



# Partikelanalytik GmbH

## Survey

## Analytical Services

# 2024

TPD  
NLDFT

Functional Materials

Heterogeneous Catalysis

ISEC Frontal Analysis

Nitrogen Sorption Nanotubes

Nanopores Density Bulk Parameters

Break-through Experiments VSSA Interactions TPO

Size Exclusion TPR Proteins Adsorption Nano-Technology Particle Size

EDX Silica Reactive Centres MOF Synthesis Chromatography BJH Ceramics

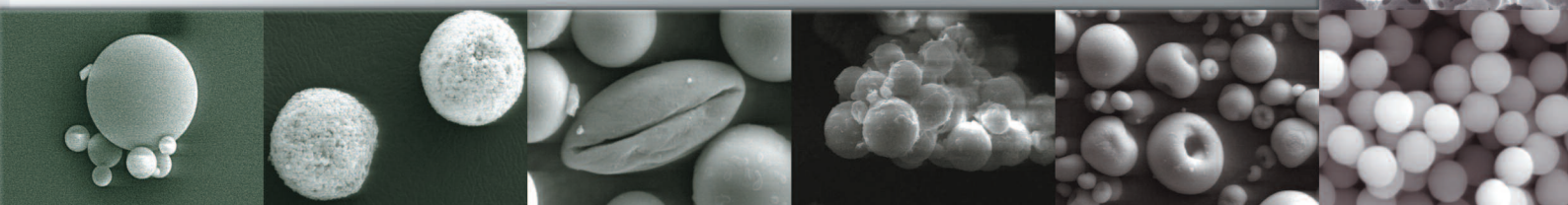
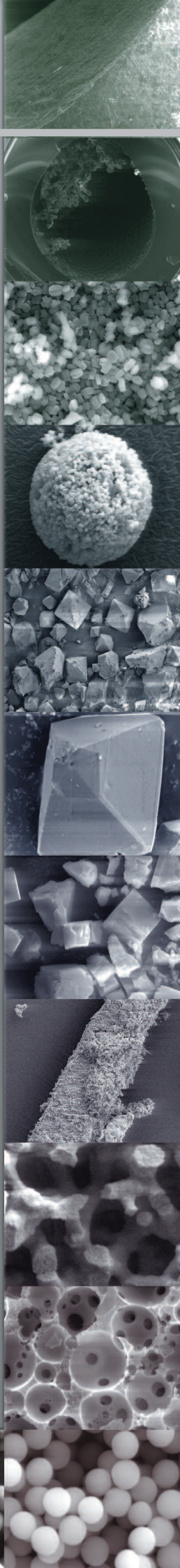
Fillers Laser-Diffraction SEM Isotherms Headspace-GC BET Adsorbent

Particle TG Elemental Analysis Desorption Pore Volume TEM Zeta-Potential

Functionalization Meso Pores XRD Chemisorption Polymer Zeolites Application Pore Size

Nano Particles Phase Development CNT Molecular Sieve Macro Pores Morphology

Surface Area Peptides Micro Pores HPLC Mercury Intrusion Pulse-Sorption



## About „ZetA“

### ...Expertise in Pore System, Surface & Morphology

**Zeta Partikelanalytik GmbH** was founded in spring 2007 as Spin-Off of Prof. K.K. Ungers working group from Mainz university. ZetA is an independant service lab for research, development and characterization of functional materials and their application. Since the end of 2007, "ZetA" operates in well-equipped facilities outside the campus. Our activities are based upon long-term activities in industry-related academic research, enabling us to serve customers worldwide as very effective partner in materials development. At the end of 2022 our strive for quality is documented in ISO9001:2015 certification, covering the whole range of our services.

**We provide** our customers not only with raw measurements and data but also with relevant background information for the evaluation and correct interpretation related to their specific task. This enables our customers to find a solution for a given challenge, while keeping focussed on their own topic.

**Dr. Zöfre Bayram-Hahn** conducted her PhD- Thesis under the guidance of Prof. K.K. Unger at the Institute of Inorganic Chemistry and Analytical Chemistry of Johannes Gutenberg University of Mainz, which she finished 2007. Her main research subject was the synthesis, characterization and optimization of polymer-coated stationary phases for the separation of biopolymers by maintaining their biological activity. This was achieved via systematic variation and optimization of the load and hydrophilic/hydrophobic properties of the polymer-coating and included intense use of chromatographic methods like ISEC (pore

size determination), frontal analysis by staircase-method (adsorption isotherms) or H-vs.-U-curves (mass-transfer based on Knox-equation or van-Deemter) and test for biocompatibility of coated stationary phases (relative enzymatic activity). For optimum reproducibility and comparability of the results she developed well-defined protocols for performing these characterization tasks, now being an important part in the lab services offered by ZetA Partikelanalytik. Ms. Bayram-Hahn is your contact person at "ZetA" with a special expertise for chromatographic sorbents.

