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Analytical Chemistry at EMPA Dübendorf with Respect to Environmental Technology and Materials Science. Activating Synergy by Networking

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Abstract. For solving complex analytical tasks networking has proven to be extremely useful. Networking in Analytical Chemistry shows various aspects. The use of a pool of different, complementary instrumental methods for ambitious analytical investigations is fundamental. Furthermore, stimulating a transdisciplinary approach through dynamic team formation within a transparent, flexible organization is an essential management task. Two case studies illustrating the networking approach at EMPA in complex analytical service and R & D projects are presented. The result is a comprehensive approach in analytical chemistry, activating synergy.

1. Networking in Analytical Chemistry

1.1. *Analytical Methods, Instrumentation and Quality Management*

EMPA has systematically built up a high level of knowledge and experience in physical and chemical characterization of solid materials, liquids and gases. This is strongly related to the activities in environmental technology, in materials science and in the field denominated as 'Safety and Quality'. The analytical knowledge is based on a sufficient number of adequate methods and a state-of-the-art instrumentation (*Table 1*).

A wide variety of modern instrumental methods [1–3] based on atomic and molecular spectrometry, X-ray spectrometry and mass spectrometry, different types of chromatographic techniques, thermometry and calorimetry are employed on a routine basis. They are supplemented by surface analytical and imaging techniques for solids characterization.

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Table 1. *Analytical Instrumental Techniques Currently Being Operational at EMPA*

Chemical Analysis Services available at EMPA
Analysis – Problem Solving – Contract Research

Elemental	
AAS	Atomic Absorption Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
XRF	X-Ray Fluorescence Spectrometry
ISE	Ion-Celective Electrodes
	Coulometry
	Potentiometry
	Polarography
	Combustion Analysis

Compounds	
MS	Mass Spectrometry
NMR	Nuclear Magnetic Resonance Spectrometry
DSC	Differentail Scanning Calorimetry
TG-MS	Thermogravimetric Analysis Using Mass Spectrometry
UV-VIS	UV-Visible Absorption Spectrometry
FT-IR	*Fourier* Transform Infrared Spectrometry
Raman	*Raman* Spectrometry

Surfaces	
ESCA	Electron Spectroscopy for Chemical Analysis (XPS)
SAM	Scanning Auger Microscopy (AES)
EPMA	Electron Probe Microanalysis
GD-OES	Glow Discharge Optical Emission Spectrometry
TOF-SIMS	Time-of-Flight Secondary Ion Mass Spectrometry

Chromatographical Techniques	
GC	Gas Chromatography
GC/MS	Gas Chromatography Mass Spectrometry
GC/AES	Gas Chromatography Atomic Emission Spectrometry
LC/MS	Liquid Chromatography Mass Spectrometry
HPLC	High Performance Liquid Chromatography
TLC	Thin Layer Chromatography
GPC	Gel Permeation Chromatography
IC	Ion Chromatography
CE	Capillary Electrophoresis

Morphology	
FE-SEM	Field Emission Scanning Electron Microscopy with EDX
AFM	Scanning Force Microscopy
STM	Scanning Tunneling Microscopy
LP	Laser Profilometry

Crystallography	
XRD	X-Ray diffraction, including
	Texture and Residual Stress Analysis

Miscellaneous
- Diffusion Measurements
- Biodegradation and Toxicity Measurements
- Chemical Analysis of Textiles
- Weathering and Aging Measurements

Quality management is specified by official accreditation according to EN 45001. Regular control analysis on reference material samples, as well as active participation in international round-robin tests help EMPA to guarantee high quality standard analytical results both in research and for services.

1.2. *Application, Impact and Trends in Analytical Method Development*

Either a specific analytical method in a specific application or a suitable combination of the different instrumental techniques provide the basis for a distinguished expertise in the different fields of activity. To fulfill the needs of its customers or to open new analytical services, analytical method development, *e.g.*, in specific projects [4], or as a thesis work [5] is a permanent task for EMPA.

