



SIEMENS

Engineering & Consulting

Laboratory Services 2016

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Siemens Engineering and Consulting's "Laboratory World"

Siemens Engineering and Consulting's "Laboratory World" and its design and consulting offices are located in the Industrial Park Höchst, an industrial complex with more than 80 manufacturing plants for chemicals and pharmaceuticals.

We offer engineering and consulting services for almost every phase in the lifecycle of a chemical or pharmaceutical production plant. As a result of our long-standing experience and history we have gathered an excellent know how. This experience and our professional methods ensure the successful resolution of our customers projects, questions and tasks.

As not everything can be calculated or simulated with modern tools we are maintain a modern laboratory available. We offer physicochemical investigation for the safe handling of chemicals as well as for the optimization of process technology or redesign of your process or process step. This brochure gives you an overview about our lab services for process optimization.

If you have any questions, requests and suggestions please contact us at any time.



The laboratory world of Siemens Engineering & Consulting in brief:

The Laboratory World has 13 fume hoods, 7 walk-in fume hoods, 10 safety cabinets for handling of harmful dust and 4 autoclave chambers to perform experiments with high safety demand.

Furthermore the following features are at hand:

- Distillation area with space for four columns up to a height of 8 m each
- A large walk-in fume hood (5 m x 2 m) for miniplant operation and hybrid processes
- Modularly operated areas for drying, distillation, membrane technology, physical properties measurements, crystallization, chemical reaction engineering and micro reaction engineering
- Chemical analytics
- Safety engineering / Thermal analysis / dusts

Multi-functional area with autoclave chambers, scale-up / pilot plant area with explosion protected stirring test facility, safety cabinets for sample preparation and post processing .

Properties Data – The Basis for reliable Calculations and Simulations

A reliable simulation of any process equipment performance requires a profound and verified quality of the input parameters, i.e. the properties data and the selection of appropriate physical properties models.

We offer measurements of the parameters according to the customer's needs as well as the selection of appropriate physical properties models. The required data are determined according to state of the art methods.

Method/Equipment	Parameter
101 Ebulliometer	
Glass apparatus, dynamic method Temperature range: $20 < T/^{\circ}\text{C} < 240$ Pressure range: $3 < P/\text{hPa} < 1013$	Vapor pressures of pure substances
102 PTx-Apparatus in heating chamber	
Steel apparatus, static method Temperature range: $30 < T/^{\circ}\text{C} < 230$ Pressure range: $0.01 < P/\text{bar} < 26$	Vapor pressures of pure substances / vapor-liquid equilibrium (VLE)
103 VLE-Circulation apparatus	
Glass apparatus, dynamic method Temperature range: $20 < T/^{\circ}\text{C} < 240$ Pressure range: $10 < P/\text{hPa} < 1013$	VLE
104 GC Headspace	
Static method Temperature range: $30 < T/^{\circ}\text{C} < 180$ Pressure range: $5 < P/\text{hPa} < 2500$	VLE / activity coefficient measurement
105 Differential Ebulliometer	
Glass apparatus, dynamic method, for phase equilibria at high dilutions Temperature range: $20 < T/^{\circ}\text{C} < 240$ Pressure range: $3 < P/\text{hPa} < 1013$	Activity coefficients at infinite dilution
106 Gas Saturation Method	
Entrainment of components into the gas phase, pressureless operation Temperature range: $20 < T/^{\circ}\text{C} < 250$ Pressure range: $0.0001 < P/\text{hPa} < 10$	Vapor pressures of pure substances / VLE
107 Gas Saturation Apparatus	
Henry-coefficients, steel apparatus, static method Temperature range: $-30 < T/^{\circ}\text{C} < 200$ Pressure range: $0.01 < P/\text{bar} < 50$	Henry-coefficients, Gas-Liquid Equilibrium (GLE)

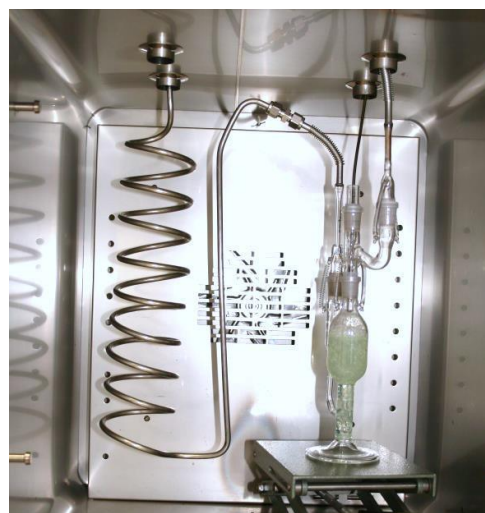
We have the expertise to advise you on which method(s) should be applied for your substances.

