

Central Department for Chemical Analysis

The Central Department for Chemical Analysis

Since 1893 the chemical and analytical research laboratories at Heraeus are primarily specialized in the analysis of metals and inorganic substances.

Since then tasks, equipment and procedures have been constantly adapted to new requirements. The scope of supply and services is continuously increasing. Besides inorganics, driven by the demand of internal and external customers, we are also analysing a large range of organic materials. The wide range of products and the great number of different raw materials and products of our company call for a high degree of analytical experience, flexibility and effectiveness.

With a team of 50 employees at present, using traditional and ultra-modern procedures of analysis, we are able to satisfy all requirements.

We also wish to offer our knowledge and our instruments for analysis outside of our company. A long experience in collaboration with customers and suppliers substantiates our analytical performance so that we are approved as a laboratory for umpire analysis.





Heraeus

Since 155 years Experience in the Production and Analysis of Precious Metals and other Materials

Heraeus, the precious metals and technology group headquartered in Hanau, Germany, is a global, private company in the business segments of precious metals, dental health, sensors, quartz glass and specialty lighting sources.

With revenues of more than EUR 9 billion and more than 10,600 employees in over 100 companies, Heraeus has stood out for more than 150 years as one of the world's leading companies involved in precious metals and materials technology. Founded in 1851, the company's broad product portfolio has made Heraeus relatively independent of developments in individual industrial sectors.

Traditionally Heraeus strengthens his international market position and innovation leadership in different industrial domains by the development of products (that are characterized in most of the cases through an extreme specialization) close to the customer and through well directed acquisitions. Additionally we have a extensive operating experience with high temperature and exceeding and superior materials as well as in handling and analysing precious metals and other materials.

Special Features in The Analysis of Precious Metals

The in-depth knowledge of our analysts and their experience, together with the wide range of specialized standards, enables us to determine the contents and purity of precious metals as well in raw materials and products to a high degree of accuracy. For this purpose there are three types of analysis:

Accounting and Umpire Analysis

are used for the determination of the exact value for the content of precious metals in raw materials and products, serving as an accurate payroll basis for customer and producer (e.g. ore concentrates of precious metals, spent catalysts, jewellery scrap, electronic scrap etc.)

Survey Analysis

permits an economic and rapid identification of the elemental composition, with a satisfying degree of accuracy. This type of analysis serves primarily for the production control.

Purity Analysis

permit the determination of the purity of raw materials and product. Impurities of metal and non-metal samples will be determined by trace analysis. For almost all kinds of analysis a specific sample preparation is required.

■ **The Fire Assay**

Fire assaying is the oldest and most reliable method of determining the content of precious metals in solid samples, based on metallurgical procedures. In a melting process the precious metals are separated from the base metals with the aid of a collector and are subsequently refined. This is a very simple but effective enrichment procedure for precious metals. Fire assaying is the first step for the determination of the precious metals concentration of raw materials by wet chemical or spectroscopic methods.

■ **The Wet Chemical Analysis**

For most instrumental analytical techniques a sample preparation is required to dissolve the precious metals. The classical route for the quantitative determination



Au-granulate

of a precious metal is its isolation from a solution in a weighable form. The accuracy of gravimetric determination of precious metals is still unsurpassed. However, the wet chemical analysis is being replaced more and more by fast spectroscopic methods for solutions.



Sample preparation balance



Sample preparation



Sample preparation – Muffle furnace

■ **The Spectroscopic Analysis**

Spectroscopic methods are routinely used in the manufacturing and quality control of our production processes, as well as the purity check of precious metals materials. The most widely used and well established technique

for trace elemental analysis in solutions is the inductive coupled plasma spectrometry (ICP-OES). Further also atomic absorption is applied.

For direct solid sample analysis x-ray fluorescence, glow discharge (GD-OES) and spark discharge are used.

