Introduction

Since the company’s founding in the year 1897, Lurgi has actively supported research and development. The establishment of the company’s own R&D center in 1937 bears evidence to the importance attached to the development of proprietary technologies as an essential element of the international plant engineering and contracting business. Located to the East of Frankfurt, on premises covering more than 50,000 square meters, this is where Lurgi pooled its research facilities. The necessary test equipment was installed, laboratories and pilot facilities were erected and a comprehensive infrastructure set up. Today, many services – at the workshop level for example – are no longer rendered by own Lurgi staff but are outsourced. This arrangement permits the company to focus on its research activities.

In July 2007, a strong partnership also in the field of R&D led to the acquisition of Lurgi by Air Liquide. Air Liquide operates its own R&D centers around the world, e.g. in France, Japan, USA, etc. They command an excellent knowledge of the respective fields of expertise. The shared target is to develop energy-efficient processes, invent clean processes by limiting nitrogen oxide and CO₂ emissions, and develop new energy solutions such as hydrogen, gasification, biomass, clean coal, etc. for a sustainable environment.

The Lurgi R&D Center now forms part of the worldwide Engineering & Construction operations of Air Liquide.

How is “Research and Development” defined at Lurgi?

For Lurgi, Research and Development means the definition and implementation of R&D projects, which are derived from the corporate objectives through a holistic strategic approach. The R&D tasks are chiefly driven by the market.

Consequently, R&D projects have the following objectives:

- Develop new products/processes that are technically and commercially mature just in time (applied research)
- Preserve/boost the market attractiveness of existing products/processes in a continuous improvement process (development)
- Support the marketing efforts for existing products/processes by specific testing and analysis of feedstocks, intermediates, end products, catalysts, etc. (technical improvement and service)

These clearly defined tasks determine the structure and equipment of the research laboratory that will be presented below. The core activities of Lurgi have been focused on 50 product groups from the areas petrochemicals, gas production and treatment, gas-to-chemicals technologies as well as renewable resources. As a result, virtually all ongoing R&D projects handled by the research laboratory derive from these business segments. Experimental work at the research laboratory is supported by the development activities of the associated process and engineering disciplines from the relevant technology group. An important element of Lurgi strategy is to execute R&D projects together with partners and customers, or to commission universities or institutes with such tasks. This strategy is designed to allocate competences and resources appropriately and timely to the corresponding research and development targets.
Structure and Organization

Providing R&D services at an optimized cost/benefit ratio is a challenge for every company. To this end it is essential not only to make available the necessary resources in terms of labor, equipment and budget but also to implement an efficient organization and structure. R&D at Lurgi is assigned to the technology business segment and, hence, subordinated to the Board of Management member responsible for technology. By meshing R&D with process engineering, experimental work at lab scale is directly tested against the planned commercial scale implementation. This translates into a shortening of development times and avoids technical as well as commercial risks early on. A discipline-specific university training as a chemist, physicist or process engineer is therefore the best starting point for a career in R&D. The highly qualified staff of our research laboratory ensures competent assistance to the business segments of Lurgi.

Process development always implies an efficient analytical laboratory, which will be presented below. It is also essential to organize round-the-clock shift operation to feed the test equipment as required and monitor operational safety and reliability.

Current R&D Projects

Following, some typical R&D projects are presented for the business segments in order to illustrate the diversity of subjects handled, and provide at the same time an overview of the technical capabilities of the research laboratory.
Test units are available at the Lurgi R&D center to test the high-pressure and high-temperature reforming of gas and naphtha. These units are used to support catalyst developments and to test new process conditions arising from new gas production concepts for optimum heat integration. A demonstration unit for high-pressure partial oxidation processes (HP-POX Plant) was built at the Institute of Energy Process Technology and Chemical Engineering of the Technical University and Mining Academy of Freiberg. This joint research project is backed by the German Ministry of Economics and Technology (BMWi) and by the Saxon Ministry of State for Science and Art (SMWK).