Methods for measurements of boiling points and melting points, enthalpy and other process safety parameters can be found in our Process Safety Brochure or under the following link:

www.siemens.com/EC and then > "Downloads"



or click or scan here



Equipment for the gas saturation method

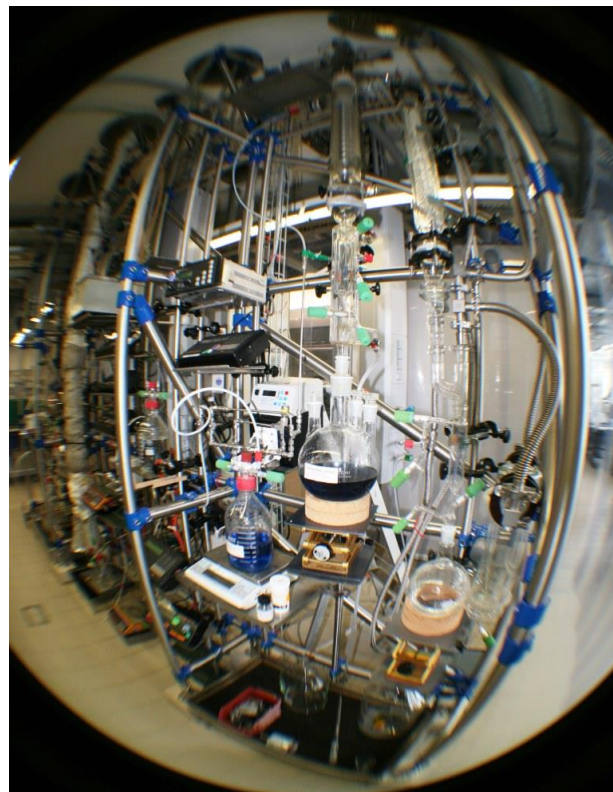
Thermal Separation Technology

We are your competent partner for all questions concerning thermal separation technologies. Our services include all important thermal separation operations such as distillation, absorption, adsorption and extraction as well as ion exchange and membrane technologies.

Our modern and state-of-the-art laboratory equipment allows us to perform scalable investigations which include data logging and can be used for the validation of simulated calculations and the description of the product behavior.

We offer you testing facilities for batch and continuous processes, for vacuum and pressure distillation, reactive distillation, extractive distillation and pressure swing distillation. The synthesis and analysis of sample quantities is also available upon request. Since we are independent of column or membrane manufacturers as well as equipment manufacturers, our recommendations incorporate the whole palette of equipment available on the market. As a result, we offer you the most effective and cost efficient solution for your specific separation problem.

Being situated in the Industrial Park Hoechst, we are well familiar with the handling of hazardous materials. We therefore possess the appropriate equipment for the handling and disposal of such materials.



201 Distillation	Equipment/Method
Laboratory distillation	(Silvered) glass column with bubble-cap trays or structured packing
Laboratory measurements for scale-up	Diameter: 50 mm, also 30 mm und 80 mm (upon request)
Individual, problem-specific setup, also with additional units (e.g. washers)	8 m height
Measurements relating to the fluid dynamics and mass transfer performance of new random packings and structured packings	Temperature range: $20 \leq T/^{\circ}\text{C} \leq 250$ Combination of several columns
202 Evaporation	
Proof of Principle trials	Rotary evaporator
Investigation of:	Thin film evaporator
<ul style="list-style-type: none"> • Deposits (fouling) • Heating surface • Product cracking 	Short path evaporator Falling film evaporator
203 Absorption	
Elaboration of the absorption and desorption process	(Silvered) glass column with bubble-cap trays or structured packing
Laboratory measurements for scale-up	Diameter: 50 mm, (30 mm , 80 mm)
Measurements relating to the fluid dynamics and mass transfer performance of new random packings and structured packings	Temperature range: $20 \leq T/^{\circ}\text{C} \leq 250$
204 Adsorption	
Proof of Principle trials	Fixed bed adsorber
Selection of the adsorbent	Shakers
Laboratory measurements for scale-up	

205 Extraction	
Exploratory trials as basis for solvent selection and recovery	Hollow fiber extraction module
Experimental determination of the extraction parameters	Karr column
Laboratory testing for the extraction process	Packed column
206 Ion Exchange	
Proof of Principle trials	Glass apparatus
Selection of the ion exchange material	
Laboratory measurements for scale-up	
207 Membrane Technology	
Proof of Principle trials for membrane screening and membrane selection	Cross-flow filtration: microfiltration, ultrafiltration, nanofiltration, reverse osmosis
Trials relating to the long-term stability of membranes	Pervaporation / vapor permeation
Process development with mobile laboratory and pilot plant equipment (also GMP)	Electrodialysis / dialysis
Measurements for scale-up	Gas separation with membranes
Provision of sample quantities	



Our mobile membrane module test stands can be installed temporarily at your site - we evaluate alternative membranes with realistic feed materials under manufacturing conditions.