Method Description

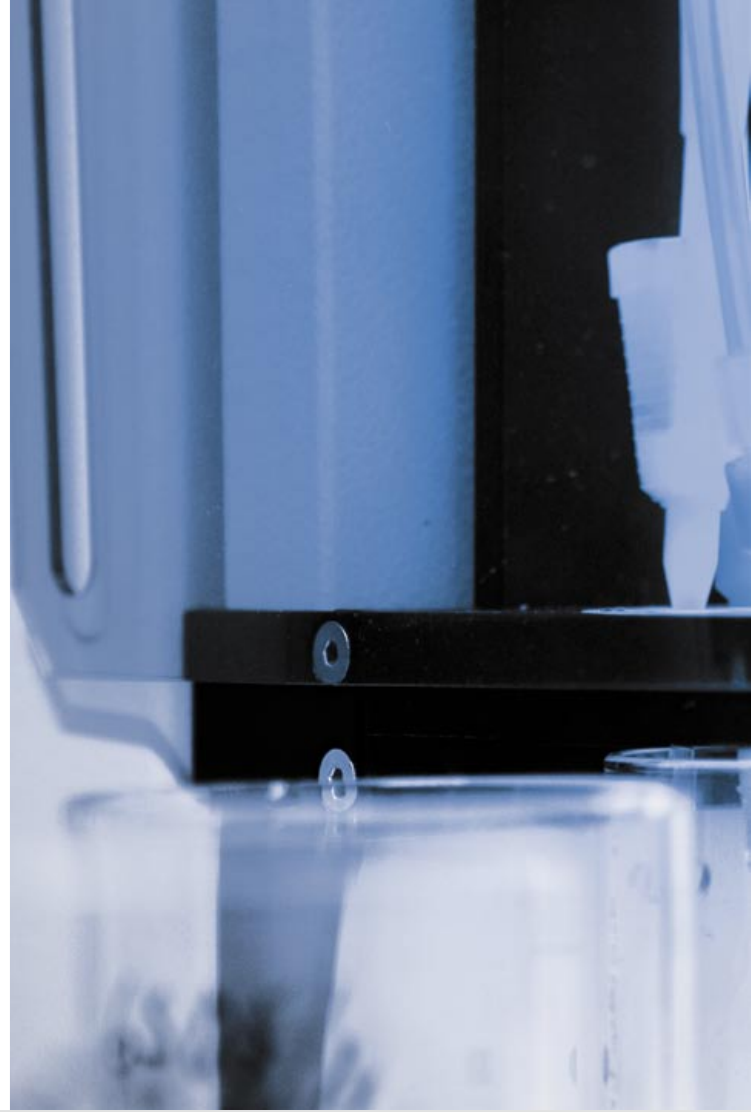
Main Focus Precious Metals

Energy dispersive X-ray fluorescence

In addition to the wavelength dispersive X-ray fluorescence, which serves primarily the accurate quantitative determination of precious metals alloys in the percent range, the energy dispersive X-ray fluorescence is used for a rapid determination of a survey analysis as well as for the determination of the quantitative composition of solid materials.

X-Ray Powder Diffraction

Since 1983 the analytical department of Heraeus has been equipped with a x-ray powder diffractometer. It is designed for the qualification and semi-quantification of crystalline phases in samples at room temperature.



X-Ray Diffractometer



Thermo-Microbalance Scale



ICP Spectrometer

Data evaluation is made by comparison of the data obtained from the sample with the data of pure substances stored in a special data base. We can employ the Bragg-Brentano and the transmission method. The X-ray powder diffraction analysis supports the development and the production of a variety of Heraeus products.