This pilot unit is designed as multi-process test facility. Tests have been performed for catalytic autothermal reforming (ATR) and non-catalytic partial oxidation of natural gas (Gas-POX) as well as gasification of liquid feedstock (MPG). The maximum operating pressure is 100 bar and the throughput is 500 kg/h of liquid feedstock and 500 m³/h for natural gas, respectively.

Gas Generation and Purification

Lurgi Syngas Technologies

The efficient production of synthesis gas is the key process for an economical conversion of various inexpensive feedstocks to valuable products such as transport fuels, methanol and ammonia. Large syngas capacities are required to economically convert natural gas in remote locations to transportable products on the Gas to Liquids (GTL) and Gas to Chemicals (GTC) routes. Synthesis gas as well as hydrogen and carbon monoxide are primarily obtained by reforming natural gas. In addition, the high-temperature gasification of inexpensive refinery residues is economical.

The processes available for this purpose are
- Steam reforming (Lurgi reformer)
- Autothermal reforming (catalytic partial oxidation)
- Non-catalytic partial oxidation (Gas)
- Multi Purpose Gasification (MPG) (partial oxidation of liquid feedstock)

The processes, which have been established for decades, are continually developed further, prompted by progress achieved in catalysts, construction material and the need for larger single train capacities.

Gas Generation and Purification
Gas Purification
Lurgi commands the full range of gas purification technologies, partly as proprietary technologies and with outstanding know-how for open-art technologies like amine treating. The design basis of these technologies was developed at the R&D center in the pilot plants available and by determining solvent properties with specially designed laboratory equipment.

The gas purification test unit is designed as multi-process test facility. Removal of hydrogen sulfide, carbon dioxide and other typical trace components has been tested extensively with physical as well as chemical solvents. The pilot plant comprises several columns designed for pressures up to 100 bar. When employing a wide range of structured and random packings as well as trays, gas loads of 12,000 and solvent loads of up to 20 l/h per hour can be applied.

The different columns and the process equipment can be connected according to the process requirements and parameters to be tested. Operation of the plant is fully automatic. A state-of-the-art DCS and safety system is employed for process control and data acquisition. The pilot plant is used to determine the selectivity and capacity of various solvents as well as their interaction with trace components and construction materials. Measurement campaigns in the commercial-scale plants of Lurgi’s clients are performed. They complement the existing database and improve the scale-up procedure. A specialized laboratory is operated for the quantitative analysis of sulfur components as well as trace components prevailing typically in raw gases from the gasification of oil, coal and natural gas.

Sulfur Management
Refineries and gas producers have to cope with increasingly more stringent environmental protection and emission control regulations. Lurgi’s well-known sulfur recovery technologies, such as OxyClaus®, Sulfreen®, Lurgi Tail Gas Treating and Omnisulf®, are continually optimized at the R&D center.

In this field, Lurgi cooperates with research partners like the University of Strasbourg and with industrial partners, e.g. the oil and gas producer Total Fina Elf and the catalyst supplier Süd-Chemie. The resulting combination of the most current research results, experience in catalyst development and production as well as in-depth knowledge of the process and our customers’ needs enables Lurgi to offer optimized process solutions to reduce industrial emissions and meet or exceed the environmental regulations.
Gas-to-Chemicals

MTP® – Methanol to Propylene
During initial pilot plant tests at the Lurgi R&D Center, the catalyst performance was investigated and the process conditions were optimized for maximum propylene yield. After the results showed the high potential of the MTP® process, a skid-mounted demonstration unit was built during the summer of 2001. This demonstration unit was then transported on a truck to the Statoil methanol plant in Tjeldbergodden (Norway), where it was operated between November 2001 and April 2004. The main target was to determine the service life of the catalyst under realistic conditions by continuously taking in the methanol feed from the final purification column of the Statoil plant. The results from the demo unit tests proved that the catalyst life exceeds one year of operation. The results also showed that the catalyst can be easily regenerated more than a dozen times.1

The data from the demonstration unit also provided the basis for the reactor model, product yields and stream compositions to be used for the design of the commercial MTP® process. In this respect it was extremely important that the relevant process conditions were identical in the demo unit and in the commercial-scale unit, so that sound data for the scale-up process were obtained. Hence, the adiabatic fixed-bed reactors in the demo unit and the commercial-scale unit have identical catalyst bed heights, identical pressure drops, identical gas velocities, identical temperature profiles and identical residence times. So, while the scale-up to the commercial plant is impressive and amounts to several thousands, the risk of this scale-up was minimal.

