBlue demand of HD diesel vehicles can thus be guaranteed in an area covering Europe-wide manner by the privately owned service stations of the fleet operators and the service stations at truck stops and main expressways. There is no need for equipping those service stations, which are not frequented by trucks with SCR systems, with AdBlue filling units.

7 Conclusions

The stricter exhaust gas regulations of the EU, Euro 4 and 5, apply to new types of HD commercial vehicles in 2005 and 2008, respectively, and they become effective for all newly commissioned vehicles one year later. The technology of selective catalytic reduction has proven to be a reliable and safe method for meeting these rigid regulations in a number of field tests, not only in stationary, but also mobile applications – even under extreme climatic conditions.

Starting in 2005, the car manufacturers will offer their customers HD trucks equipped with this technique. The European urea producers will provide the AdBlue needed for the SCR method in sufficient amounts and of required quality, and they have announced co-operations with distributor partners for the set-up of an AdBlue distribution network.

Two thirds of all HD trucks in Germany will cover their AdBlue demand at the service stations of their truck depots, provided the fleet owners have made the necessary investments for AdBlue tanking. The other vehicles can be supplied at truck stops and public service stations.

In the meantime, some mineral oil companies have started to equip their first service stations with AdBlue filling systems. Others plan to offer AdBlue in 10 litre cans at their stations. Due to the relatively small AdBlue consumption of less than 2 l/100 km, vehicles equipped with an AdBlue tank of 100 l capacity can criss-cross Europe without the need to refuel the reducing agent. Therefore the AdBlue distribution network does not need to be too tight.

In view of these facts, the stepwise expansion of the SCR technology should be on the right target in the very near future.

8 References

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9 Attachments

- 9.1 CEFIC: Automotive Grade Urea – Quality Assurance Guidance Document: <http://www.petrochemistry.net/Product & Sector Groups/Automotive Grade Urea>
- 9.2 Thermal stability of AdBlue at 70 °C – investigations by Aral Research Department
- 9.3 AdBlue analytical results and diagram of storage stability of AdBlue at the OMV service station at Dingolfing, Bavaria, investigations by AMI Agrolinz Melamine International GmbH
- 9.4 AdBlue analytical test results of the Total service stations at Berlin and Stuttgart, investigations by SKW Stickstoffwerke Piesteritz GmbH