

# Research and Development Laboratory and Pilot Plant Facility



ThyssenKrupp Uhde



ThyssenKrupp

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The facility is located in Ennigerloh, about 70 km north-east of our head office in Dortmund and, together with our R&D department in Dortmund, it is certified to ISO 9001.



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## 1. Company profile

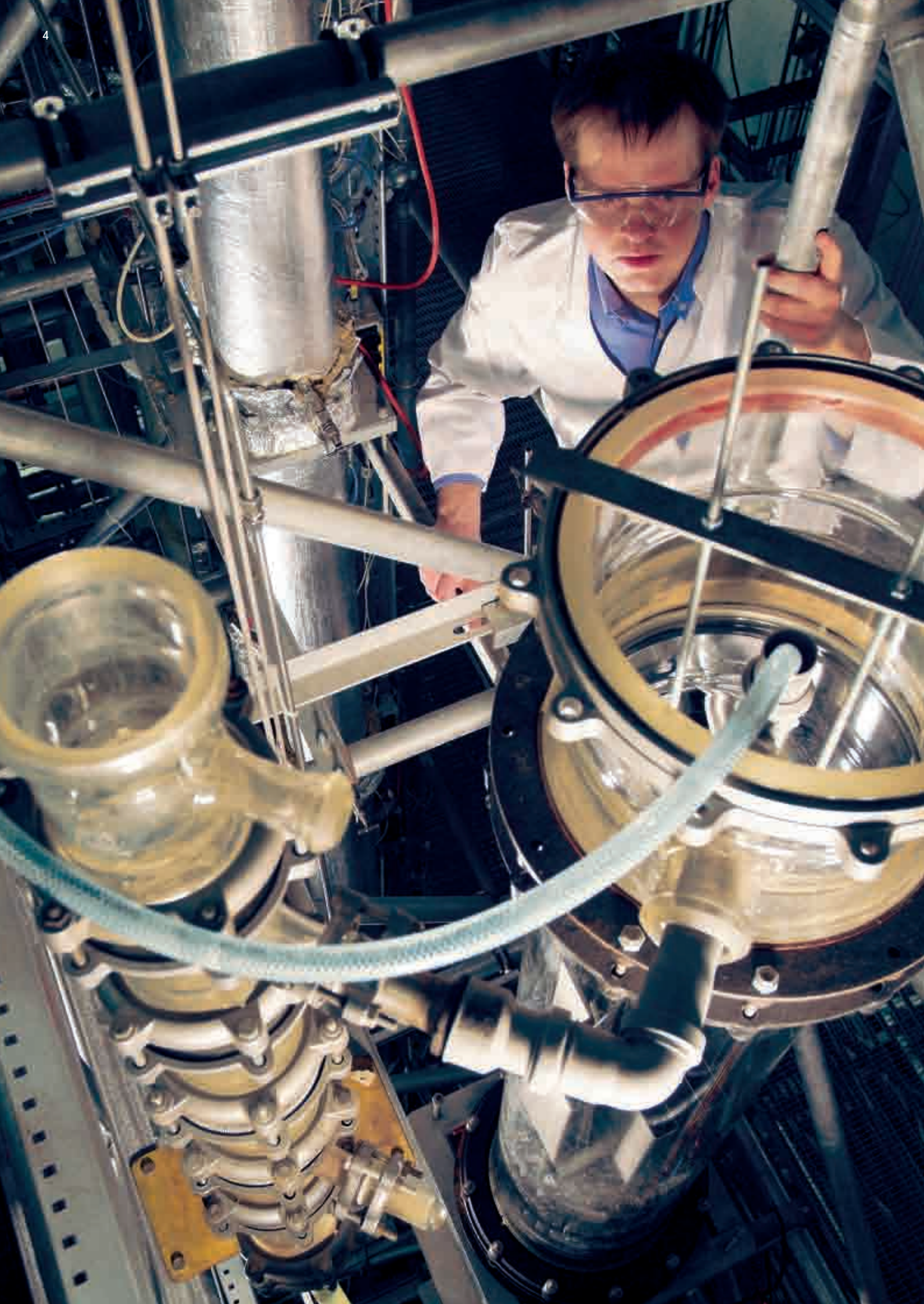
With its highly specialised workforce of more than 4,500 employees and its international network of subsidiaries and branch offices, Uhde, a Dortmund-based engineering contractor, has to date successfully completed over 2,000 projects throughout the world.