Another important milestone of the process development was the confirmation that the propylene product meets all required specifications for polymer-grade product. Samples of MTP® propylene were therefore sent to the polymer laboratory of Borealis, one of the world’s leading plastics producers, where they were successfully polymerized to obtain polypropylene. The result was the world’s first plastic cup made from natural gas instead of petroleum.

All together, the MTP® process made a considerable impact in the world market. Two large commercial plants are currently in the engineering and construction phase: both are located in China, have a capacity of more than 460,000 mta and use coal as feedstock for the production of methanol, then propylene, and finally polypropylene. With the realization of these MTP® projects in China, the crucial step towards a commercially attractive, large-scale methanol-based hydrocarbon synthesis was successfully achieved.
MtSynfuels® – Methanol to Synthetic Fuels

The rising demand for diesel and gasoline and the declining oil reserves have triggered worldwide efforts to curb the dependence on crude oil as the sole energy source for transportation. One of the promising alternatives developed is the new MtSynfuels® process. Based on Lurgi’s market leadership in both, methanol synthesis (MegaMethanol®) and methanol conversion to olefins (MTP®), and on Lurgi’s experience with the conversion of olefins to diesel, the MtSynfuels process integrates these technologies for the production of synthetic fuels from synthesis gas. Due to Lurgi’s extensive experience with gasification processes, this process route enables the production of fuels from a wide range of feedstocks such as natural gas, coal, oil sands or biomass.

To demonstrate the complete process chain from methanol to diesel, a pilot plant was erected in Lurgi’s R&D Center comprising

- the conversion of methanol to olefins,
- the oligomerization of the olefins,
- the separation of the resulting product into recycle streams, product gasoline and high-boiling distillate, followed by
- the hydrogenation of the distillate to diesel.

The pilot plant produced the first diesel sample eight months after the design work started. The quality of the diesel product was very promising from the beginning. Most of the relevant specifications were met or even exceeded. While the lower density is typical for all modern GTL-based fuels, there is still potential to improve the cetane number of MtSynfuels diesel.

Currently, Lurgi is cooperating with car manufacturers to test the MtSynfuels® diesel in cars to determine its ignition, combustion and exhaust properties. Preliminary results already indicate that the MtSynfuels® diesel has unique advantages over conventional diesel qualities.

The next step in the commercialization of the process will be the construction of a larger scale demonstration plant in cooperation with industrial partners to further optimize the process, improve the product quality and support fleet testing of the new fuel.
Petrochemistry

Butanediol (BDO): the GEMINOX Process
The GEMINOX process for the production of 1,4-butanediol (BDO) was jointly developed by Lurgi and BP Chemicals. In this process first butane is oxidized in a fluidized-bed reactor to maleic acid, which is then hydrogenated to BDO. For this second process step, BP was attracted by Lurgi’s expertise in the area of high pressure (>275 bar) hydrogenation of organic acids in trickle-bed reactors. In 1994, a pilot plant was built at the Lurgi R&D Center, the process was optimized and a pilot plant was built.

Since then many tests with industrial feeds containing all byproducts of the butane oxidation have been carried out. The reaction conditions were optimized and the expected lifetime of the most suitable catalyst was determined. Furthermore, the complete purification sequence was developed and demonstrated using various lab columns. These data were crucial for the design of the commercial plant since even the best process simulation software available on the market cannot predict the distillation behavior of organic trace components, which needed to be removed down to ppm level in the purified BDO product. Moreover, material-specific examinations were performed to determine the corrosion resistance of various materials to the highly aggressive organic acids.