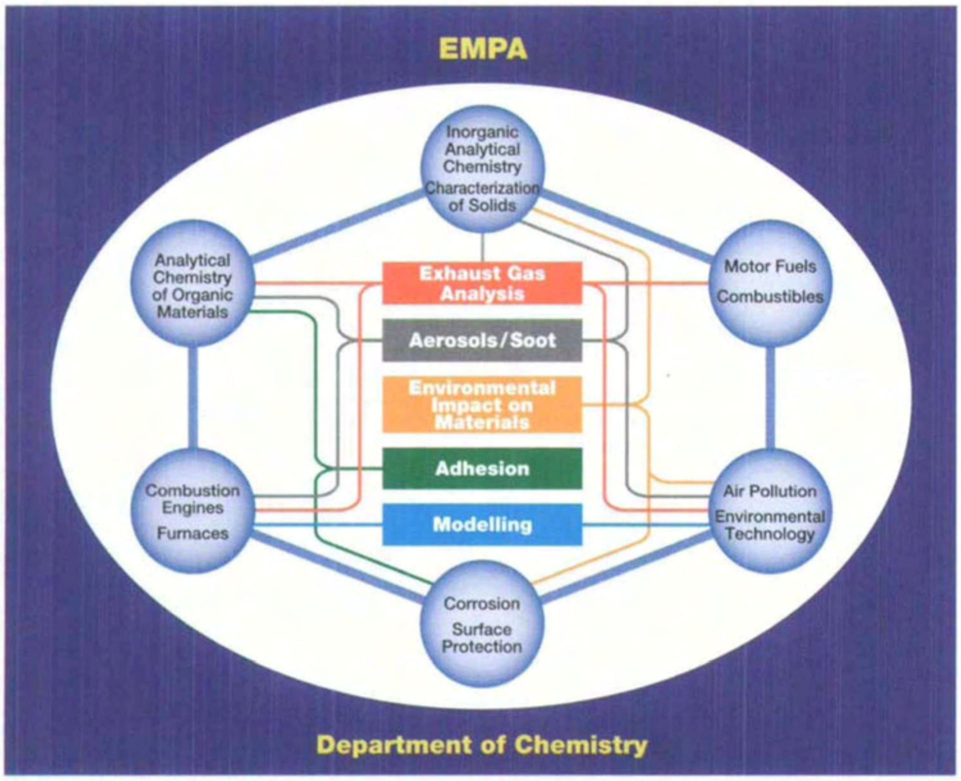

Fig. 1. *Intersectional network of analytical projects at the EMPA Department of Chemistry*

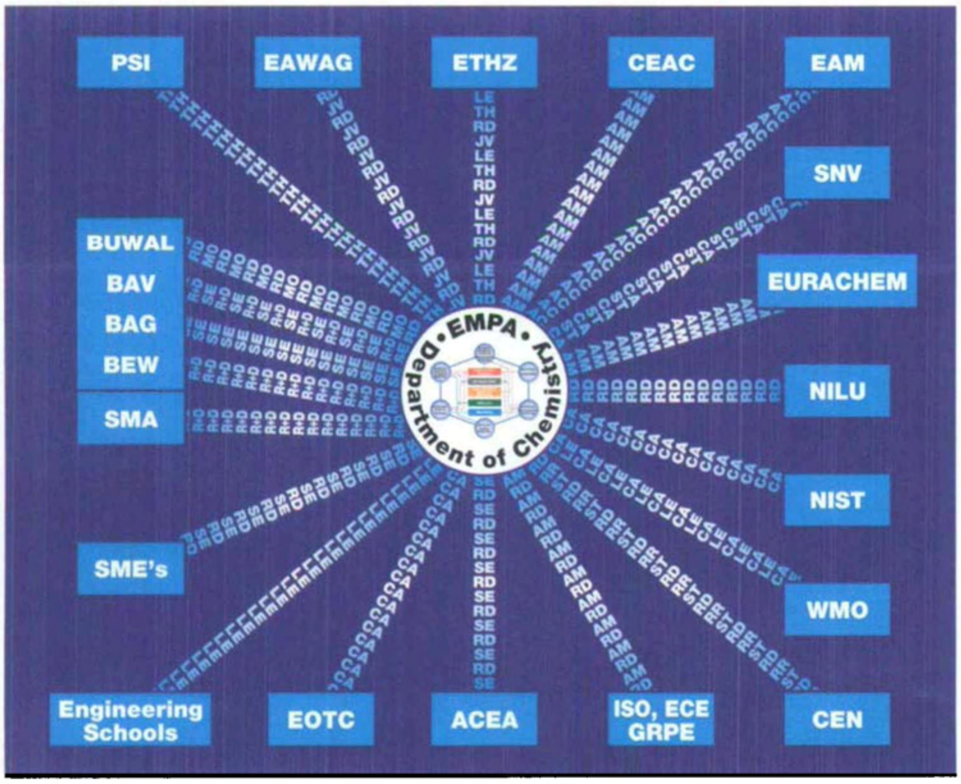

Fig. 2. *External networking of the EMPA Department of Chemistry*

Main features in the development of new methods or for novel equipment installations are: *i*) higher analytical sensitivity for elemental and molecular species leading to improved detection limits, *ii*) increased accuracy in quantifying major, minor and trace components, *iii*) new types of analytical information (*e.g.*, compound identification on surfaces), *iv*) analytical techniques for micro samples with high resolution for local analysis and *v*) improved time resolution in on-line analysis for process control investigations.

1.3. *Interdisciplinary Approach and Collaboration. Internal and external Networking*

Complex analytical problems can be solved by applying a combination of different techniques delivering complementary physical and chemical information in a sort of networking approach. The process of networking is developing on three different levels: *i*) instrumental methods, *ii*) disciplines and fields of application and *iii*) organization.