**Dr. Andreas Hahn** conducted his PhD- Thesis under the guidance of Prof. K.K. Unger at the Institute of Inorganic Chemistry and Analytical Chemistry of Johannes Gutenberg University of Mainz, which he finished 2003. His main research subject was the investigation of the nucleation and growth process of spherical nano-sized porous silica beads and MFI-zeolites. Therefore he modified and simplified the synthesis route for MFI-zeolites to work under atmospheric pressure to achieve optimum sampling conditions. This enabled the gathering of interesting in-sights in the nucleation process - mainly by em-

ploying specific online-investigations based on neutron- and synchrotron-scattering techniques. He joined Prof. Ungers working group in 1995 and was actively involved in several industry-related research projects covering the whole range of heterogeneous catalysts from synthesis and characterization to the application of sorbents. In his recent project he was investigating materials for the use in heat-storage devices. Mr. Hahn is your contact person at "ZetA" with a special expertise for inorganic sorbents for heterogeneous catalysis (zeolites, MOFs, silica etc.) and for materials with active surfaces.

**Our Services** are based upon long-term experiences in the field of research and development of inorganic sorbents for different applications. In this field, we provide as well analytical services, consulting services or may act as active partner in long- or short-term-R&D-projects. Our special focus is on the optimization of specific surface interactions including ad- and desorption processes. We perform specific measurements, provide relevant data and help with the implementation and development of methods into your own routine analysis - including training and briefing of your staff on-site or at "ZetA"...

This brochure provides an overview about our standard services - please feel free to contact us directly if you like to get more or specific information - or what we could do together: **+49-6131/210 31-23**



## *Dreams are made of this...*

### **...SiO<sub>2</sub> as reliable foundation for tailor-made sorbents**

Quartz sand consists of pure SiO<sub>2</sub>, which is the stuff that “dreams are made” of - and not only this sort of dreams the sandman is responsible for. Materials scientists or solid-state chemists rely upon SiO<sub>2</sub> since a long time for realizing their „dreams“ or ideas of tailor-made sorbents or catalysts. Being both rigid and tunable, SiO<sub>2</sub> is the ideal base for a skeleton, which may be surface-functionalized to deliver any desired interface for molecular interactions. Thus it enables to develop tailor-made and specific sorbents for a large variety of different applications.

In fact SiO<sub>2</sub> is hardly ever used as quartz sand for the design of specific sorbents. The majority of sorbents is either based on the amorphous form, named silica, or, if crystalline species are employed, on zeolites with a well-defined micropore-system. Namely silica offers a huge variability and tune ability of all properties by variation of the synthesis parameters controlling the morphology from the macro- to the nanoscale. Besides a well-defined and large specific surface-area, silica based sorbents may provide a well-accessible, hierarchical pore-system ranging from meso- to macropores with a porosity from zero to several ml/g. And even better, silica is commercially available in a huge variety with different morphologies for all kind of applications, which are just waiting to be modified for a specific application.

At the same time this huge variety offers chances for materials development, it is also a drawback as this task looks like a bottomless pit for the sheer number of possible sorbents to be checked, not to mention investments in specific testing equipment and for gathering the specific know-kow.

And this is exactly what ZetA offers you as a shortcut. Being a fully independent service lab, we provide as well specific know-how to reduce the choice of possible sorbents to only a few, but promising ones and perform meaningful measurements to drive your materials development process to the desired direction.

## *„ZetA“ as partner in materials development*

### **...our focus is your application**

The ideal starting point for the development or optimization process of a sorbent or heterogeneous catalyst is a detailed knowledge of the processes, interactions and conditions of the desired application. Equipped with a wealth of experiences, we can develop an exact idea of how a base material for a desired application should look like. Therefore, the development process starts from a few promising materials rather than using a high-throughput approach.

In heterogeneous catalysis, the intended process or interaction takes place at active sites on the surface of the sorbent. In case silica is used as skeleton, a lot of pathways are available for the modification, enabling to place any desired kind of interaction or any type of active site onto the surface.

The range of procedures for implementing specific functional groups or hetero atoms to the sorbent starts from in-situ methods during synthesis (especially in case of zeolites), to impregnation with salts, hydrophilization or hydrophobization via acid-leaching, functionalization with silanes (for chromatographic sorbents) or polymer coating by physisorption or chemisorption – just to mention a few.

Combined with our long-term experience and know-how, we provide after only a few steps a capable sorbent for your desired application - or provide information for further optimization steps. Materials development with ZetA is a highly efficient and solution-driven process in strong interaction with the customer, where each evaluation step is mostly based on ideas and knowledge rather than employing coincident.