Thermal Analysis

The thermal analysis generates a large amount of physical and chemical information about a sample. The samples

are exposed to a defined modification of temperature. Information about characteristic parameters of the sample or the clarification of the chemical behaviour can be achieved. In our house the thermal analysis is primarily used in the development of solder pastes and conductive adhesives with special properties. This techniques also serves for quality control of raw materials and products



IC and high temperature pyrolysis furnace



Glove discharge spectrometer



Coulometric titration

Potentiometry

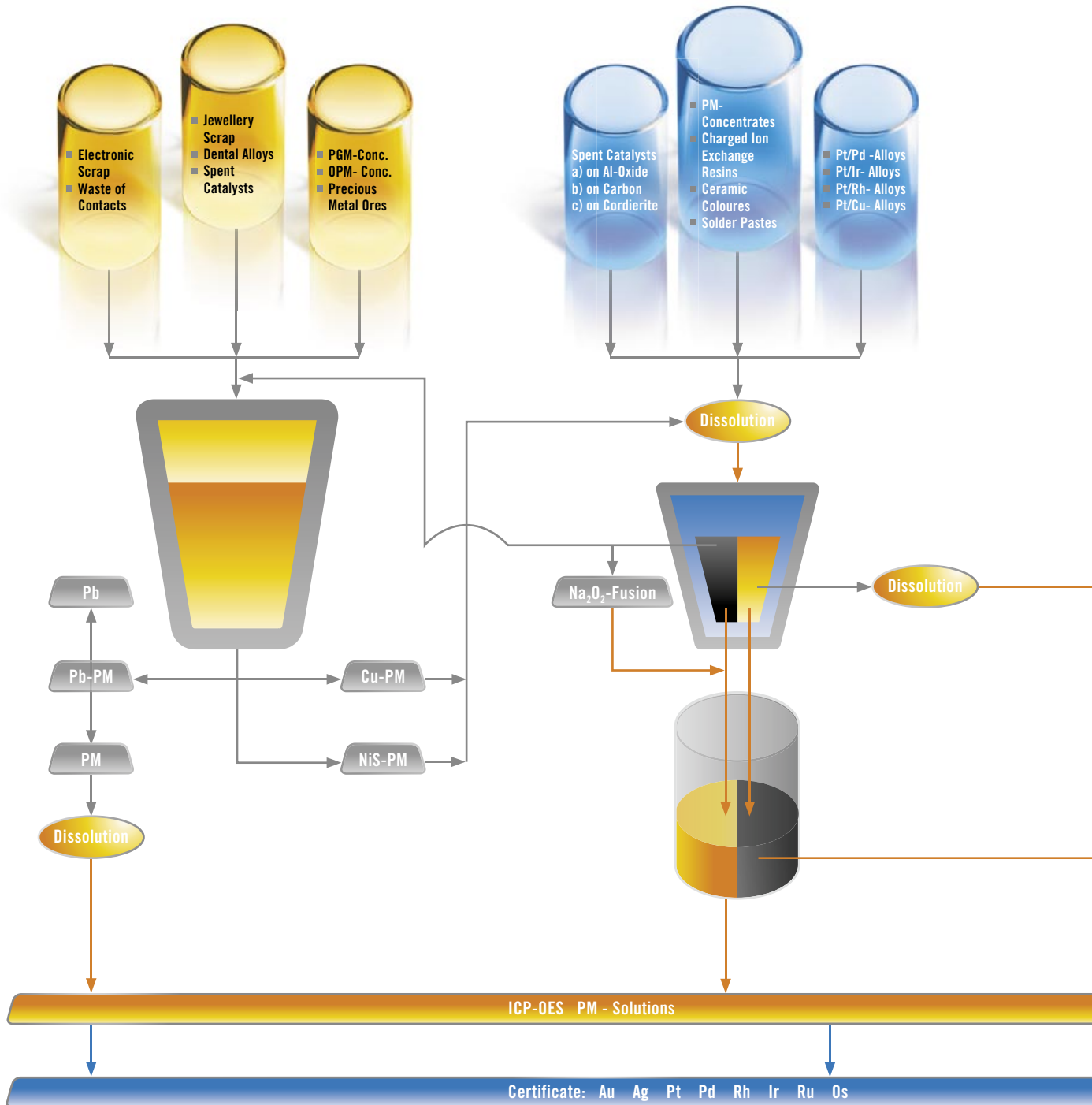
This very simple and very reproducible method is used primarily for the determination of the concentration of Ag. Also she is used for the determination of bases and acids in aqueous samples. The possibility to use this method also in non aqueous samples is applied in the analysis of pharmaceutical products.