The free access to all the instrumental techniques for organic and inorganic analysis and the scientific communication between the experts involved is a precondition for success in a multimethod approach, which is increasingly becoming standard practice at EMPA. In this context, 'Project Management' has proven to be a very powerful management tool to support interdisciplinary collaboration. This results in a sort of matrix organization.

Actually, a number of intersectional analytical projects are operating in the Department of Chemistry at EMPA (*Fig. 1*). In each of them, up to five sections are involved simultaneously.

Teamwork is supported by Project Management. One of the main fields of activity is in environmental technology. For illustration, two parent projects involving modern exhaust gas analysis are presented in the second part of this paper.

For a couple of sophisticated, high cost instruments operated at EMPA, *e.g.*, TOF-SI-MS (Time-of-Flight Secondary Ion Mass Spectrometry), NMR (Nuclear Magnetic Resonance), ICP-MS (Inductively Coupled Plasma Mass Spectrometry), and XRF (X-Ray Fluorescence Spectrometry), joint ventures with ETHZ and other associated institutions have been established. The above-mentioned instruments are installed at and operated by EMPA. The benefits of such agreements are savings in

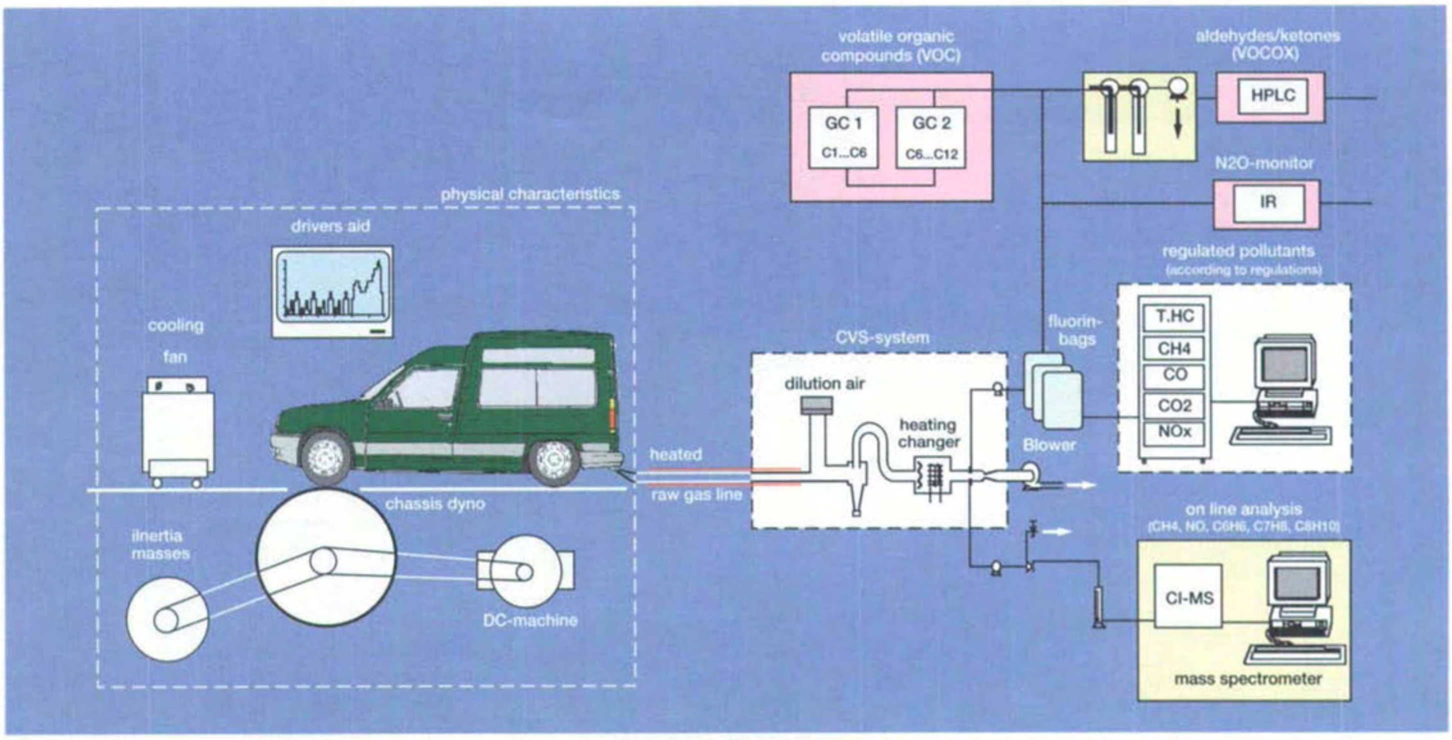

Fig. 3. *Measurement setup on the chassis dynamometer for passenger cars and light-duty trucks.* The following analysis modules were used; 'physical characteristics', 'restricted pollutants', 'green house gases', 'on-line analysis', 'VOC and VOCOX analysis', and 'particulate analysis'.

cost and manpower. In addition, joint ventures of this kind stimulate valuable inter institutional cooperation. In order to strengthen its ties with other analytical laboratories, EMPA has been a founding member of the Center of Excellence in Analytical Chemistry ETHZ (CEAC). Periodical discussions about improvements in instrumental techniques and the exchange of experimental findings from various analytical applications lead to fruitful partnerships, both with external experts and instrument manufacturers. Important external connections to partner institutions and customers are represented in *Fig. 2*.

