When a chemical or pharmaceutical company contacts a provider of services and equipment for the separation and purification of its chemical products, it usually has completed thorough studies on its processing capacity needs. The nameplate capacity of the plant and the required product quality are determined by existing market demands and projections for the future. World-scale capacities are increasing in response to rising demand and due to streamlined production capacities. With that, the scaling up from pilot test plants to full industrial sizes is becoming more challenging. The method the engineer uses to calculate and design a piece of equipment of industrial size based on a test at a smaller scale is called “scale-up.” Some equipment can be scaled directly from laboratory size to industrial size; others require the construction of a pilot or semi-industrial-sized plant as an intermediary step. The scale-up factor is defined as the size of the large equipment or plant divided by the size of the pilot unit. Consideration of typical scale-up factors limits the risk of incorrect engineering, so that the plant finally produces according to the customer’s specifications. But each step represents a substantial investment. At Sulzer the scale-up is based on pilot tests realized for the production rate at a scale of a few kilograms per hour. The results allow the design of a plant that typically processes thousands of kilograms per hour. Because most of the equipment that Sulzer delivers to

1 The successful scale-up of a large distillation complex led to additional orders of similar magnitude.

Sizing beyond limits

In separation technology, the methodologies developed to scale the results obtained in pilot equipment to large industrial units are limited in their validity. Engineers are sometimes challenged with the task of constructing units of exceptionally large sizes—sizes that are far beyond the usual limit of confidence. The extrapolations required are then based on extensive experience, knowledge of the specific separation technologies, and careful attention to the customer expectations.
customers is of proprietary nature, this is a demanding task that requires a solid theoretical basis and a profound understanding of the technologies. Based on built-up experience, the Sulzer engineers are able to provide a scaled-up design that is accurate enough to supply the customer with guarantees on the plant performance. Yet, from time to time, the customer’s specifications ask for a capacity that is much larger than anything designed before. The size may be so large that the basis and even the method for the scale-up become questionable. These situations require special skills to successfully predict the unusual complications that go with these giant steps.

Scaling up distillation columns
Distillation is a well-known and a very powerful separation technology. Sulzer has built and operated rectification towers of many possible sizes. Very slim, tall columns up to 90 meters high and less than 0.10 meter in diameter are used to separate isotopes. Sulzer has also designed and manufactured column sections of more than 15 meters in diameter and less than 2 meters high for the wash bed of crude oil vacuum towers. Some rectification towers operate at high pressure and others at vacuum. The scale-up challenges have been studied and described by many authors and are quite well known. Nevertheless, new requirements from the market, such as higher purity and higher production rates, continuously push beyond the limits of experience.

A customer required a purification unit for the monomer MDI (methylenediphenyl diisocyanate), which is used for the production of polyurethane. Although experience had been gained with piloting in a column of 0.10 meter diameter and industrial experience was available for production up to 50 kilotons per year (ktpa), the design for production of a few 100 ktpa, combined with a very low operating pressure represented a challenge. Sulzer managed to ensure proper liquid distribution over the whole diameter and to keep the pressure drop to a minimum. In addition, the liquid holdup had to be kept as small as possible to lower the formation of side products. Another challenge was the sizing of the equipment to condense the top vapor with a duty of several megawatts at an absolute pressure of a few millibars. The unit was successfully started and has been running profitably for several years (Fig. 1).

Design of large liquid-liquid extraction systems
There are several industrial technologies for liquid-liquid extraction. One of the most efficient setups is the agitated contactor. The solution is based on a collection of agitated compartments arranged vertically in a column. Each compartment is equipped with an agitator and is separated by partition plates (Fig. 2, left). The main challenges for the design of liquid-liquid extraction systems are the changing physical properties along the column and the strong variation of the internal flow rates when a large amount of material is exchanged between the two liquid phases. The Kühni agitated column ECR can be designed for such conditions. Once the equilibrium diagram of the phases is available, a test at pilot scale of 0.06 meter diameter is run to validate the design. For a scale-up to a size

2 For this liquid-liquid extraction unit (left), Sulzer scaled the agitation system up to a diameter of almost 3 meters (right).
above 1 meter in diameter, it is necessary to verify hydrodynamic parameters such as the flooding point using a pilot column of 0.15 meter diameter. A customer wanted to produce food-grade phosphoric acid from a crude acid stream that contained several impurities such as sulfate and different metallic ions. Sulzer already had several references for this application that used the agitated ECR columns. Usually industrial size columns process a few tons of feed per hour. In this case, however, the feed was over 30 metric tons per hour, and the process required three very large liquid-liquid extraction columns in series (Fig. 2, right). In scaling up the agitation system to a diameter of almost 3 meters, it was critical not to increase the height of the agitated compartments too much in order to minimize the overall height of the columns. On top of this, the feed-to-solvent ratio, being above 25 to 1, was exceptionally high, which added to the general complications. An important part of the solution was to adapt the geometry of the column internals along its height to cope with the changing hydrodynamic conditions. The columns were delivered to the customer in the USA and have been in operation for over 25 years.

**Scale-up at Sulzer**

Sulzer Chemtech’s Process Technology business unit looks for the most economical solution based on experience and process simulation. Once the solution matches the customer’s expectation, Sulzer offers a pilot test to validate the concept with the actual feed and to confirm the product concentrations specified. On this basis, Sulzer designs, fabricates, and delivers the industrial-sized equipment to the customer.

www.sulzer.com/Process-Technology

**Melt crystallization units with huge capacity**

Crystallization is a very efficient separation technology. Melt crystallization is based on the thermodynamic principle that impurities prefer to remain in the liquid phase rather than in the crystal phase. It is used for many applications, like paraffin deoiling and monomer purification. It has been successfully applied in combination with distillation to obtain pure MDI and chloronitrobenzene, for example. In general, this purification process is tested and optimized in a pilot plant. The scale-up for the industrial unit is then a straightforward
“Scaling up process technology is an exciting challenge because large vessels and complex plants are simply fascinating.”

Laurent Zuber, Chemtech Process Technology
of the vapor and the boiling liquid are exactly the same (called azeotropic composition). There are different ways to overcome this obstacle by combining different unit operations—like distillation and membrane separation (for example, for ethanol drying), or distillation and extraction (the extractive distillation like applied for benzene/cyclohexane separation). In some cases, the azeotropic composition depends strongly on the operating pressure, and the components can be separated by pressure swing distillation. This method uses a combination of two distillation columns that operate at different pressure levels.

A customer producing an active ingredient of a blockbuster drug used ethyl acetate and cyclohexane as solvents and needed to recycle these. The mother liquor coming from the reaction also contained traces of other compounds. So, a first large-scale batch distillation was used to remove most of these unwanted components and obtain the solvent mixture that needed to be recycled. The next step was a pressure swing distillation. The first column, operating under pressure, produced pure cyclohexane at the bottom and the azeotropic mixture at its top. The top product was processed in a second column which was operated under vacuum and produced pure ethyl acetate at its bottom. The azeotropic mixture obtained at the top of the second column was recycled back to the first column.

The challenges in this project were not only the difficult separation tasks, but also the delivery of the solvent recovery plant as completely preassembled skids (Fig. 5). These only needed to be set up and tied in to the customer’s plant and utilities. The plant was delivered and started up within a very short time, and there was minimal interference to the existing production site. The client has three such plants in operation, and all have been running for more than eight years.

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Literature