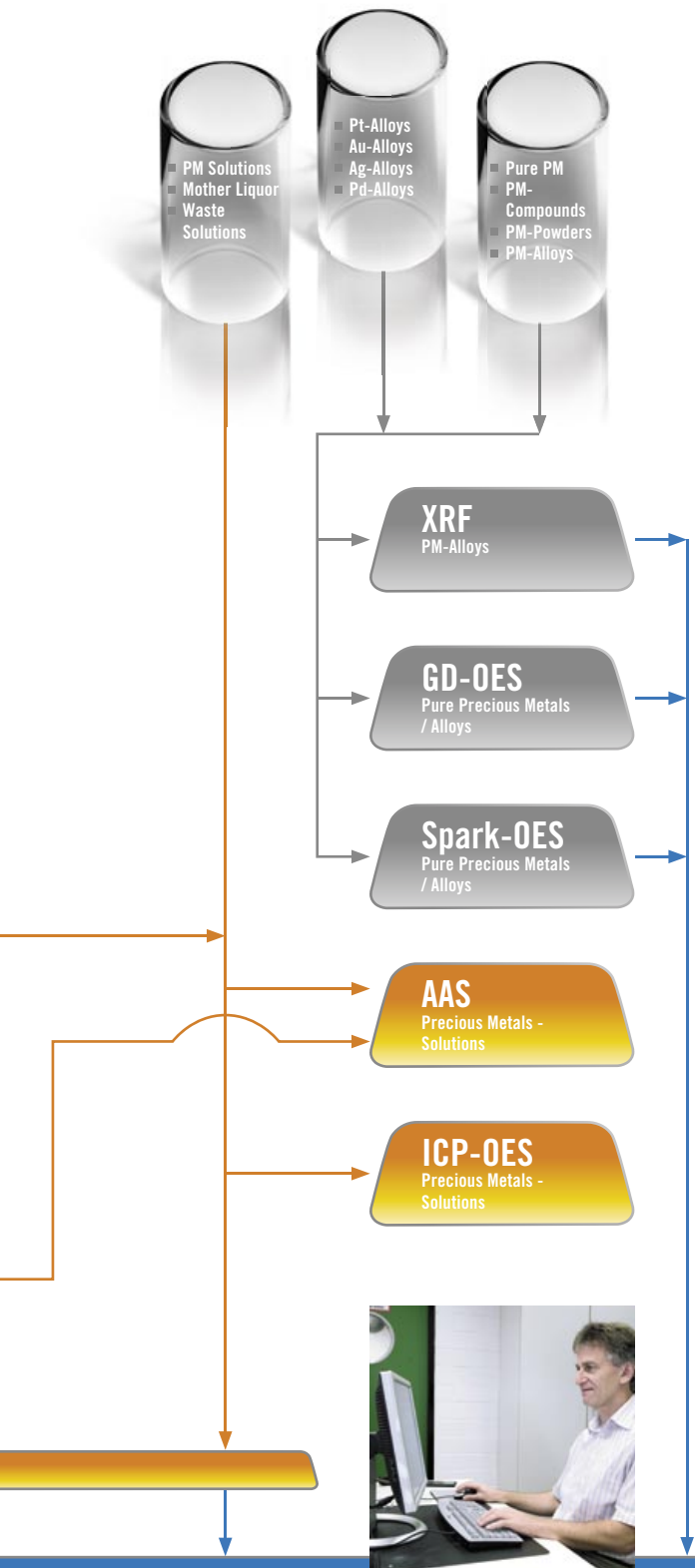
Coulometry

For the determination of the content of water in samples, the Karl-Fischer method is used, which is based on coulometric measurements. This analysis ist characterized by a very low detection limit (<100 ppm water)