The process of networking is of strategic interest for EMPA and, therefore, strongly promoted by the management and supported by key people within the organization. At EMPA, this means mainly joining the activities in natural sciences with those of the engineering departments. There is a significant gain of synergy and a stronger position of EMPA compared to its competitors by consequent networking.

2. Two Case Studies in the Field of Exhaust Gas Analysis

Even with modern combustion technologies and efficient methods for exhaust gas treatment, conversion of fossil fuels still results in the production of not only the major components, water and carbon dioxide, but also many non- or partially oxidized compounds which have varying effects on man and the environment. Comprehensive characterization of the emission behavior of stationary and mobile emitters is, therefore, a major task. Emissions produced by internal combustion engines are heavily dependent on the actual engine load. Drawing conclusions about this dynamic emission behavior requires a correspondingly fast detection rate. Time-resolved exhaust gas analysis is, therefore, unavoidable for the characterization of internal combustion engine emissions produced under working conditions.

Two typical examples illustrate how increasingly comprehensive emission analysis is being attained. The first part describes the measurement setup for a chassis dynamometer for passenger cars and light-duty trucks used for the project 'Effect based assessment of automotive emissions' [6]. The second example describes the analysis modules that are used on the dynamic engine test stand for the project 'Influence of modern particulate filter systems on the composition of diesel exhaust emissions'. The significance of the two research projects and the assembly of the analysis modules are briefly outlined.

2.1. *Effect Based Assessment of Automotive Emissions*

Current European exhaust emissions standard regulations apply specifically to gasoline and diesel systems and only partially take into account the particular attributes of alternative fuels such as natural gas. Increasingly, any comparative examination of these three fuels must include an evaluation of environmental impact and energy efficiency.

Therefore, the exhaust gas emissions were evaluated with respect to their ozone-forming potential, the content of greenhouse gases, the carcinogenic potential and their contribution to atmospheric acid formation. In addition, energy consumption per kilometer was determined.

The investigations were carried out on two compact passenger cars (Renault Express) which were in accordance with Swiss road vehicle regulations and which were otherwise comparable in terms of vehicle weight and engine characteristics. The measurements were performed on a chassis dynamometer at EMPA. *Fig. 3* shows the clustering of the analysis units (modules) around the chassis dynamometer that were required in the project 'Effect based assessment of automotive emissions'.

The results show that the emissions produced by the fuels under investigation differ substantially in their effect on man and the environment. The information gained from these measurements was used to determine whether or not the statutory emission standards as applied to natural gas vehicles should be changed so that an effect based comparison of the three fuels can be performed. The findings also form the basis of a 'Recommendation for the assessment of exhaust gas emissions from