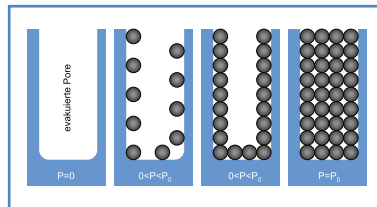


## Characterization in Evaporated State

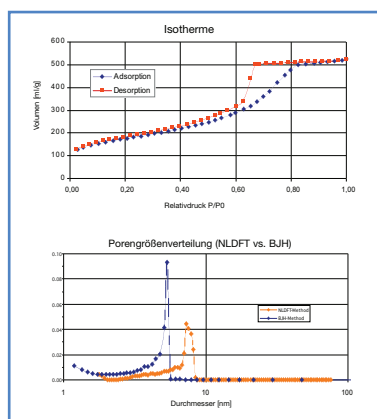
### Standard Surface- & Pore System Investigation - from mm to sub-nm

#### **i** Gas-Sorption / Argon-Sorption@87 K - Nitrogen-Sorption@77 K

**Principle:** in **gas-sorption analysis** the measurement starts from a fully evaporated sample in full vacuum, kept at the temperature of the fluid adsorbate, which is in case of argon 87 K or nitrogen 77 K. The adsorption branch of the **isotherm** is then gathered by stepwise dosing of argon/nitrogen and measuring the resulting relative pressure value. After the maximum pressure value is reached, the desorption branch is recorded by stepwise removing of argon/nitrogen from the sample. All data treatment is based upon the assumption, that the thickness of the adsorbed layer is depending on and increasing with the relative pressure. The knowledge of the layer thickness and the amount adsorbed at any value enables thus the evaluation of parameters like **specific surface area**, **pore volume** or **pore size distribution** based on geometrical considerations.

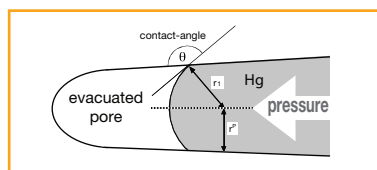


**Data treatment & measurement range:** evaluation of gas sorption data can be performed by employing various data treatment methods, generating valid data for specific pore systems and materials only. The most commonly applied ones are **BET-method** for the determination of **specific surface area** or the so-called **BJH-method** for the determination of **pore volume** and **pore size distribution**, which are based upon the assumption, that the adsorbate layer has the same density and surface tension like the bulk liquid phase. These methods are valid mainly for the comparison of samples of the same type like in quality control. If completely different materials have to be compared, more refined methods should be employed like the recently introduced **NLDFT-method**, which also takes surface-adsorbate interactions into account. Gas sorption enables the determination of both **meso-** and **micro-pores** ranging from 0.4 nm up to 50 nm. Nitrogen-sorption@77 K and argon-sorption@87 K are best suited for the determination of **BET-surface area** of samples from ca. 0.1 m<sup>2</sup>/g up to several thousand m<sup>2</sup>/g.

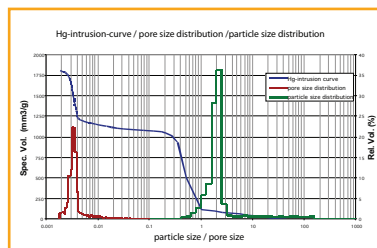


#### **i** Mercury Intrusion Porosimetry

**Principle:** **mercury intrusion porosimetry** starts from an evaporated sample under full vacuum in a dilatometer as measuring cell, kept at a constant temperature of 25 °C. During measurement, mercury is pressed into the sample cell and the intruded volume is recorded with the pressure, giving the **intrusion curve**. For most materials mercury is a non-wetting liquid, therefore it has to be pushed into the pore system of porous media by applying pressure - with a direct correlation between the pressure and the pore size of the sample.



**Data treatment & measuring range:** data treatment for mercury intrusion porosimetry is usually based on the **Washburn-equation**, giving the diameter of the meniscus of the mercury front in dependence of the contact angle and the pressure. Linked with basic assumptions about the nature of the empty space at a given range (slit-pores, spherical pores or inter-particle-space) this enables to gather information in a single experiment about **particle size distribution**, **pore volume**, **pore size distribution** and **specific surface area**. **Mercury intrusion porosimetry** is able to investigate a pore size range from the mm-range down to 1.9 nm (4,000 bar).



### Gassorption Measurements: Nitrogen @ 77 K (or Argon @ 87 K\*)

- GN1** BET Surface Area (5-Point) **DIN ISO 9277 / EuPh 2.9.26 / USP <846>**  
*Determination of specific surface area by nitrogen sorption @77 K (at least 5 data points) according to DIN ISO 9277 (volumetric); at least 1 m<sup>2</sup> surface area of sample required*
- GN4** Micropore Volume / Micropore Surface Area / BET-Surface Area **DIN 9277 & 66134 / ISO 15901-2 & -3**  
*Determination based on t-method or Dubinin-Radushkevich according to DIN 66135-3 (volumetric method); including determination of BET surface area (DIN ISO 9277) by nitrogen sorption @77 K (5-point); at least 1 m<sup>2</sup> surface area of sample required*
- GN5** High Resolution Mesopore Determination / BET Surface / Pore Volume **DIN 9277 & 66134 / ISO 15901-2 & -3**  
*40 points adsorption / 39 points desorption; pore size distribution based on BJH-method (DIN 66134); including pore volume & BET surface area determination (DIN ISO 9277) by nitrogen sorption @77 K (5-point); at least 1 m<sup>2</sup> surface area of sample required*
- GNM** Micropore Analysis **DIN 9277 & 66134 & 66135 / ISO 15901-2 & -3**  
*Micropore size distribution by method of customer's choice (DR, DA, HK, SF, NLDFT, GCMC) according to DIN 66135 parts 1 to 4 by nitrogen sorption @77 K; at least 1 m<sup>2</sup> surface area of sample required*
- GN+** Micropore and Mesopore Determination **DIN 9277 & 9277 & 66135 / ISO 15901-2 & -3**  
*70 data points adsorption / 19 data points desorption; micropore and mesopore size distribution by method of customers choice (BJH, DR, DA, HK, SF, NLDFT, GCMC) according to DIN 66135 parts 1 to 4 by nitrogen sorption @77 K; at least 1 m<sup>2</sup> surface area of sample required*