Uhde's international reputation has been built on the successful application of its motto **Engineering with ideas** to yield cost-effective high-tech solutions for its customers. The ever-increasing demands placed upon process and application technology in the fields of chemical processing, energy and environmental protection are met through a combination of specialist know-how, comprehensive service packages, top-quality engineering and impeccable punctuality.

These are underpinned by research and development work at our laboratory and pilot plant facility: a key factor in maintaining our state-of-the-art technology base.

Uhde's head office  
Dortmund, Germany





## 2. Employees and tasks

Highly skilled employees  
enable quick, in-depth process development



As a technology-driven engineering company, Uhde is committed to ensuring that its existing technologies are among the best. We are also constantly striving to expand our technology range, either by adding new technologies on a proprietary basis or through research and development in cooperation with partners.

A central R&D division is therefore essential if we are to guarantee that research and development is undertaken efficiently and professionally. At Uhde this division is staffed by around 45 employees, most of whom are graduate chemists or chemical engineers.

One department of our R&D division is our laboratory and pilot plant facility at Ennigerloh, Germany, which employs the following staff:

- 4 chemists
- 3 chemical engineers
- 9 chemical technicians
- 1 mechanic.

These highly skilled employees are involved in the investigation of new reaction or separation concepts as well as in the improvement of existing processes. This also includes the determination of physical properties

and the performance of extensive analytical evaluations. The laboratory staff assists, and is assisted by, the other departments of our central R&D division as well as our specialists in the technology divisions.

The specific tasks of the laboratory and pilot plant facility includes:

- design, erection and operation of pilot plants for process development and improvement
- testing and comparison of catalysts for proprietary processes
- performance of analytical evaluations on all types of samples (internal samples from our pilot plants or external samples from our customers)
- provision of support to our customers' laboratory staff
- compilation of analysis manuals and revision of the analytical procedures to be applied
- design, procurement and commissioning of working laboratories.

We provide all of the above services for in-house projects as well as for external customer projects.

## 3. Pilot plant facilities

### 3.1 Thermal separation



Piloting of an extractive distillation process

Since process simulation does not always supply definite results for the behaviour of trace components in thermal separation processes, experimental tests are imperative to validate product quality. We have a wide variety of distillation and extraction units available for performing such tests.

The Uhde pilot plant facility is a 12.6 m three-storey building with adjacent chemical laboratories.

The distillation columns in this facility are surrounded on all sides by scaffolding made of cube-shaped structural components which can be accessed every 2 m to allow fast, easy installation of equipment.

The following separation techniques can be carried out:

#### Conventional distillation

can be demonstrated in columns with up to 160 theoretical stages. Stainless steel modules with internal diameters of 100 mm, 72 mm and 40 mm respectively are available. The largest-diameter column is equipped with bubble-cap trays and the smaller ones with Sulzer EX/DX-type structured packings.

#### Extractive and azeotropic distillation

can be performed in columns with up to 70 theoretical stages. Stainless steel modules which have an internal diameter of 72 mm and are equipped with Sulzer DX-type structured packings are available.

**Divided-wall distillation**

with up to 40 theoretical stages in the partitioned section and up to 40 theoretical stages for both the section above and the section below the partitioned section.

**Liquid-liquid extraction**

can be carried out in a column with up to 50 theoretical stages. Glass modules which have an internal diameter of 40 mm and a height of 2 m are available. Each module is equipped with Sulzer BX-type structured packings.

**Gas washing**

can be done in glass columns with 40 mm internal diameter and column heights up to 12 m. Absorber and desorber columns are operated in parallel to ensure proper piloting of the selected process.

The operating pressure range for distillation and extractive distillation can be varied from vacuum up to 6 bar. The liquid-liquid extraction unit and the gas washing unit can be operated at a pressure of up to 1.5 bar.