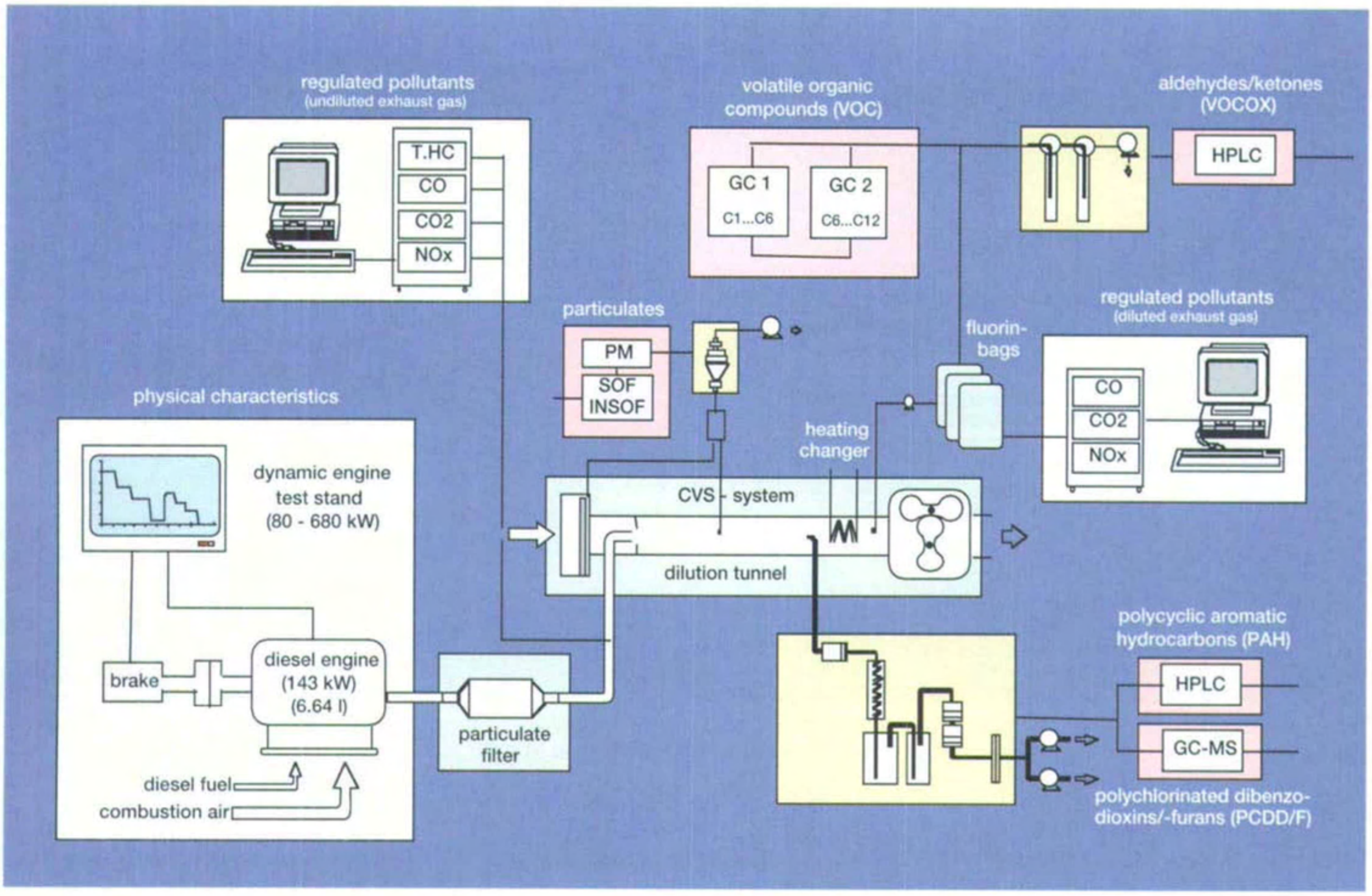

Fig. 4. *Measurement setup on the test stand for heavy-duty Diesel engines* (80–680 kW). The following modules were used; 'physical characteristics', 'restricted pollutants', 'VOC and VOCOX analysis', 'particulate analysis', 'PCDD/F analysis', and 'PAH analysis'.

natural gas vehicles during the type approval test' which was submitted to the Group of Experts on Pollution and Energy (GRPE) acting for the UN Economic Commission for Europe (ECE).

2.2. *Influence of Modern Particulate Filter Systems on the Composition of Diesel Exhaust Emissions*

Due to increased temperatures and pressures in the combustion chamber, diesel engines produce higher nitrogen oxide emissions than comparable gasoline driven engines. In addition, incomplete combustion leads to higher particulate emissions, which are not only hazardous to health but also adversely affect any additional exhaust gas treatment processes.

The emission of particulate matter has already been reduced by optimizing combustion conditions and improving fuel quality. Attempts are being made to further reduce particulate emissions by means of suitable filter systems. Different types of particulate filters are commercially available and, in regular use, demonstrate a clear reduction of the particulate load. Furthermore, burning-off of diesel soot can be improved by means of certain fuel additives. This feature should be increasingly combined with the use of particulate filters.

It is known that certain operating conditions during the treatment of waste incinerator flue gas can lead to the formation of polychlorinated dibenzodioxins and furans (PCDD/F). It is to be expected that a *de novo* PCDD/F synthesis will occur in the temperature range 260–440° if potential chlorine-containing precursors are present. Increased PCDD/F formation has also been observed in the presence of fly ash or certain metal oxides and chlorides. Such *de novo* synthesis phenomena are not only known from waste incineration plants but have also been observed during other cleanup processes *e.g.*, during the catalytic conversion of exhaust gas from gasoline driven engines.

At present only insufficient information is available about the extent to which modern particulate filter technology influences the composition of diesel exhaust gases and is, therefore, the focus of current investigations. A measurement setup has been designed to detect relevant toxic substances emitted by a diesel engine (143 kW, 6.64 l) operated under various load levels according to the test cycle for construction machinery engines (ISO 8178/4 C1). *Fig. 4* shows the analysis modules that were used for the project 'Influence of modern particulate filter systems on the composition of diesel exhaust emissions'.