*\*All measurements can be performed with Argon @87 K - please ask for details.*

### Gassorption with alternative gases

- GK1** BET surface area determination (5 data points) based on krypton sorption for small surfaces
- GCM** Pore analysis on carbons by carbon dioxide measurements  
*Data treatment by method of best choice based on carbon dioxide measurements at 273 K*
- GSX** Customer-specific measurement setup or different non-corrosive gases

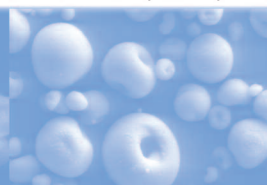
### Volume Specific Surface Area - Characterization of Manufactured Nanomaterials

- VSSA** „Volume Specific Surface Area of Manufactured Nanomaterials" - OECD Test Guideline No. 124  
*Determination of volume specific surface area based on BET-surface area (ISO 9277) via nitrogen sorption at 77 K and skeleton density measurement by gas pycnometry (DIN 66137-2) for characterization of nanomaterials*

### Mercury Intrusion Porosimetry

- Q01** Pore size distribution / pore volume by mercury intrusion porosimetry **DIN 66133 / ISO 15901-1**  
*determination of pore size, pore volume and pore size distribution according to DIN 66133; including record of extrusion curve for further evaluation of rigidity of the sample structure*
- Q02** Extended mercury intrusion porosimetry including determination of porosity **DIN 66133 & 66137-2**  
*same as Q01 with additional characterization via helium pycnometry for determination of sample density (DIN 66137) and porosity in percentage*
- Q0X** Additional sample analysis like comparison between measurements for sample evaluation

Further investigations or characterization methods upon request.



## Investigation in contact with liquid phase

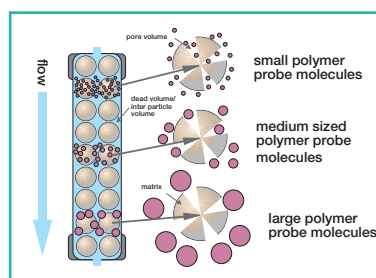
### Chromatographic Methods: Specific Interactions, Pore System etc.

**Sample preparation:** prior to the measurement all samples have to be packed in chromatographic columns, as samples are investigated as stationary phase in contact with the desired mobile phase. Once packed in a chromatographic column, a large variety of different interactions of the sample can be investigated under well-defined conditions. Due to the sensitivity of the equipment, this strategy is not limited to chromatographic media only, but is recommended for any type of sorbent intended to be used in contact with a liquid phase. Please ask for further possibilities to gather meaningful information for your specific sorbent in the desired field of application.



### i Inverse Size Exclusion Chromatography (ISEC)

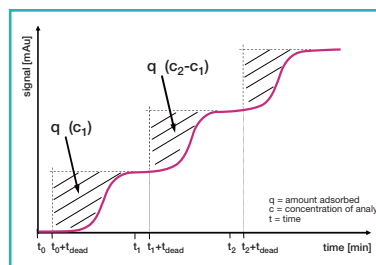
**Principle:** in **inverse size exclusion chromatography (ISEC)** a set of well-defined polymer standards with narrow molecular weight distribution is employed as testing probes to evaluate the pore system of a sample, being packed as stationary phase in a chromatographic column. In **ISEC**, the interactions of the polymers are suppressed by choosing the proper experimental conditions, namely by the proper solvent/polymer-combination. The larger the polymer, the less volume is accessible and thus the earlier the molecule elutes in an ISEC-run. Small polymers can access a larger volume as they may intrude in smaller pores and thus elute later. The results are usually presented by plotting the **capacity factor  $K_{ISEC}$**  (which is related to the elution volume) vs. the radius of gyration of the polymer testing probes.



**Relevance & measurement range:** the evaluation by **ISEC** is based upon the knowledge of the radius of gyration of the polymer testing probes in the given solvent at the given temperature in combination with basic assumptions related to the pore geometry of the sample. In most cases cylindrical pores are describing the situation best. Together with the elution volumes (from the retention time) of the polymers, determination of **pore size distribution** of **meso-** to **macropores** including **pore volume** can be performed. **ISEC** is especially useful for the characterization of **porous polymers** or **membranes** in their swollen state and enable to follow the swelling behavior of samples in contact with different solvents or upon temperature changes.