Analysis of Precious Metals in the Central Analytical Laboratories

The way from the sample to the result
via the various analysis methods

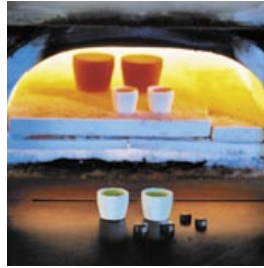




Sample divider



Charging the refining furnace



Refining furnace



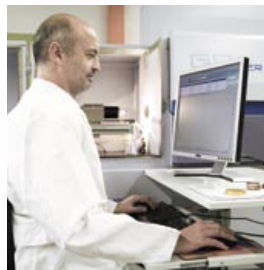
Sample preparation



XRF Spectrometer



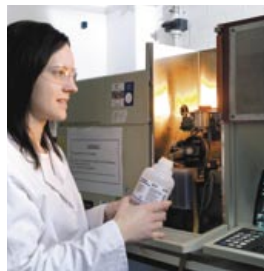
Sample preparation pressing



Glow discharge
OES Spektrometer



Spark OES Spectrometer



Atom absorption Spectrometer



ICP-OES Spectrometer

Specific Pharmaceutic Analytic General

Handling of Hazardous Substances

Trained staff members and adequate equipment allow the analysis of hazardous substances. For this purpose safety laminar flow cabinets in accordance with DIN are used that guarantee the safety of staff members. The fundamental goal is the protection of persons and products which is achieved by regular safety tests and periodically requalifying.

Long-term stability studies

Long term and accelerated stability studies are performed for the determination of the shelf life or retest date of pharmaceutical substances. Test samples are stored in qualified climatic cabinets. The stability of the sample is periodically analysed by validated tests. Temperature and humidity, as requested by the customer, are continuously monitored.

Release Analysis

Our QC lab is routinely testing API and final dosage forms according to customer and pharmacopoeial requirements. As a result, we will issue a GLP / GMP conforming certificate of analysis to our customers. Throughput times are strictly optimised for all procedures.

Special Analysis of the behaviour of cytostatics

The chemical behaviour as well as the decomposition of cytostatics are analysed under defined conditions. These tests are performed in close agreement with our customers and can be realized within short.

Qualification of reference standards

All reference standards needed for pharmaceutical testing are qualified in our labs. Among other parameters, we do check for identity, quality, purity and assay. The extend of analytical characterisation is defined by the intended purpose. Special examination (NMR, MS) are made with partner laboratories. All reference substances are stored under defined conditions and periodically requalified.



Handling of dangerous goods

Documentation in accordance with GLP

The analysis of pharmaceutical substances is done under the view of GLP. If needed, special methods are developed and validated. According to GLP all planning, realisation and conclusions are documented and archived.



Long-term stability studies



Gas Chromatography



Microbiological testing

Microbiological testing

We offer the complete service for sampling, analysing and evaluation of microbiological testing. We do routine determination of microbial load in air, water and APIs. Endotoxines testing is done as well. In close collaboration

with specialised laboratories sterility testing of final products samples are performed. As well, the identification of micro organisms, found in samples, is offered.

Method Description

Main Focus

Pharmaceutical Analytic

FTIR – Spectral Photometer

Our FTIR spectral photometer enables the analysis of solid or liquid samples. The identification of components is achieved through a comparison between the spectra recorded with spectra of pure substances from a data-base or from special handbooks. In addition the method is used for the characterisation of mixtures of substances. Beside the qualitative determination of substances it is also possible to make standardised quantitative determinations (e.g. asbestos; the determination of hydrocarbons in aqueous solutions, ...)