Chemical Reaction Engineering

The chemical reactions are ruling the structure of a production process. E.g. low conversions lead to raw material separation and recycle. High selectivities lead to less effort in the subsequent product separation. This is the reason why improvements addressing the chemical reactor might have a significant impact on profitability although this is often just a small part of the process.

Our broad background fed from diverse customer projects show that efficient teamwork is the key for solving such challenging situations. We bring in our abilities in process design to understand the economics of a process. The close interaction with our customer keeps him involved in the multi-level decisions.

As the possible project tasks are very broad we address this by working in an iterative procedure. The planning is revised in short intervals to proof that the ultimate goal is still in focus. A typical initial structure of a project might look like this:

1. Theoretical evaluation
2. First evaluation trials
3. Review of the initial hypothesis
4. Detail evaluation of the kinetic system
5. Process design.

300 Chemical Reaction Engineering Services	Reactor Types
<ul style="list-style-type: none">• Proof of Principle trials• Experiments for the determination of kinetics• Investigations in micro-reactor systems• Fitting of kinetic parameters to experimental data• Modeling and simulation of measured kinetics (e.g. with Presto®/Predici®)• Integration of kinetics into the Aspen Engineering Suite®• Development of reactor concepts based on kinetic data	<ul style="list-style-type: none">• Batch or semi-batch operated agitated reactors with different stirring systems• Tube reactor with/without static mixers• Fixed-bed reactors• Micro-reactors• Gas-liquid-reactors (e.g. stirred cell reactor)• Gradientless or Loop reactors (Berty type)• Fluidized bed reactors• Trickle bed reactors



Catalysis

You are the owner of a process plant but unsure, if you are using the best-suited catalyst in order to achieve the maximum plant capacity?

We have the expertise to support you. Our independence of catalyst manufacturers and subcontractors allows us to supply an optimized process solution based on an objective analysis of your specific requirements.

Custom made set-ups could be assembled in our labs for the evaluation intended. Later transfer to the customer enables the long term tests within his supporting plant infrastructure.

Pilot Plant for one of our customer projects



Catalysis – Services	Reactor Types
401 Individual equipment setup for catalyst testing, characterization of gas phase reactions	Fixed-bed reactors (glass or steel) Diameter: 20 to 80 mm Temperature range: up to 1000 °C Pressure range: up to 300 bar
402 Trials relating to the long term stability of catalysts	Gradientless or loop reactor (Berty type)

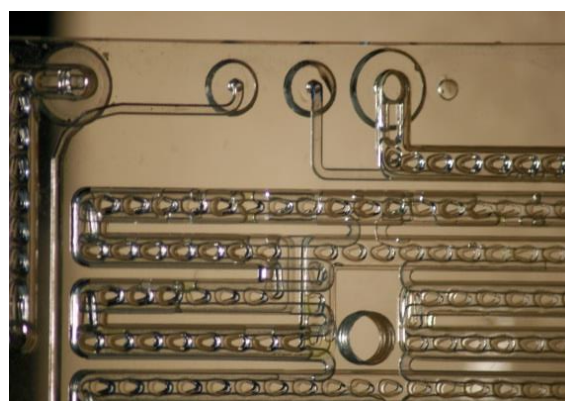
Microreaction Technology

Microreaction technology allows us (even under inert conditions) to analyze and optimize mixture-sensitive or highly exothermic reactions as well as challenging reactions in regard to process safety.

Examples are reactions in the field of metalorganic or high energy chemistry. Highly unstable intermediates benefit from a continuous operation. As an example, metalorganic intermediates can be converted at moderate temperatures of -10 °C to 0 °C. An extensive cooling of the system to dry ice temperature is often not necessary.

Our modularly designed microreaction technology equipment allows us to select and test a broad range of process parameters for you in a very short time period. This includes the manufactur-

ing of first sample quantities up to kilogram scale.



