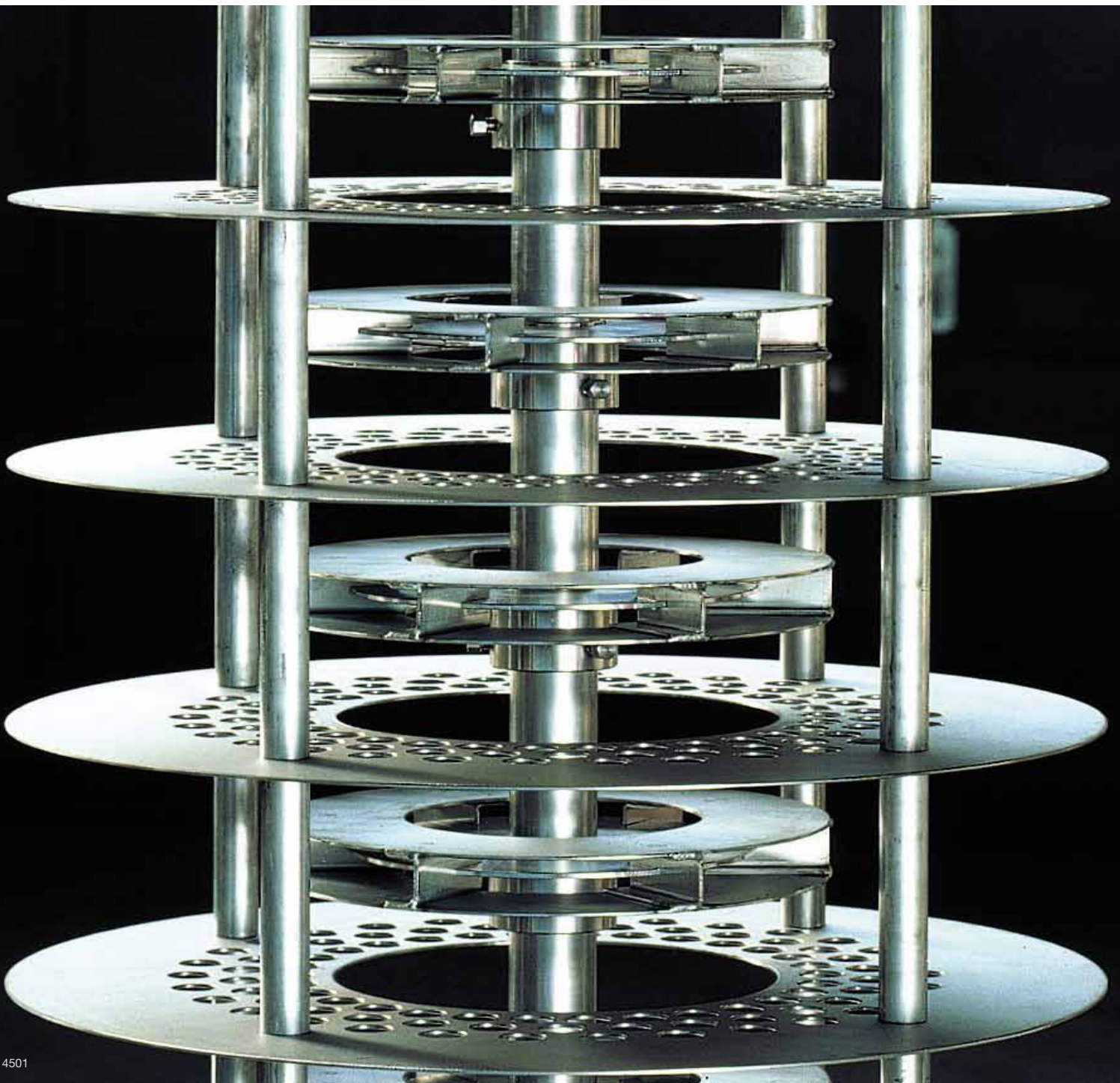


Liquid-Liquid Extraction Technology



Liquid-Liquid Extraction Technology at Sulzer Chemtech

Sulzer Chemtech, a member of the Sulzer Corporation, with headquarters in Winterthur, Switzerland, is active in the field of process engineering, employing 3'000 persons worldwide. Sulzer Chemtech is represented in all important industrial countries setting standards in the field of mass transfer and static mixing with its advanced and economical solutions.

Sulzer Chemtech is organized into four business units, one of which is the Process Technology group. This business unit was formed in early 2009 following the acquisition of Kühni, a Swiss company with more than 75 years experience in innovative separation processes. Today, Sulzer Chemtech Process Technology is headquartered in Allschwil (Basel), Switzerland. We provide a unique and wide portfolio of separation and application technologies, amongst which liquid-liquid extraction.

Working Principle

Liquid-liquid extraction is an important separation technology, with a wide range of applications in the modern process industry. The extraction process is based on different solubilities of components in two immiscible, or partially miscible, liquids. The components that need to be recovered are extracted from the feed stream with the help of an extractant (often called solvent). Both liquids have to be thoroughly contacted and subsequently separated from each other again. To achieve high purities and yields, it is necessary to operate with multiple stages and with the liquids flowing countercurrently.