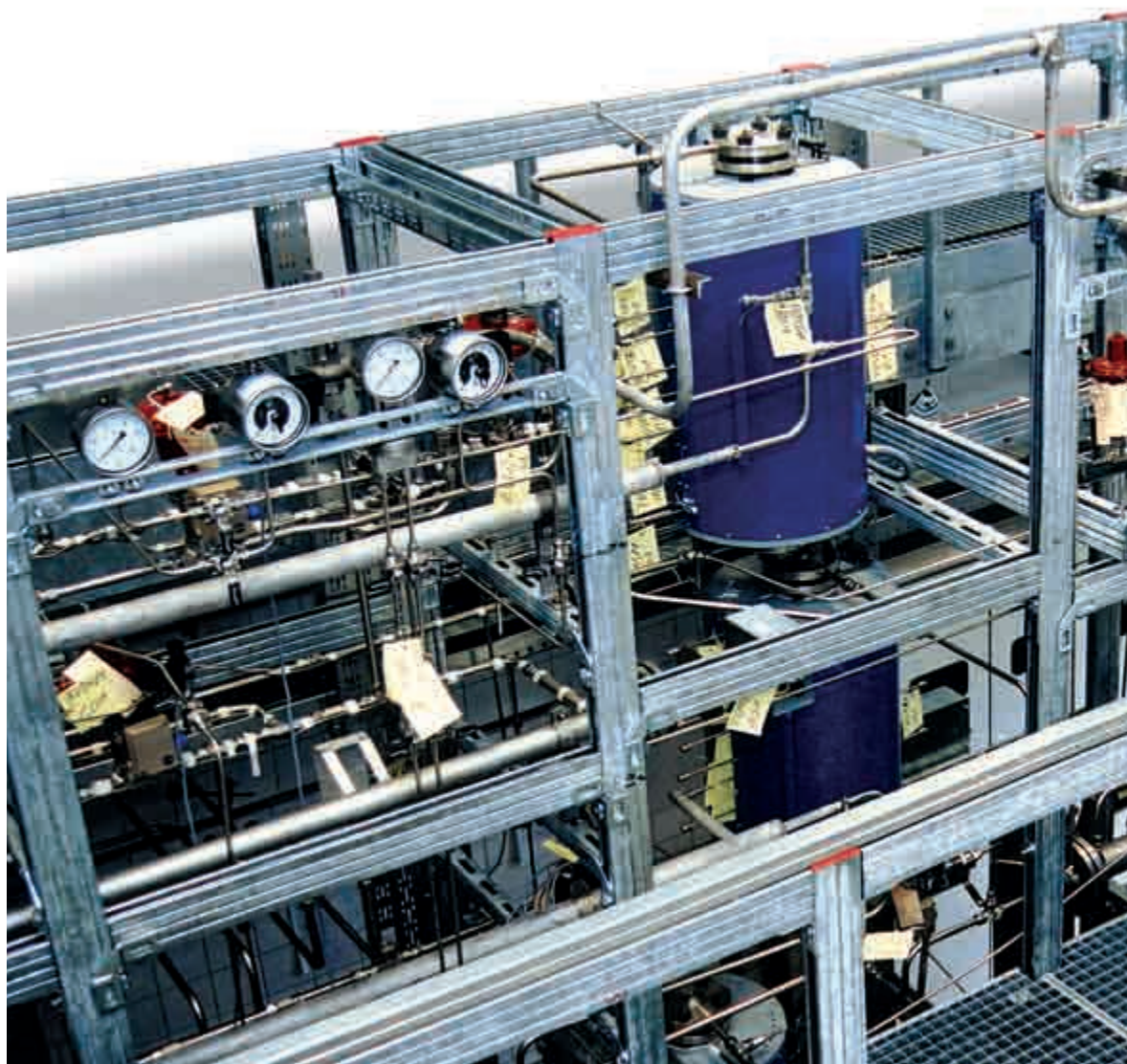
The plant, which is operated continuously in 3 shifts, enables all necessary recycle streams to be established. Extensive instrumentation allows a wide range of process data to be recorded and a modern DCS allows for precise control of the plant. A two-channel gas chromatograph with flame ionisation detectors is used for the on-line liquid analysis of products or intermediate streams.

## 3.2 Dehydrogenation processes

Medium- and long-term forecasts predict growing demand for on-purpose technologies for the production of light olefins (e.g. propylene, butylenes) and dienes (1,3-butadiene) through, for example, the dehydrogenation of light paraffins. Uhde's STAR process<sup>®</sup>, which involves mainly the catalytic reaction of light paraffins to the respective olefins and/or dienes, is one such technology. For continuous improvement of this process and the catalyst used, we have installed a pilot plant which enables us to test different catalyst formulations and new process concepts. The plant is equipped with a DCS and an emergency shutdown system which al-