### i Adsorption Isotherms by Frontal Analysis

**Principle:** gathering information about the adsorption isotherm of an adsorbate in solution is a precise and straight-forward task via chromatographic techniques due to the intense contact and well-defined interaction with the densely-packed adsorbent as stationary phase. In **frontal analysis** the uptake and desorption of analytes is followed via investigation of the response of the sample by designated changes of the adsorbate concentration via **break-through-curves**. Namely employing the **staircase-method** enables to gather these informations in a rather efficient way via stepwise increase of the adsorbate-concentration, where the **break-through** of the **adsorbate front** is clearly visible and detectable as separate steps in the chromatogram.



**Data evaluation:** the time between the concentration change until the **break-through** occurs (minus dead-time) multiplied with the flow rate and concentration gives directly the amount of adsorbate-uptake at the given steps – and thus enables to generate the **adsorption isotherm** under the well-defined conditions of the measurement.



### Sample Preparation for Chromatographic Evaluations

- I0P** Standard-column packing L x ID: 150 x 4.0 mm  
*Requires ca. 2.5 g of packing media, packed by a dry standard-packing-procedure; optimum packing not guaranteed - packing according to customer-specific procedures are welcome and performed without surcharge*
- IXP** Standard-column packing other dimensions  
*Further dimensions from 50 to 250 mm column length are available with an inner diameter of 2,1 - 3,0 - 4,0 - 4,6 - 8,0 - 20 mm determining the required amount of sample; prices for packing of columns with classed chromatographic media are based on a fixed price table; pricing for column packing with other media is based on the required effort for development of the packing procedure; please ask for details*

### High-Resolution Inverse Size-Exclusion Chromatography (ISEC)

- I01** Polystyrene-standards in THF  
*Determination of pore system by parallel pore model (PPM) with 20+ well-defined polystyrene-standards in tetrahydrofuran (THF); standard data treatment based upon peak-maximum-method*
- I02** Polystyrene-standards in dioxane  
*Determination of pore system by parallel pore model (PPM) with 20+ well-defined polystyrene-standards in dioxane; standard data treatment based upon peak-maximum-method*
- I04** Data treatment based upon peak-area method
- IXL** Polystyrene-standards in different solvents
- IXS** Further polymer-standard / solvent combinations

### Adsorption-Isotherm by Frontal Analysis

- HPA** Determination of adsorption isotherm by break-through curves (frontal analysis: staircase-method)  
*Data treatment based on point of deflection, standard protocol includes 10 adsorption steps plus determination of total amount desorbed; determination of adsorption capacity or interaction parameters by customer's choice. Please note, that the price depends on desired mobile phase and desired analytes according to customers choice.*

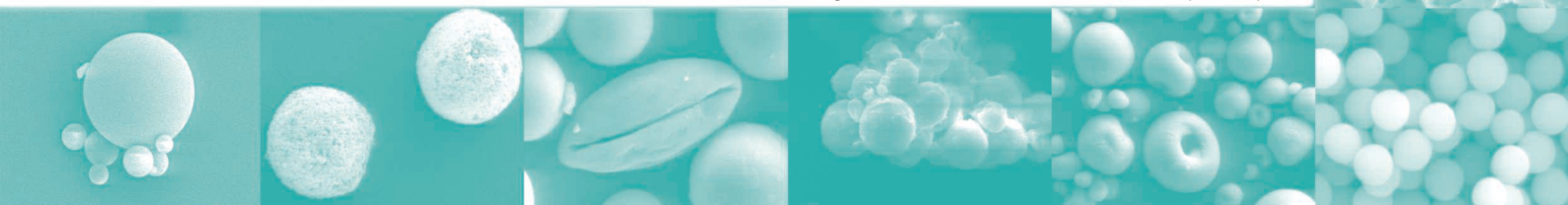
### Investigation of Specific Interactions

- HPW** Investigation of specific interactions by HPLC  
*Determination of relative surface interaction parameters of stationary phase media via the relative retentivity of suitable analytes. Choice of mobil phase and analytes is performed task-specific according to customers requirement.*

### Investigation of Desorption Phenomena in Contact with Liquid Phase

- DSL** Investigation of desorption phenomena / leaching of compounds by HPLC  
*Determination of desorption phenomena or leaching of compounds by HPLC. Choice of leaching conditions and investigation of desorbing compounds as analytes is performed task-specific according to customers requirement.*
- DSG** Investigation of leachable compounds by Headspace-GC  
*Determination of leachable compounds like rest-monomers in soluble polymers by Headspace-GC. Choice of leaching conditions and investigation of desorbing compounds as analytes is performed task-specific according to customers requirement.*

Further investigations or characterization methods upon request.