Dimethyl Ether (DME)
Carrying two methyl groups as organic substituents, dimethyl ether (DME) is the simplest ether. It can be produced by an acid-catalyzed condensation of two moles of methanol under release of one mole of water:

\[ 2 \text{CH}_3\text{OH} \rightarrow \text{CH}_3\text{OCH}_3 + \text{H}_2\text{O} \]

Its direct production starting from syngas via a selective catalytic process is also possible and has been commercially realized. Under ambient conditions, DME is a colorless, non-toxic, highly flammable gas with slightly narcotic properties. DME is water soluble and easily liquefied under pressure, thus being an interesting alternative to conventional LPG. DME is a polar agent and as such shows excellent solvent properties in its liquid form. Purified DME finds application as a propellant, e.g. in hair sprays. Due to its high cetane number of 55 to 60, dimethyl ether can be used as a diesel motor fuel. For this use, only slight modifications of the conventional diesel motor are required which mostly concern the injection pump. The motor combustion of DME proceeds in a very clean way without soot formation. At the Lurgi R&D Center, several process variants for commercial DME production are studied in detail. The gas phase process, in which vaporized methanol is catalytically converted over \( \alpha \)-alumina at temperatures between 250 and 300 °C, is a commercially proven process, exhibiting low operating and investment costs. It is also part of the MTP® process where methanol as the starting material is first converted to DME before entering the MTP® reactor itself. Main current R&D activities relate to the investigation (both by simulation and by experimental work) of possible simplifications of a stand-alone DME plant.
Finally, in 1998, BP decided to build the first commercial GEMINOX plant and Lurgi’s engineers designed the plant using the pilot plant data with an impressive scale-up factor of ~13,000. The plant was built in Lima (USA) and has been in operation since June 2000. Product quality was excellent from the beginning.

In early 2005, during the restructuring of BP, the BDO technology and the Lima plant were sold to the US company ISP (International Speciality Products), a consumer of BDO, who plans to further develop the process and also license it to third parties.

Renewable Resources

Biodiesel from Vegetable Oils
The generation of biodiesel from vegetable edible oils is based on a technology involving the transesterification of triglycerides (oils) with methanol to methyl ester and glycerine, which was originally developed for the production of fatty alcohol. The pilot plant for the continuous production of fatty acid methyl esters was commissioned in the early 1990s and the process has since been optimized in terms of chemicals consumption, product quality and yield, and patented. Based on the results of the pilot plant, the first industrial-scale plant with a capacity of 250 tons per day methyl ester for fatty alcohol synthesis was smoothly started up in Batamas Megah in 1994.

The Kyoto Protocol as well as the EU Biofuels Directive (blending of petrochemical diesel with biodiesel) have drastically changed the market situation. Instead of small production units with batch operation, which so far characterized the biodiesel production market, the customers were now demanding continuously operating industrial-scale plants. Due to the design of the process the scale-up was easy for Lurgi. The change of the DIN standard from RME (rapeseed methyl ester) to FAME (fatty acid methyl ester) now also allowed the use of other natural oils as a feedstock for biodiesel production.
This “biomass” could be an important supplement to oil and gas in the transportation fuel business. The European target of replacing 20% of conventional fuels with such alternative fuels (1st and 2nd generations) by 2020 is ambitious.

Lurgi GmbH and Forschungszentrum Karlsruhe GmbH (FZK) are cooperating on a development program (Bioliq®) to demonstrate the complete process from the clod to the petrol pump at the Karlsruhe Research Center with financial support from the Federal Ministry for Food, Agriculture and Consumer Protection.

With the multi-step Bioliq® concept high-grade synthetic biofuels and important base products for the chemical industry can be produced. The bioliq® process is suited for biomass, which is found distributed over large areas and mostly exhibits a low energy content. In a first, decentralized step, the biomass is converted to a transportable, liquid intermediate product with a high energy density (BioSyn-Crude) in a so-called fast pyrolysis process and can then be economically transported over longer distances to centralized plants for the production of synthesis gas and fuels. Or it could be used as feedstock for refining or power plants.