Microreaction Reactor

700 Micro Reaction Services	Equipment/Method
701 Microtechnology consulting (unstable intermediates, millisecond-reactions, high energy chemistry)	Our microreaction technology system <ul style="list-style-type: none"> • Pressure range: $1 \leq p/\text{bar} \leq 10$ • Temperature range: $-20 \leq T/^\circ\text{C} \leq 200$
702 Experimental feasibility studies in regard to microreaction technology	
703 Process optimization, batch to continuous	Individual setup of test equipment
704 Manufacturing of sample quantities	

Stirring Technology

Even though the basics of stirring are well-known for quite some time, the optimization potential during operation is often considerable.

Our laboratory equipment allows for the investigation of various stirring and mixing applications. In particular, trials with chemical reactions (even at higher pressures and temperatures and with corrosive, flammable or poisonous substances) can be performed in our laboratory world.

Based on the data gained in the experiments our experts can determine the scale-up criteria. These criteria will later be used in scale up models and calculations by our engineers.



The glass and stainless steel test stand for stirring installed in a hood.

Part of our stirrer collection

Stirring / Mixing Services	Reactor Types
400 Optimization trials for the selection of appropriate stirring systems in regard to critical process parameters, with and without chemical reaction and also under explosion-proof conditions.	Glass or steel vessel: 1 to 20 L Pressure: -1 to 65 bar Temperature range: - 50 to 300 °C
401 Design trials with respect to all basic operations in stirred vessels <ul style="list-style-type: none"> • Heat transfer • Suspending of solids • Aeration • Dispersing of liquids 	
402 Scale-up adequate trials in combination with scale-up models/modelling for technical reactors up to 100 m ³	
403 Supplier-independent selection of stirring systems	
404 Process supportive determination of the viscosity via torque measurement	
405 Supervision and analysis of process plant trials for selection/dimensioning of adequate stirrer systems	

Crystallization

High substance yields along with a high purity and low production costs: These are the major requirements for many chemical processes. In theory, crystallization is simple. Dissolution of the raw material during heating, slow cooling-down, process finished!

In practice there are many challenges. What about additional product requirements apart from purity? Is the modification as specified? How is the particle size distributed? Can the solids be

filtrated? Or does the product include a large amount of finest fractions, i.e. spicular or dendritically shaped crystals which technically impede a solid/liquid separation or the subsequent drying process? Is the nucleus formation impeded, leading to a time-consuming process? How well can the process be reproduced?

We can supply you with the answers to these questions.

500 Crystallization	Equipment/Method
501 Optimization of crystallization and precipitation processes in regard to <ul style="list-style-type: none"> • Yield • Purity • Particle size and form • Filterability and drying characteristics • Energy savings 	Crystallization in a standard stirred vessel (with or without guide tube) Cooling crystallization in solvents Evaporation crystallization Melt crystallization Volume: 0.1 to 20 L Temperature: -40 to 250 °C Pressure: up to 65 bar possible
502 Scale-up from laboratory to technical scale	Operating mode: batch or conti Special setups of test equipment upon request
503 Supervision of process plant trials	
504 Troubleshooting for existing processes	
505 Inline particle measurement in the laboratory or process plant via FBRM-sensor (e.g. for number of particles und size distribution)	
506 Inline process video microscopy in the laboratory or process plant via PVM-sensor (e.g. for particle form and polymorphy)	

Process Analytics

In most cases we use process analytics to verify the success of the use of alternative process parameters. We develop the analytical method best suited for your specific field of activity or apply

your analytical instructions/procedures for the analysis of our experimental trials.

Our laboratory provides the best possible conditions in order to support you in the field of process analytics.

Process Analytics Services
801 Gas Chromatography (GC) including Headspace
802 Liquid Chromatography (HPLC)
803 UV/VIS Absorption Spectroscopy
804 Near-Infrared Spectroscopy (NIR)
805 Process Infrared Spectroscopy
806 Raman Spectroscopy
807 FBRM (Focused Beam Reflectance Measurement) for Suspensions/Emulsions
809 Process Video Microscopy (PVM)
810 Titration, Zetasizer, Conductivity Measurement



Individual Process Issues are our Passion

Our state-of-the-art laboratory world constitutes the necessary infrastructure for the installation and operation of complete miniplants, even in autoclave chambers, in open laboratory spaces or in man-sized (walk-in) fume hoods with up to 10 m² area.

You benefit from our extensive experience, our interdisciplinary approach and our industrial environment in the Industrial park Höchst. We will gladly assist you on your way to an optimized process. Please find some examples of recent customer projects:

Which Catalyst is the best?