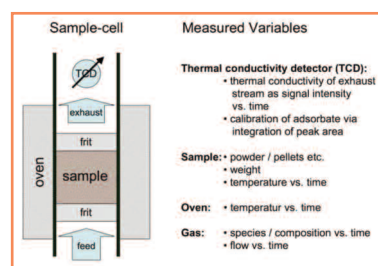
## Dynamic Characterization Methods in Contact with Gases

### Ad- & Desorption of Sorbents and Catalysts in „real-life“-Conditions

**Characterizations under defined gas atmospheres** represent in most cases the „real-life“ situation for the application of sorbents or catalysts. As for the layout and design of adsorbers for air purification or for heterogeneous catalysts for gas-phase reactors: data based on dynamic experimental layouts have a higher relevance for the application than data derived from static methods. Dynamic methods generate integral values by means that they do not give the maximum loadability as in equilibrium, but take kinetic effects into account. Therefore the influence of morphological parameters like crystal size or textural parameters upon the ad- or desorption process may also be evaluated. Furthermore by variation of temperature gradients or gasflow these methods enable to differentiate between single effects and therefore may push further the optimization process in materials development.

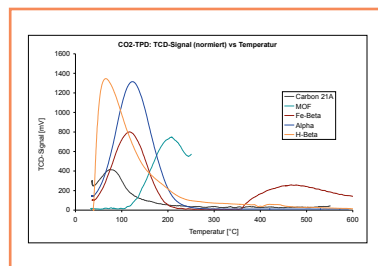
#### i Temperature-Programmed Sorption Techniques

**Principle:** **temperature-programmed sorption** experiments gather information about temperature-dependencies of ad- and desorption processes including chemisorption of samples at normal pressure under defined gas-atmospheres. If only one species is investigated, both sample loading and desorption can be followed via changes in the thermal conductivity of the carrier gas due to changes of the composition employing a **thermal conductivity detector (TCD)** - in other cases a **ms-detector** can be employed. In **temperature-programmed**



**desorption (TPD)** experiments, the desorption process of a previously loaded sample is followed by heating up to 1,100 °C employing helium as carrier gas to gather information about the desorbed amount related to the temperature. **Chemisorption** processes can either be followed at a constant temperature via **pulse-sorption** experiments or via **temperature-programmed reduction (TPR)** / **-oxidation (TPO)** by following the uptake of reactive gases. As the whole measurement protocol is highly flexible a wide area of applications may be evaluated. Even breakthrough-experiments and stability tests might be performed with a very small amount of sample.

**Relevance:** **TPD/R/O** and **pulse sorption measurements** enable to gather quantitative information under dynamic conditions - and thus in a real-life situation - of fast temperature-dependent ad- and desorption processes of sorbents or heterogeneous catalysts, namely for the evaluation of **active sites**. For example ammonia is employed to evaluate the acid strength of materials, while carbon dioxide is used for the determination of basic sites. Information about the **active surface area** of catalysts can either be gained via pulse-sorption of carbon monoxide or hydrogen (Pt, Pd, Ru...), nitrous oxide (Cu, Fe, Zn, Mn....) or by heating a sample under a continuous flow of 5 % hydrogen in argon (**TPR-experiment**)...



#### i Thermogravimetry

**Principle:** following the weight-loss of a sample by applying a well-defined heating procedure is the principle of **thermogravimetry (TG)**, while in the same experiment the energy transfer may also be followed by **differential thermal analysis (DTA)**. Variables of the procedure are mainly the temperature gradient and the composition of the gas atmosphere.

**Relevance:** **TG-curves** give directly the weight-loss of a sample in relation to the temperature, which can be used to design desorption- or cleaning procedures of sorbents. While **DTA-curves** give valuable information if a process is endo- or exothermal under the given conditions by means of gas-atmosphere they allow to gather critical values like a possible ignition temperature during a cleaning procedure.





### Temperature-Programmed Desorption (TPD)

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- TP1** Temperature-programmed desorption of ammonia (standard-protocol up to 650 °C)  
*Evaluation of acid sites, used as fingerprint-technique for the comparison of zeolite samples*
- TP2** Temperature-programmed desorption of carbon dioxide (standard-protocol up to 650 °C)  
*Evaluation of basic sites*
- TPM** Temperature-programmed desorption (employing MS-detector) of ammonia (standard-protocol up to 650 °C) *evaluation of basic sites*
- TPX** Temperature-programmed desorption of specific absorbates by customer-specific protocol

All data provided depending on customer's choice as machine report or Excel data-sheet; customized data formats or customer-specific measurements possible - please ask for details and conditions.

### Chemisorption (TPR / TPO)

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- TC1** Temperature-programmed reduction (TPR)  
*Evaluation of samples like heterogeneous catalysts in a stream of well-defined reducing gas-mixture (5 % H<sub>2</sub> in Ar), employing a linear heating ramp according to customer-specific measurement protocol*
- TC2** Temperature-programmed oxidation (TPO)  
*Evaluation of samples like activated carbons or carbides in a stream of well-defined oxygen-containing gas, employing a linear heating ramp according to customer-specific measurement protocol*
- TCO** Chemisorption analysis by pulse-sorption/-titration with carbon monoxide (CO; DIN 66136-3)  
*Evaluation of active metal surface area of noble metal containing catalysts, metal dispersity, median crystal size, including sample preparation according to customer-specific protocols. Other pulse-gases available upon request.*
- TNO** Chemisorption analysis by pulse-sorption/-titration with nitrous oxide (N<sub>2</sub>O; DIN 66136-3)  
*Evaluation of active metal surface area of copper-containing or related catalysts, metal dispersity, median crystal size, including sample preparation by customer-specific protocol.*
- TCH** Chemisorption analysis by pulse-sorption/-titration with hydrogen (H<sub>2</sub>; DIN 66136-3)  
*Evaluation of active metal surface area of noble metal containing catalysts, metal dispersity, median crystal size, including sample preparation according to customer-specific protocol.*
- TCX** Further data treatment or special sample pretreatment according to customers protocols

All data provided depending on customer's choice as machine report or Excel data-sheet; customized data formats or customer-specific measurements possible - please ask for details and conditions.