The joint test operations of Lurgi GmbH and FZK are yielding valuable, sustainable results allowing conclusions to be drawn on the viability of the new technology for application in commercial-scale plants in the near future.
Fatty Alcohol Technology
Fatty alcohols on the basis of renewable raw materials have become increasingly important in the detergents industry as a base material for the production of surfactants. Given their biodegradability, they are preferred over surfactants produced from petrochemical raw materials.

1st Generation
In the early years with the typical small production volumes, the Lurgi slurry batch process for the fatty alcohol synthesis on the basis of fatty acids from a fat-splitting process was widely sufficient.

In view of a rising demand for fatty alcohol, however, a new, continuous technology for the synthesis of fatty alcohols had to be found. Given the sensitivity of the fixed bed catalysts to acids, free fatty acids could no longer be used for hydrogenation.

2nd Generation
Lurgi solved this problem by converting the raw material edible oil by transesterification with methanol to methyl ester and glycerine. This methyl ester could now be easily split with hydrogen to fatty alcohol and methanol on a trickle bed. The resulting methanol was recycled to the transesterification step.

3rd Generation
A further simplification of the fatty alcohol synthesis was achieved by combining both processes. Like in the 1st generation plant, the fatty acid from a neutral oil splitting could be used for the synthesis of the intermediate product, wax ester. This product was converted with a fatty alcohol (end product) without catalyst consumption to the corresponding wax ester at 250 °C. In the downstream hydrogenation step, two fatty alcohols were generated from this wax ester. One fatty alcohol was now processed to obtain the end product while the second one was recycled to the wax ester synthesis.

An additional cost saving potential (materials costs) for such a fatty alcohol plant could be leveraged if we succeed in reducing the plant pressure from the 250 bar required so far. New, highly active catalysts allowed for the first time to conduct tests aimed at considerably reducing the pressure required for the process.
Equally important are the “classical” analytical methods that are performed in the chemical laboratory. Methods applied here include:
- various titration methods,
- coulometry (Karl-Fisher analysis),
- spectrometric (UV/VIS) methods, and
- various extraction and filtration devices.

One particular focus is in the field of Biodiesel analyses, which is increasingly gaining in importance. Due to the broadened and often less-defined feedstock base for this process, the identification of traces of impurities that could affect the properties of the final Biodiesel product is becoming increasingly valuable.

Customer and Project Services
In addition to the aforementioned routine analyses, the services provided by the Analytical Department also include examining samples from commercial plants worldwide in order to cross-check the product quality or analytical methods applied. The Analytical Department is also involved in project execution. In cooperation with the project engineers they conceive the analytical laboratories (laboratory space, power and gas supply, ...), design them (layout, furniture, safety devices, ...) and propose the required equipment (analyzers). This task also includes the preparation of an Analytical Manual for the plant including all analytical procedures to be applied as well as training of the customer’s laboratory staff and possible start-up assistance.
Test Facilities

At the core of the R&D Center are the various pilot plants for the development of new and the optimization of existing processes. The operating conditions cover a very wide range:

- **capacity:** 50 mg/h up to several kg/h
- **pressure:** vacuum up to 350 bar
- **temperature:** up to 950°C

Apart from the widely used fixed-bed reactors for heterogeneous gas catalysis also reactors for three-phase flow (trickle bed, bubble column) and for suspended catalyst (stirred tank reactor, agitated autoclave) are in use. These reactors are operated in all different modes from isothermal to adiabatic to polytropic for the fast investigation of catalysts.

Various compressors are installed in a separate compressor room and used for the feed or recycle in high-pressure processes. In addition, a large automated gas mixing station is used to generate the pressurized synthesis gas used as feed for the methanol pilot plant.

There are also many distillation columns in the labs, either integrated into pilot plants for product purification or pretreatment or as stand-alone units for multi-purpose distillation tests. Since simulation programs do not always supply clear results for the behavior of trace components in the distillation sequence, it is imperative to confirm the product quality and purity by experimental results. Besides vacuum and pressure columns there is also a two-column set-up for distillative separation of azotropic mixtures.