3. Analysis Modules in Exhaust Gas Analysis

Module 1: Physical Characteristics

In order to calculate the consumption related emission factors from the measured pollutant concentrations, it is necessary to have precise details about fuel consumption and vehicle or engine performance. The volume of the emitted exhaust gas must be known as well as the pressure, temperature and moisture conditions in the exhaust gas. When sampling of diluted exhaust gas is carried out, *e.g.*, by a constant volume sampling-device (CVS) the gas flow and the corresponding dilution ratio must also be determined. Both the chassis dynamometer and the dynamic engine test stand at the EMPA are certified according to EN 45001 and each possess a CVS installation and the necessary infrastructure for standardized measurements according to regulations.

Module 2: Major Exhaust Gas Components and Classes of Regulated Pollutants

Carbon monoxide (CO), the classes of nitrogen oxides (NO_x) and hydrocarbons (T.HC), pollutants that are regulated under statutory emissions standards, are important variables for the evaluation of different emission sources. The content of the major combustion products, carbon dioxide (CO_2) and water (H_2O), are also determined as well as the residual oxygen (O_2) content. The class of nitrogen oxides can, when required, be investigated in more detail. The major component, nitric oxide (NO), can be directly determined by chemiluminescence detection or by chemical ionization mass spectrometry (*Module 4*), and the nitrogen dioxide content (NO_2) can be calculated from the difference between the total NO_x and the NO contents (with or without NO-converter).

Most of the techniques listed (*Table 1*) can be used for both dynamic or off-line analysis. Fast analysis of the CO, O_2 and T.HC contents in exhaust gas helps to identify critical engine conditions which contribute for most of the emissions. *Module 4* describes further possibilities for on line exhaust gas analysis.

Module 3: Greenhouse Gases

The methods for the determination of the greenhouse gases, CO_2 and CO, have already been described. Due to its high warming potential, methane can also contribute to climatic warming. Methane was measured independently by flame ionization detection (off-line) and by chemical ionization mass spectrometry (on-line, see *Module 4*) which gave comparable results. Non-methane hydrocarbon emissions (NMHC) can be calculated from the difference between the T.HC and the CH_4 contents. Nitrous oxide (N_2O) which also has a high warming potential was investigated by means of non-dispersive IR photometry as shown in *Table 2*.

Module 4: On-line Exhaust Gas Analysis

Critical driving conditions can be identified by the use of time-resolved analysis of exhaust gas components. On-line emission measurements using frequencies of 1–5 Hz are necessary to identify the emission peaks that occur for example during runs carried out according to the 'New European Driving Cycle'.

Continuous exhaust gas analysis is performed to measure the major exhaust gas components (CO, NO_x, CO_2 and O_2) as well as the total hydrocarbon (T.HC) group of compounds. In addition, the contents of selected individual pollutants can be analyzed every second by means of chemical ionization mass spectrometry (CI-MS). Up to ten exhaust gas components can be monitored simultaneously. Aromatic hydrocarbons (benzene, toluene, ethyl benzene and xylenes) are investigated in this way as well as methane. *Fig. 5* shows the methane emissions *vs.* time in diluted exhaust gas (CVS system) from natural gas- and gasoline-powered vehicles as determined by CI-MS with xenon as the ionizing gas (Xe^+). Both test runs exhibit typical 'cold-start' behavior with increased emissions at the start of the driving cycle due to the sub-optimal operating temperature of the three-way catalyst.

Module 5: VOC and VOCOX Analysis

Nitrogen oxides (NO_x), volatile organic compounds (VOC) and carbonyl compounds (VOCOX) are all involved in the formation of atmospheric ozone under the influence of sunlight. Control of the secondary air pollutant, ozone, is one of the principle reasons for seeking to reduce the VOC emissions in road traffic. The individual compounds exhibit differing reactivities with respect to the atmospheric ozone formation. Therefore, the total ozone-forming potential (g ozone/km) of an exhaust gas is calculated from the measured content of an individual compound and its specific ozone forming potential. The calculated ozone-forming potential is an

Table 2. *Analytical Methods for the Determination of the Regulated Pollutants and the Major Exhaust Gas Components*

Analyte	Symbol	Measurement method	Analysis
Carbon dioxide	CO_2	non-dispersive infrared photometry (NDIR)	on/off-line
Carbon monoxide	CO	non-dispersive infrared photometry (NDIR)	on/off-line
Oxygen	O_2	paramagnetism	on/off-line
Water	H_2O	dew-point determination	on/off-line
Nitric oxide	NO	chemiluminescence	on/off-line
Nitrogen dioxide	NO_2	difference (NO_x–NO)	
Nitrous oxide	N_2O	non-dispersive infrared photometry (NDIR)	off-line
Nitrogen oxides	NO_x	reduction to NO (converter)	
Total hydrocarbons	T.HC	flame ionization detector (190°)	on/off-line
Methane	CH_4	flame ionization detector	off-line
Methane	CH_4	chemical ionization mass spectrometry	on/off-line
Non-methane hydrocarbons	NMHC	difference (T.HC–CH_4)	off-line