### Investigation of Multi-Component Desorption Phenomena in Gas Phase

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- TSG** Investigation of desorption phenomena of compounds by Headspace-GC  
*Determination of desorption phenomena of multicomponent-systems by Headspace-GC after heating up to defined temperature. Choice of leaching conditions and investigation of desorbing compounds as analytes is performed task-specific according to customers requirement.*

Further investigations or characterization methods upon request.



## Special Techniques

### Sometimes Chemistry Matters...

#### X-Ray Powder Diffraction (XRD)

- X01** X-Ray Powder Diffraction by  $\text{Cu}_{\text{K}\alpha}$  / range:  $5^\circ - 45^\circ = 2\tau$
- X0X** Additional range, customer-specific setups, data treatment, crystallite size by Debye-Scherrer etc.

#### Elemental Analysis

- E01** Determination of C, H, N in % via digestion in O<sub>2</sub>-Stream according to DIN 51732
- E02** Determination of C, H, N, S in %
- E0A** Elemental distribution atomic absorption spectroscopy (ICP/OES) according to DIN EN ISO 11885 (E22)
- RFA** Elemental distribution (screening) via RFA according to DIN EN 15309
- E0X** Additional elements, determination by further methods, trace analysis etc.

#### Bulk Parameters

- PYK** Determination of skeleton density by gaspyknometry (He) according to DIN 66137-2 / ISO 12154
- SD0** Shipping weight according to DIN ISO 60
- SWT** Angle of repose according to ISO 4324 (Funnel) / European Pharmacopeia 2.9.35
- SV1** Particle size distribution by sieving analysis according to DIN 66165
- TrV** Inner water content: loss on drying at 106 °C according to DIN 51718
- TrV30** Outer water content: weight loss at 30 °C vDIN 51718
- WRV** Water retention value according to ISO 23714:2014-01
- CrS** Crushing strength (equivalent to DIN 8948 Part C), minimum particle size is 0.5 mm

#### Zeta-Potential

- Z01** Determination of zeta-potential via electroacoustic-spectrometer *measurement of colloidal vibration-current and determination of zeta-potential as is (original conc.) up to 50 Vol. %*
- ZpH** Determination of zeta-potential as function of pH (titration) *including Z01, with additional measurements at different pH-values (between pH 0.5 (min) to pH 13.5 (max)) and determination of isoelectric point*

#### Laser Granulometry

- LG1** Particle size distribution by Laser diffraction in water  
*Determination of particle size distribution according to ISO 13320-1 in water; size range 100 nm to 2 mm; sample pretreatment as customer's choice*
- LG2** Particle size distribution by Laser diffraction in air  
*Determination of particle size distribution according to ISO 13320-1 in air; size range 100 nm to 2 mm; sample pretreatment as customer's choice*
- LGi** Particle size distribution by Laser diffraction in isopropanol  
*Determination of particle size distribution according to ISO 13320-1 in isopropanol; size range 100 nm to 2 mm; sample pretreatment as customer's choice*
- LGX** Particle size distribution by Laser diffraction in other solvents
- DLS** Particle size distribution by Dynamic Light Scattering technique  
*size range 0.5 nm – 10 µm; sample pretreatment as customer's choice*

Results will be sent depending on customer's choice as original-machine report or Excel data-sheet. Customized data formats and customer-specific measurements also possible - please ask for details and conditions.

Please ask for further investigation methods like NMR, IR, XPS etc.



## Imaging Techniques

### A Picture is Worth a Thousand Words ...

#### Scanning Electron Microscopy (SEM)

- RM1** Sample screening by scanning electron microscopy
- RM3** Particle size distribution via image analysis from SEM images
- RMD** Sample preparation by embedding in conductible resin incl. thin-section
- RMX** Sample report according to customers demands

Images will be provided via e-mail as TIFF- or any other desired format. Particle size distributions are provided as Excel data-sheet according to customers requirements.