The design and engineering of the pilot plants is performed by the respective project manager within the R&D department. There is also access to the respective experts in the Lurgi engineering department for specific issues, such as selecting construction materials, stress calculations for flanges or determination of EX zones (if required).

In general, all test equipment such as reactors, vessels, heat exchangers is made from stainless steel. Where highly corrosive components are present in the feed or product streams, Hastelloy is normally used. Equipment made from glass or plastic has also been successfully applied for certain

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### Test Facilities

<table>
<thead>
<tr>
<th>Property</th>
<th>MtSynfuels</th>
<th>DIN EN 590</th>
</tr>
</thead>
<tbody>
<tr>
<td>cetane number (1)</td>
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<td>&gt;51</td>
</tr>
<tr>
<td>density @ 15 °C, kg/m³</td>
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<td>820–845</td>
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<tr>
<td>PAH, wt.% (1)</td>
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<tr>
<td>sulfur, ppm (1)</td>
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<td>&lt;350</td>
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<td>flash point, °C (1)</td>
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<td>kinem. viscosity, mm²/s</td>
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<td>2.0–4.5</td>
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</tr>
<tr>
<td>distillation 350 °C, vol.% (1)</td>
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<td>&gt;85</td>
</tr>
<tr>
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<td>cloud point, °C (1)</td>
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</tr>
<tr>
<td>oxidation stability, g/m³ (1)</td>
<td>7</td>
<td>&lt;25</td>
</tr>
</tbody>
</table>

(1) ASG Analytik Neusäss
The fabrication, construction and erection of the pilot plants is all done on-site, with the assistance of service companies and workshops that are also operating on the premises of the R&D Center.

The measuring and control instruments of the test facilities have been designed for continuous operation. Most plants are computer-controlled with automated data collection, control and alarm responses. Well-trained shift personnel attend to the plants and supervise their operation around the clock. Samples of the feed, product and recycle streams are then analyzed in the analytical laboratories.

The main purpose of the pilot plant tests is to determine accurate design parameters for the respective process including but not limited to
- reactor and reaction models,
- material balances and stream compositions,
- purification sequence and quality of the end product.

The data can then be used directly by the technology and engineering departments for the design of the corresponding commercial-scale process. For some processes it is also necessary to produce representative samples of specific products in order to prove the quality of the products to potential customers.

Outlook

In the coming years, research and development is expected to contribute even more effectively than in the past to the corporate success of Lurgi.

We want to reach this goal by:
- developing additional proprietary/exclusively licensed technologies
- acquiring market leadership for proprietary/exclusively licensed technologies
- acting as trendsetters for technologies

As a consequence, the following action plan has been agreed:
- doubling of the R&D budget over five years
- enhanced R&D and product development
- extension of alliances

These strategic objectives, set by the company’s management for research and development, will not only secure but also strengthen the position of Lurgi as a technology-oriented engineering contractor on a sustainable basis well into the future.
## Key Figures of Lurgi’s R&D

<table>
<thead>
<tr>
<th>Infrastructure</th>
<th>R&amp;D Manhour Capacity</th>
<th>Budget</th>
</tr>
</thead>
</table>
| - More than 30 pilot and bench-scale plants  
- 3 analytical laboratories  
- 24 hour shift operation | - 40,000 at the R&D Centre  
- 17,000 at the in-house process department | - € 10.5 million/year |
Lurgi is a leading technology company operating worldwide in the fields of process engineering and plant contracting. The strength of Lurgi lies in innovative technologies of the future focusing on customized solutions for growth markets. The technological leadership is based on proprietary technologies and exclusively licenced technologies in the areas gas-to-petrochemical products via synthesis gas or methanol and synthetic fuels, petrochemicals, refinery technology and polymer industry as well as renewable resources/food.

Lurgi is a company of the Air Liquide Group.