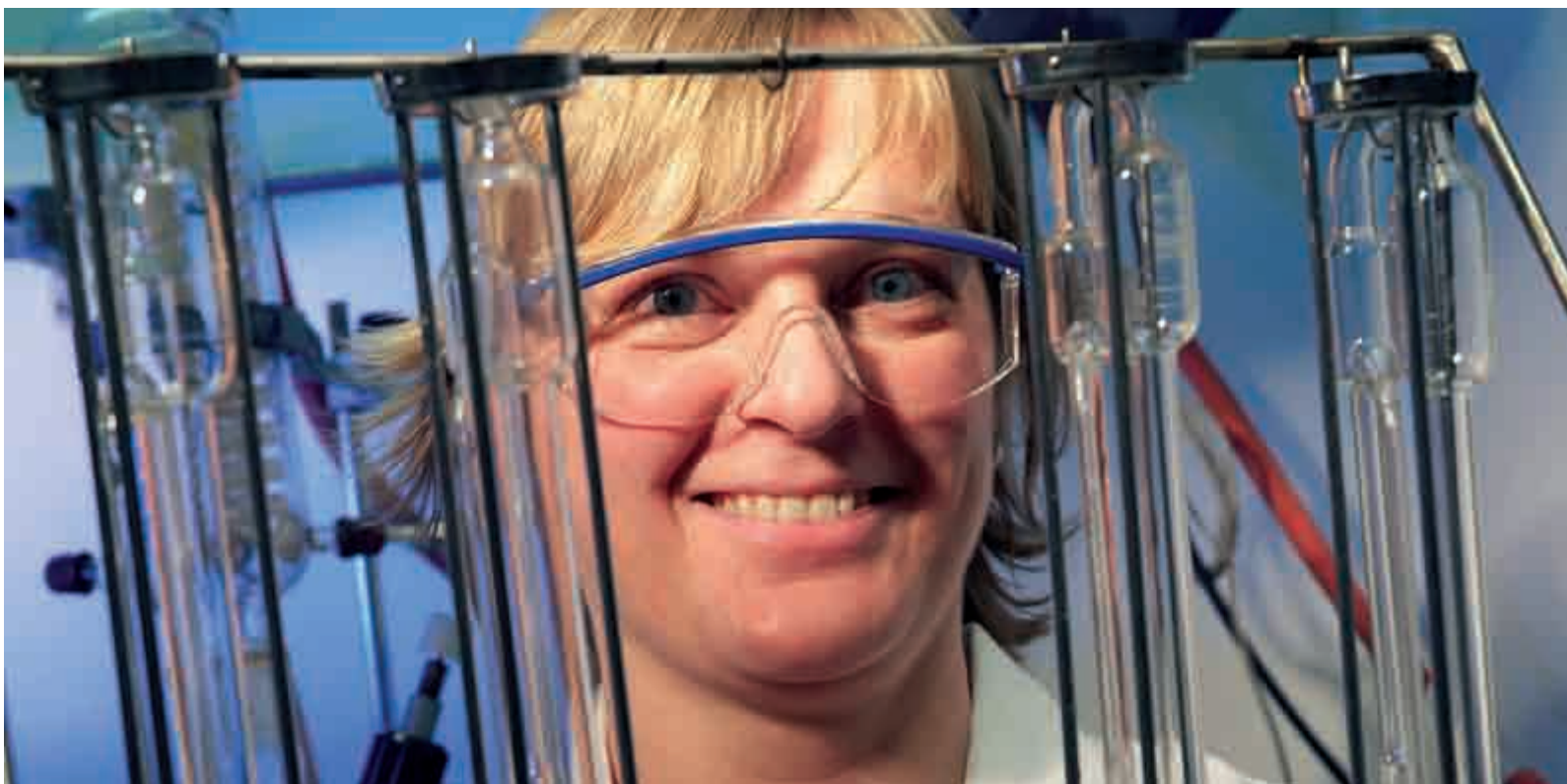
lows the plant to be run unattended 24/7. The paraffin and water feed rates are in the range of a few kilograms per hour; temperature profiles and pressures are set according to the commercial process. Feed, intermediates and product streams are continuously analysed by an on-line gas chromatograph. In addition to process control, the commercial DCS allows a matrix to be set for test runs with planned parameter variations.

The dimensions of key equipment are in a range which allows a reliable scale-up of the process as well as testing of the catalyst with its original shape and diameter.