UV-VIS Spectral Photometer

This classical photometer is engaged in some special problems. Photometric measuring methods, especially developed for our purposes, ensure low detection limits.



FTIR Spectrometer



UV-VIS Spectrometer



High Pressure Liquid Chromatography

For example it serves for the determination of the concentration of coloured complexes in the visible range with the aid of previously generated calibration lines and calibration solutions.

High Pressure Liquid Chromatography – HPLC

Our HPLC instrument can be equipped with an UV- or conductivity detector. It satisfies many requirements.

One main application is the quantitative and qualitative determination of substances in mixtures. In addition it serves for purity checks of products and allows specification control.

Gas Chromatography with Flame Ionization Detector

The instrument in our laboratory works with a flame ionisation detector. This method serves for the determination



Gas Chromatography



Polarimeter

of organic impurities in vaporisable liquid or solid mixtures. With the help of GC-FID we identify residual solvents in solid products. Furthermore this technique is used for the quality control of organic raw materials as well as of products.

Polarimetry

The polarimeter is used for the determination of the optical activity of a sample, so that it will be possible

to determine the purity of an enantiomer. This is very important for our pharmaceutical agents.

Analytical Performances


Offered by the Analytical Department of Heraeus

The extremely highly-qualified staff and the state of the art equipment of the analytical laboratories ensure that your problems are solved in a minimum of time with a maximum of quality. Most instruments are equipped with an autosampler for increasing the throughput of samples.

Apart from general procedures such as pH- or redox measurements, that are also naturally realised in our laboratory, the main strengths of our laboratories are in the following areas:

1. **The Analysis of Precious Metals** (see page 4-5)
2. **The Preparation of Samples for all instrumental techniques** (e.g. splitting of samples, melting, decomposition procedures, solubilization, extraction etc.)
3. **Atomic Absorption Spectroscopy AAS**
4. **Optical Emission Spectrometry**
with the following possibilities for excitation:
 - Inductive Coupled Plasma Emission ICP
 - Glow Discharge GD
 - Spark
5. **X-ray spectrometry XRS**
 - Wavelength Dispersive X-ray Fluorescence WDX
 - Energy Dispersive X-ray Fluorescence EDX
 - X-ray Powder Diffraction XRD
6. **Infrared Spectroscopy IR**
7. **Photometry in Ultraviolet and Visible UV-VIS**
8. **Polarimetry**
9. **High Pressure Liquid Chromatography HPLC**
10. **Gas Chromatography GC**
11. **Ion Chromatography IC**
12. **Potentiometric Titration**
13. **Coulometry**
14. **Thermal Analysis TA**
 - Thermogravimetric Analysis TGA
 - Differential Scanning Calorimetry DDK
 - Thermomechanical Analysis TMA
15. **Studies of Long-time Stability**





Our operations are certified according to DIN ISO 9001. Special topics are analysed following GLP rules under strict control by the FDA.

With pleasure we look forward to responding to and serving queries from elsewhere.

**Heraeus
Chemicals Division**

Zentrale Analytik
Postfach 1553
63450 Hanau
Germany
www.wc-heraeus.com

Contact Persons

Ulrich Nordheim
Phone: +49 6181.35-5445
Fax: +49 6181.35-5916
e-mail: ulrich.nordheim@heraeus.com

Gerhard Schullerus
Phone: +49 6181.35-5488
Fax: +49 6181.35-5987
e-mail: paul-gerhard.schullerus@heraeus.com

Wolfgang Rollmann
Phone: +49 6181.35-9414
Fax: +49 6181.35-4632
e-mail: wolfgang.rollmann@heraeus.com

Responsible for the content: Gerhard Schullerus
Images: Wolfgang Hartmann
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Germany
W. C. Heraeus GmbH
Chemicals Division
Zentrale Analytik
Heraeusstr. 12-14
63450 Hanau
www.wc-heraeus.com