#### High-Resolution Scanning Electron Microscopy (HR-SEM)

- HREM** High-Resolution scanning electron microscopy including at least 5 images on HR-SEM-instrument (spotsize down to 0.2 nm) including sample preparation
- Nano** Investigation of samples via HR-SEM including sample preparation regarding classification as nanomaterial including evaluation of particle size distribution and report

#### Elemental Mapping via SEM/EDX

- RXP** Sample preparation
- RXL** Evaluation of elemental distribution in SEM-mode via SEM/EDX
- RXM** Element mapping in SEM-mode as surface-scan via SEM/EDX
- RXE** Elemental analysis by EDX
- RXX** Sample report according to customers demands

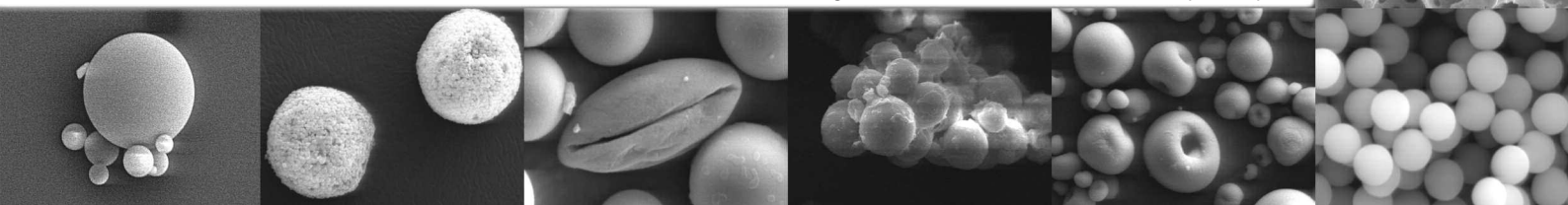
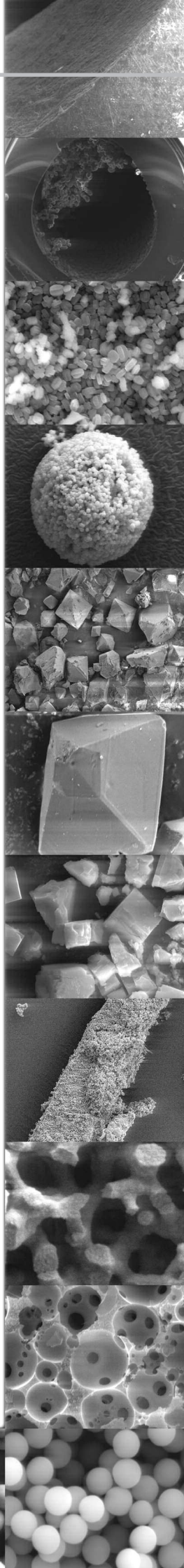
Images will be provided via e-mail as TIFF- or any other desired format. Elemental distributions are provided as Excel data-sheets or TIFF-files according to customers requirements.

#### Transmission Electron Microscopy (TEM)

- TM1** Sample screening of particulate nano materials by transmission electron microscopy  
*Morphological determination of particulate materials like nano crystalline powders, fibers etc. < 100 nm, which do not need elaborated sample preparation steps before examination*
- TMX** Sample screening by transmission electron microscopy with elaborated sample preparation  
*Investigations by transmission electron microscopy usually require complex sample preparation steps, depending on the type of sample and the desired information. Possible steps are chemical contrasting and embedding in resin prior to microtome slicing, enabling localized elemental analysis. Quotations can be given on the basis of a detailed description of the sample nature and questionaired problem.*

Images will be provided via e-mail as TIFF- or any other desired format. Elemental distributions are provided as Excel data-sheet according to customers requirements.

Further investigations or characterization methods upon request.



## **Analytical Services - Processing Information**

**Pricing:** all prices excluding VAT. without responsibility for the correctness of the information. Prices are subject to change without notice. Errors and omissions excepted. Repeated measurements of a sample, which are based upon different weighted samples are charged as individual samples.

**Duration:** samples are processed in the order of arrival. Processing times are depending on various factors like characterization method, specific queue status - and required or desired sample preparation procedure. Single samples for the following analytical methods are usually measured in 5-10 business days: gassorption measurements (mainly BET-surface area), particle size by lasergranulometry, He-pycnometry, high-resolution inverse size-exclusion chromatography, CO<sub>2</sub>-TPD / NH<sub>3</sub>-TPD, TPR and pulse-chemisorption with CO, H<sub>2</sub> or N<sub>2</sub>O. A multitude of samples may take longer processing times - please specify if needed samples with higher priorities, which we will process first. Upon request, results will be send directly after measurement per e-mail or fax. We will inform you if longer processing times are expected.

**High-priority samples:** upon special request samples may be processed with extra-high priority. This usually requires work overtime and therefore an 50 % surcharge is added to the normal price. Please ask for details, expected processing times and possibilities for speeding up.

**General information:** if possible, please send samples with a safety data sheet for handling and correct disposal or returning of samples and information for the desired sample treatment. Please indicate the desired analytical methods or protocols, eventually the sample priority and your contact information, if further questions arise. Please let us know if you wish to receive the results as machine report or as Excel data-sheet, containing the results as graphical illustrations if applicable. Samples are kept in house at least 4 weeks after the measurements, enabling to perform additional experiments, if required. Afterwards samples may be sent back or disposed - depending on customers choice.

**Terms of delivery:** sending back of non-hazardous samples is free of charge to customers in Germany starting from a net order value of EUR 500.--. Below this net order value a fixed shipping rate of EUR 25.-- is charged. Terms and conditions for delivery abroad on request.

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