A complex, automated pilot plant for the improvement of dehydrogenation processes and catalysts

## 3.3 Refining technologies



Through its refining competence Uhde offers proprietary technologies and know-how with respect to lube oil and wax production as well as hydrogenation and etherification processes. Various test facilities are available for determining process conditions regarding yield and product quality in lube oil processing or the treatment of middle distillates. These test facilities, which can be operated continuously or batchwise, comprise:

### Mixer-settler extractors

These extractors are used to demonstrate solvent extraction processes in lube oil refining. Tests can be carried out to determine yields and selectivities for the extraction, screen different solvents, generate accurate liquid-liquid equilibrium data and produce raffinate streams for further investigations.

### Vacuum filter units

These filter units are used in conjunction with scraped chillers to determine the yield and selectivity of deoiling/dewaxing processes by varying the solvent composition, dilution and temperature.

### High-pressure reactors

High-pressure reactors are available for testing hydrogenation processes, such as hydrodesulphurisation or hydrofinishing. These units can be operated batchwise or continuously in trickle-bed or submerged mode at pressures up to 200 bar and temperatures up to 550°C.

### Nanofiltration unit

This lab-scale unit with an approximate membrane area of 44 cm<sup>2</sup> can be used to screen different polymer membranes in deoiling/dewaxing processes. Additionally, fluxes and retention rates can be evaluated.

Refining technologies  
laboratory



Nanofiltration unit

## 4. Catalytic test units





We operate several catalyst test units, some of them are operated 24 hours per day. These test units allow us not only to compare the activity and product selectivity of different catalysts but also to make process developments and improvements.

The following catalyst test units are in operation:

#### **Catalyst test unit for ammonia synthesis**

This unit is for testing commercial and new catalysts for ammonia synthesis. The unit is able to operate under real process conditions, e.g. at temperatures up to 550°C and pressures up to 190 bar. It is also possible to mix ammonia into the hydrogen-nitrogen feed to check activity loss of the catalysts.

#### **Catalyst test unit for Uhde's EnviNOx<sup>®</sup>/DeN<sub>2</sub>O<sup>®</sup> processes**

This catalyst test unit for Uhde's proprietary process technologies for removing nitrogen oxides (NO<sub>x</sub> and N<sub>2</sub>O) from the off-gases of chemical plants is equipped with a multi-component gas-mixing station, analysers and an automation system for unattended 24 hour operation. The unit is used for process development and improvement, as well as for evaluating different catalysts and process schemes in order to optimise process conditions and catalyst arrangement for the benefit of our customers.

The test unit is also operated under real process conditions, not only with regard to temperature and pressure, but also with regard to feed gas composition (including moisture) and impurities, such as sulphur compounds.

#### **Catalyst test unit for refinery applications**

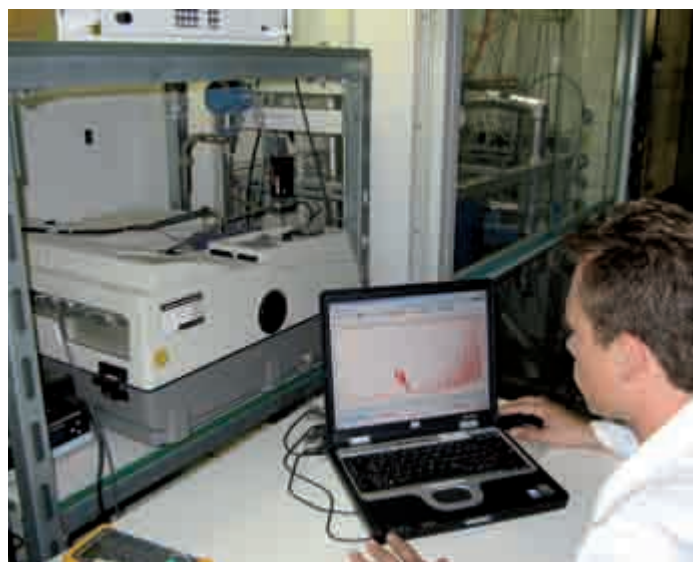
There is also a facility for testing the suitability of catalysts and feedstocks for hydrogenation processes in refineries. Hydrodesulphurisation (HDS), hydrodenitri-fication (HDN), hydrogen finishing and selective hydrogenation are the processes currently being tested.

#### **Catalyst test unit for dehydrogenation processes**

As Uhde is the owner of the STAR<sup>®</sup> process and the STAR catalyst<sup>®</sup> for the dehydrogenation of light paraffins to olefins or light olefins to light dienes continuous research efforts are made. For that purpose a special catalyst test facility is available which enables us to evaluate new process schemes and improved catalysts in very short time. Additionally, this test rig allows the measurement of the kinetics of specific reactions to derive kinetic constants for the calculation of chemical reaction rates. Once, a new reactor concept or an improved catalyst is found long-time tests are conducted in the pilot plant for dehydrogenation processes.