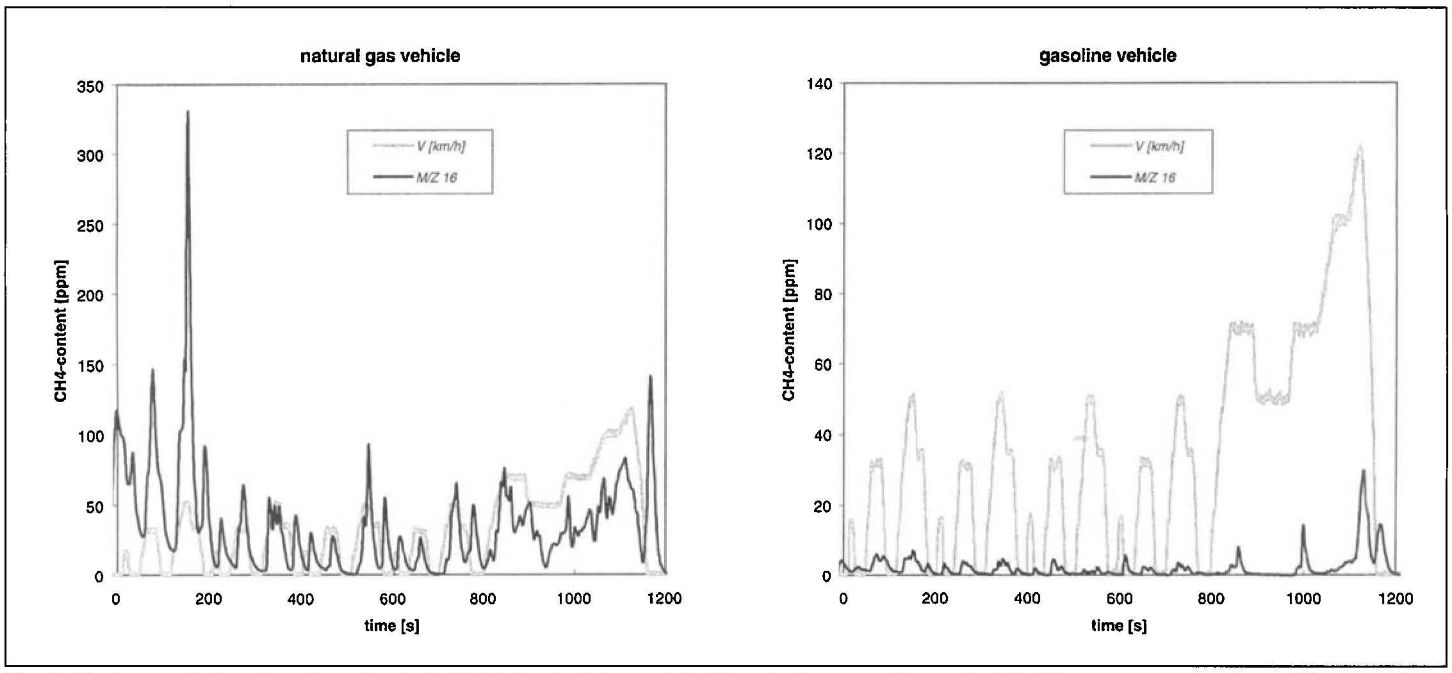

Fig. 5. *Methane emissions* (ppm) vs. *time* (*m/z* 16, Xe^+ as ionizing gas) *in diluted exhaust gas from a fuel flexible passenger car powered by natural gas* (left) *and by gasoline* (right) *during test runs made according to the New European Driving Cycle with its characteristic velocity profile from 0 to 120 km/h* (gray curve)

integral part of the Californian exhaust emissions regulations since 1994 [7].

In practice, the determination of a relevant ozone-forming potential is based on the quantitation of *ca.* 150 components. The exhaust gas profile is strongly dependent on the fuel composition. Individual exhaust gas components are separated and detected by means of gas chromatography (GC) coupled with flame ionization detection (FID) or mass spectrometry (MS) using two different GC systems for light-end HC (C_2–C_5) and mild-range HC (C_6–C_{12}).

Due to their reactive nature the quantitative determination of aldehydes and ketones requires an *in situ* transformation step (chemisorption). Upon sampling the carbonyl compounds are transformed into their corresponding 2,4-dinitrophenylhydrazone derivatives, which are separated and detected by means of high-pressure liquid chromatography (HPLC) and UV detection.

The described methodology includes the determination buta-1,3-diene and benzene as well as formaldehyde and acetaldehyde, which all are designated toxic exhaust gas components referring to the US. Clean Air Act (1990).

Module 6: Particulate Analysis

The characterization of particulate emissions from combustion processes is also increasing in importance. The statutory emissions standards for diesel engines and diesel vehicles relate to the weight of particulate matter emitted and sampled on a filter. Sensitive techniques allow the elemental composition of even small amounts (1–5 mg) of particulate matter to be determined directly on the filter.