Examination of catalyst activity for the dehydrogenation of butane for use in various processes. Evaluation of the catalyst performance. Supplier-independent assessment of the results based on comparable data. Support during implementation of the results into the process plant scale.

Drying

The properties of dry products are highly influenced by the drying process. Often only a small amount of product is available for the process development in the fine chemicals and pharmaceutical industry. In such a case, a small equipment is beneficial for the testing of product properties during drying.

Process Optimization

Setup and operation of a DN 300 scale process plant for examining the kinetics during degassing of polymer suspensions with steam and the stirring behaviour (suspension, foam behaviour, power input). The trial results enabled the design and dimensioning of a degassing process for an increased plant capacity.



Customized Catalyst Test Rig

A catalyst test rig for the treatment of a highly corrosive organic acid was designed, set up and operated. The results led to quantitative understanding of the process step and secured the design of the overall process. After finalization of the test phase the rig was transported to the customer for further and long-term tests within the infrastructure of the production plant.



Extreme Conditions

Design, installation and operation of a special laboratory scale setup for high pressure polymerisation trials at up to 3000 bar and 180 °C operating temperature.

Reclaimer

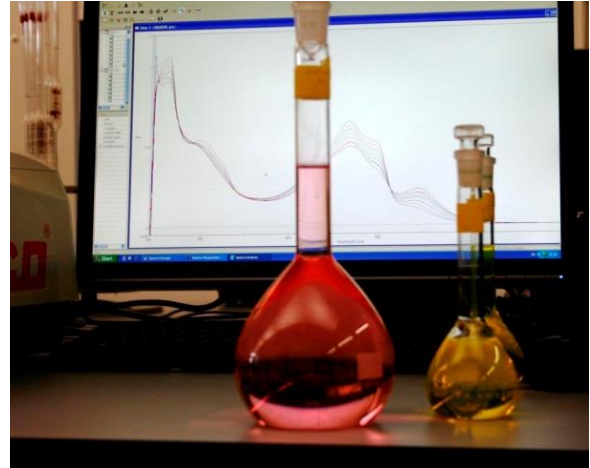
Washing agents often require a regeneration process. The organic washing agent of an absorption plant must be partially discharged and cleaned after the desorption process. Design and testing of the process on a laboratory scale pilot plant.

Batch to Conti Transfer

A well known batch process for the production of a performance additive was transferred into a semi-continuous process. The involved reaction steps were evaluated in lab scale and transferred into an economically optimized process design. A process design package with all the data necessary to start a basic engineering was provided on the newly designed process.

Concept for kinetic Evaluation

A concept for the evaluation of the reaction kinetics of a new production process for a "green" chemical was set up in close cooperation with the customer. The data should be used for the simulation of the overall production process.



900 Individualized services

901 Individually set up miniplants in laboratory scale, also under explosive conditions

902 Operation of miniplants

903 Process optimization, manufacturing of sample quantities

904 Start-up of the miniplants at your location

We are prepared to assist you in the examination of your process-specific issues with the deep know-how of our experts, our laboratory world and the infrastructure of the Industrial Park Hoechst.

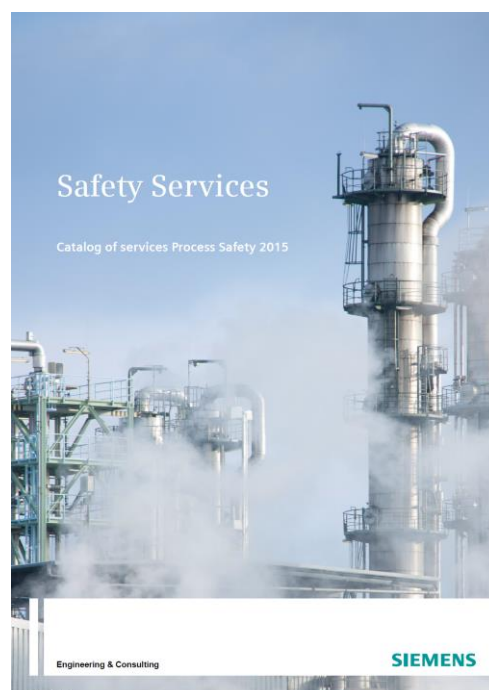
Process Safety

The sensitive subject of process safety can be controlled only by comprehensive consideration and cooperation between the necessary professional disciplines. The right combination of consulting, dimensioning and **lab investigations** ensures a safe and economical plant operation with a high level of processes control.

Our services for process safety are summarized in an own brochure which can be downloaded from our website: www.siemens.com/EC



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