Test facility for ammonia catalysts



FT-IR spectroscopy for gas analysis

## 5. Chemical laboratories



Gas chromatography laboratory

Our chemical laboratories are equipped with sophisticated analytical instruments which enable us to analyse all pilot plant feedstocks, intermediates and products - whether solid, liquid or gaseous. Samples from customers and from on-stream commercial plants are also analysed in these laboratories.

### Gas chromatography

Gas chromatographs are used to analyse all types of hydrocarbon samples. Different models are available:

- Gas chromatographs with flame ionisation detectors (FIDs). These are equipped with different types of capillary columns having a wide spread of polarities. An alumina/sodium sulphate column is also available for LPG analysis.
- A gas chromatograph with mass selective detector (GC-MSD). This GC equipped with a mass selective detector (ion-trap principle) is able not only to identify unknown components in a complex matrix but also to quantify trace impurities in the  $\mu\text{g}/\text{kg}$  range.
- A gas chromatograph with nitrogen-selective detector (N-CLD). This instrument is able to detect and quantify nitrogen compounds in the range of several ppb.

- A gas chromatograph with a sulphur-selective detector (S-CLD). This instrument is able to detect and quantify sulphur compounds in the range of several ppb.
- A transportable gas chromatograph with thermal conductivity detector (TCD) for gases. This instrument can be used to analyse all permanent gases and is suitable for use at production sites, for example.

### Chemical laboratory

A variety of modern analysers are available, including:

- an ion chromatograph (IC)
- an atomic absorption spectrometer (AAS) with flame, graphite furnace and flameless atomisation
- an infrared spectrometer (FT-IR) with gas cell
- an infrared spectrometer (FT-IR) with ATR
- an UV-Vis spectrometer
- a total nitrogen analyser (TN) with a chemiluminescence detector. This analyser is able to analyse both water and hydrocarbon samples. The detection limit is about  $30\ \mu\text{g}/\text{kg}$ .



Ion chromatograph



High temperature viscosimeter



Atomic absorption spectrometer

- a total sulphur analyser (TS) with an ultra-violet detector. This analyser is able to analyse both water and hydrocarbon samples. The detection limit is about 200 µg/kg.
- a polarograph for traces in the µg/kg range.

#### Refining technologies laboratory

Our refining technologies laboratory is equipped with the analytical equipment required to conduct specific analyses relating to lube oil and wax processing, for example determination of the pour/cloud point, viscosity, needle penetration, oil content, colour (ASTM and Seybold) and distillation curves (atmospheric and vacuum).

#### Gasification laboratory

There is also a special laboratory for the analysis of solid feedstocks, intermediates and products of gasification processes. Since Uhde is able to offer both, fluidised-bed gasification (high-temperature Winkler gasification, HTW) and entrained-flow gasification (PRENFLO®) a variety of analytical methods is available. Solid feedstocks can be, for example, wood, biomasses, lignite, bituminous coal, pet coke and mixtures thereof. For the feedstocks common chemical and physical investigations like immediate and ultimate analyses, bulk,

compacted and true densities, size distribution, heating values and ash composition are done. Moreover, specific analytical investigations like fluidisation behaviour (for pneumatic transport of feedstock or fluidised-bed characteristics), ash fusion and slag viscosities are also performed.

In addition to standard analytical methods (like ASTM, ISO, DIN), we are able to develop analytical procedures to meet the special requirements of our customers or to support troubleshooting at our customers' plants. Additionally, we provide analytical equipment for on-site measurements, such as dust measurement or the measurement of gases from combustion units or chemical plants. For process development, devices for determining physical properties such as vapour-liquid equilibria, liquid-liquid equilibria, activity coefficients at infinite dilution, etc., are available. These devices enable quick screening of alternative process variations or enhancements; in addition, physical properties can be generated for process simulation.

A quality management system which applies to all of our laboratory and pilot plant facilities ensures the services offered are of a high quality.