A fast qualitative analysis is performed by X-ray fluorescence spectrometry (XRF). X-Ray diffraction (XRD) is used to investigate crystalline components of the particles directly on the filter. Atomic spectrometry methods such as atomic absorption spectrometry (AAS) and inductively coupled plasma optical emission spectrometry (ICP-OES) can be applied once the particulate matter has been dissolved in nitric acid (HNO_3) under pressure in a microwave oven.

The ICP-OES technique is also used for multi-element analysis in diesel fuel and lubricating oil or for the detection of wear elements in lubricating oil (particle size < 20 µm).

The preferred elemental analytical method for particulate matter on filters is, however, plasma mass spectrometry (ICP-MS). Most elements can be determined with high sensitivity with this extremely sensitive multi-element analysis technique. Only the elements C, N, O, and F cannot be detected at all with this method and the elements Si, P, S, and Cl are only detectable with low sensitivity.

The amount of particulate matter which is soluble in organic solvents, soluble in water/isopropanol or insoluble can be determined gravimetrically. The resulting extracts are used for further investigations. The water-soluble fraction may contain ions such as sulfate (SO_4^{2-}), nitrate (NO_3^-) or chloride (Cl^-) which can be quantified by ion chromatography (IC). The organic solvent fraction can be investigated by means of gas chromatography (GC-FID) for the presence of fuel and lubricant components.

In addition to the chemical characterization of the particulate matter, morphological investigations using scanning electron microscopy (SEM) yield important information about the nature of the particles. Not only the total mass of the particles but also the number of particles and the size distribution are important parameters in the toxicological assessment of the particulate matter (lung penetration). Size distributions of particles in the range of 10–1000 nm can now be investigated by differential mobility analysis (DMA) at EMPA.

Module 7: PCDD/F Analysis

Polychlorinated dibenzodioxins and dibenzofurans (PCDD/F) are detectable in trace amounts in the emissions from various combustion processes. Highly efficient chromatographic separation techniques and highly specific detector systems are required for the quantitative analysis of the toxicologically relevant 2,3,7,8-substituted isomers. Since PCDD/F are only present in trace amounts in combustion exhaust gases, large sampling volumes must be taken and subsequently intensively purified. The 17 toxicologically relevant PCDD/F isomers can be separated from the total 210 PCDD/F isomers by means of capillary gas chromatography and subsequently detected with high-resolution mass spectrometry operated at a mass resolution of $m/\Delta m$ 6000–10000.

In untreated waste incinerator flue gases PCDD/F concentrations are typically in the range of 5–10 ng I-TE/Nm^3 and can be subsequently reduced to 0.1 ng I-TE/Nm^3 by means of modern cleaning processes. Concentrations of this magnitude can be detected from a sample volume of 1–2 Nm^3 using a sampling apparatus according to VDI norm 3499 and prEN 1948. Nevertheless, the PCDD/F content in gasoline or diesel exhaust gases is in the range 0.005–0.1 ng I-TE/Nm^3 [8]. Therefore, the sample volume must be increased by a factor 10–50 if an equally sensitive PCDD/F analysis technique is used. PCDD/F are ubiquitous environmental trace contaminants and, therefore, extreme care must be taken to ensure the purity of the solvents and column materials and the cleanliness of the sampling equipment.

Module 8: PAH Analysis

Polycyclic aromatic hydrocarbons (PAH) and their corresponding nitro compounds are formed during incomplete combustion of fossil fuels. The chemical analysis of mixtures of PAH is generally limited to components containing two to six ring systems. The United States Environmental Protection Agency (EPA) has listed 16 PAH including six semivolatile PAH with two or three ring systems. Also included in this list are six PAH which are probably or possibly carcinogenic to humans according to the International Agency for Research on Cancer (IARC).

With increasing ring number PAH tend to bind to particulate matter. Therefore, the sampling devices in use should not change the size distribution of the particles (isokinetic exhaust gas sampling) and the workup procedures should not discriminate semivolatile PAH if two and three ring systems are investigated. Sample preparation according to VDI norm 3872 includes a liquid-liquid extraction step and further sample purification by liquid chromatography using two different stationary phases. Quantitative analysis of the toxicological relevant PAH is performed by means of HPLC with fluorescence and UV detection.

4. Conclusion

A complete assessment of the hazard potential of exhaust gas emissions from different combustion processes demands comprehensive characterization of the individual exhaust gas components. Analysis of the pollutants that are currently recognized as being significant, which can be present in amounts ranging from several g/kg of exhaust gas to only a few pg/kg of exhaust gas, demands versatility and commitment in terms of time and resources.

Equally demanding is the operation of engines and vehicles under defined conditions in order to identify those emission factors that are directly applicable to real operating situations. EMPA is in a position to make a meaningful contribution to this subject area through a transdisciplinary cooperation where engineers and scientists work closely together.

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