## 6. Customer services

### 6.1 Piloting of customer processes

Besides being used for in-house R&D projects, the laboratory and pilot plant facility can also be used for the development of customer processes. New technologies developed in customers' laboratories can be tested on a pilot plant scale, which enables not only long-term continuous operation of the process but also the production of an end product for further examination. We have assisted customers, for example, in the following project developments:

- de-acidification of an olefin-rich stream using liquid-liquid extraction and azeotropic distillation
- purification of hydroxybenzenes using extractive distillation
- enrichment of a chlorinated hydrocarbon stream in a divided-wall column
- production of a hydrocarbon stream lean in aromatics via extractive distillation.

Our laboratory staff not only operates our pilot plants in Ennigerloh, but also pilot plants at customer sites. As well as piloting new processes, we also offer conceptual design, carried out either in our central R&D division or in one of our technical divisions.

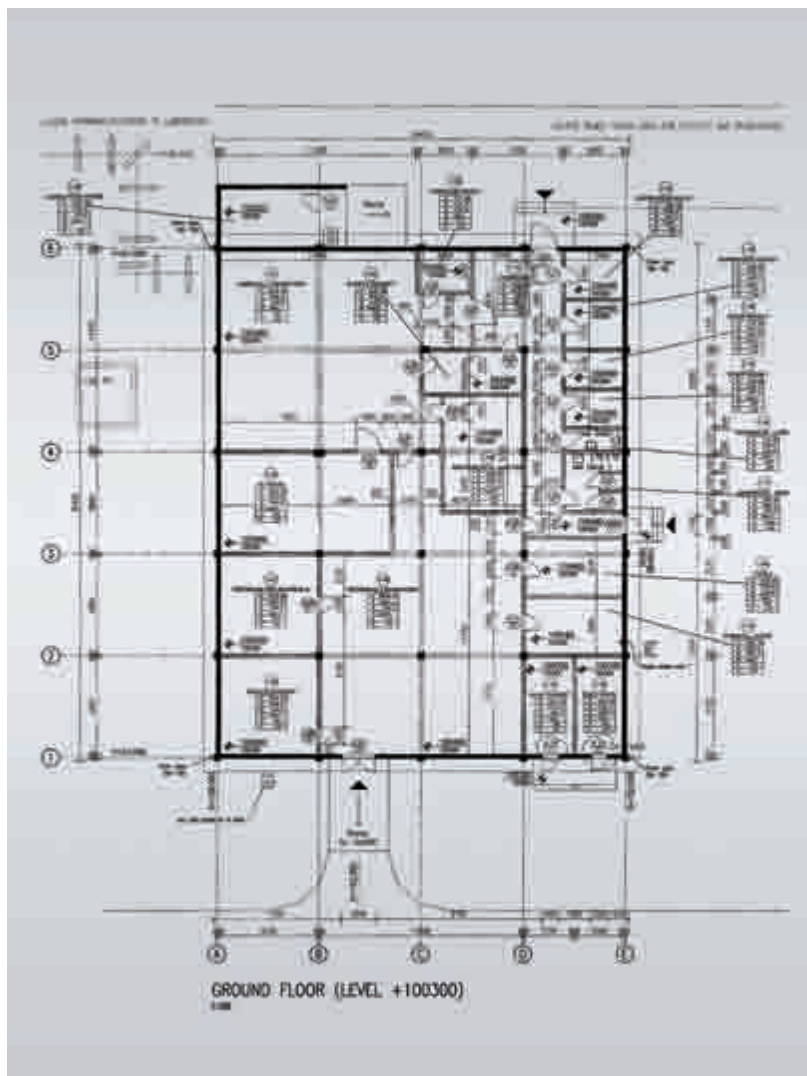


Laboratory after installation: gas chromatography



Laboratory after installation: ICP

## 6.2 Laboratory design



Plot plan of a working laboratory

In addition to our research and development activities, we design and commission working laboratories. This includes the development of plot plans, cost estimations and the specification and selection of furniture, chemicals and apparatus.

We also prepare analytical manuals for commercial plants and train customer employees.

Our recent reference list includes:

- Radici Chimica, Zeitz, Germany (nitric acid / adipic acid)
- Egyptian Fertilizer Co., Egypt (ammonia / urea)
- BCA Bitterfeld, Germany (chlorine electrolysis)
- Abu Qir Fertilizer Company, Egypt (ammonia / urea)
- Saudi Arabia Fertilizer Company (SAFCO IV), Saudi Arabia (lab extension)
- Egyptian Propylene & Polypropylene Company (EPPC), Egypt (lab for propylene, PP, off-sites and utilities)

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