For successful separation by extraction, all components must meet certain specifications. The main criteria for the extractant are a favorable partition coefficient, a high selectivity and an easy separation from the extracted product. The basic conditions for the pair of liquids are a low mutual solubility and a difference in density, which is the driving force for the motion of the droplets. In most multistage extractors,

the liquids are transported countercurrently. The viscosity and interfacial tension are additional important parameters.

In nearly all liquid-liquid extraction processes one of the liquids is dispersed into the second liquid in the form of droplets. The key for a high process performance is an adapted droplet size and a uniform hold-up profile throughout the column. This requires specially adapted equipment and in-depth know-how about two phase liquid flows.

Fields of Application

Liquid-liquid extraction is a complex separation process. An additional component has to be introduced as extractant, which makes other subsequent separation steps necessary. Therefore, liquid-liquid extraction is mostly used when separation of components by distillation is either uneconomical, or even impossible. Some examples are:

- Simultaneous separation of various components with widely differing boiling points
- Separation of high-boiling products or pollutants which are present in only low concentrations
- Separation of components with similar boiling points or components forming azeotropes
- Separation of mixtures with thermally sensitive components
- Selective separation of single components out of a multi-component mixture
- Separation of components from electrolytic solutions

Our head offices in Allschwil, Switzerland



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Overview of Equipment

Our liquid-liquid extraction is a technically sophisticated separation technology, offering you a solution where other technologies are uneconomical or even not feasible

Extractors

The main task of any extractor is to create optimal conditions for mass transfer between the two phases. This includes droplet formation / dispersing, transport and settling of the phases. Two types of dispersion are possible. The feed can be dispersed into the solvent, or be continuous with the solvent dispersed into it. These two types often show a considerably different behavior. Also, the type of contact between the two phases is an important, equipment related aspect. In the case of stage-wise contact, the liquids are completely separated from each other in every stage, whereas in the case of differential contact, the liquids are in continuous, countercurrent contact. Both systems have specific advantages affecting equipment sizes and extraction efficiency.

Classification

A wide range of equipment for liquid-liquid extraction is available, which can be categorized as follows:

- Extraction columns
- Mixer-settlers
- Centrifugal extractors

The extraction columns, also referred to as extraction towers, can be further differentiated into three groups:

- Static without energy input
- Agitated
- Pulsed

Extractor Types

You can find our equipment in almost all industrial applications. Our portfolio is based on a number of modern, efficient types of extractors. The core of this range is the agitated Kühni column type ECR with an extremely wide field of applications, and the Sulzer packed column ECP. Mixer-settler type extractors are available if process reasons do not favor the use of columns.

Column type extractors:

- Agitated Kühni column ECR
- Sulzer packed column ECP

Mixer-settler type extractors:

- Mixer-settler EMS
- Mixer-settler column ECMS

Sulzer extraction packing



Installation of an industrial ECR type column on site



Agitated Kühni Column ECR

Our flexible, multipurpose extraction column

Basic and Mechanical Design

The agitated Kühni column is a well-proven, highly efficient extractor. Its main characteristics are the agitated compartments, arranged one on top of each other. The concept is simple, yet, its hydrodynamic performance is outstanding.

Turbine Mixers and Partition Plates

Turbine agitators installed in each stage are responsible for the formation of the dispersion. They create a torus-shaped, rotary flow pattern and also mix the liquid phases radially, which is an important condition for safe and reliable scale-up. Perforated plates form the boundaries between each agitated compartment and reduce back-mixing to enhance the separation performance.

The hydrodynamic conditions can be influenced by varying four geometrical parameters:

- Turbine diameter
- Turbine height
- Open area of the partition plates
- Height of the compartments

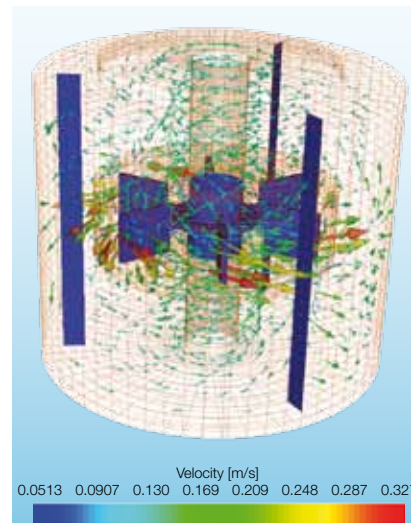
With this flexible geometry, it is possible to optimize performance regarding throughput and mass transfer, even in cases where process parameters and physical properties vary strongly along the length of the column.

The agitated Kühni column ECR has a simple and robust construction. The drive unit and the shaft are supported at the top of the column. All common types of shaft seals can be used (stuffing box, mechanical seals). In special cases, the seal can be replaced by a magnetic drive. Only radial slide-bearings are necessary inside the column, which are accessible through hand- or manholes. The sturdy design, the materials selected and the low rotational speed of the agitator in industrial columns (typically 10-70 rpm) allow a practically maintenance free operation.

Advantages and Characteristics

Based on its flexible design and our long-standing experience, the Kühni column ECR has a range of advantages:

- Universal use
- High flexibility for varying process parameters and physical properties
- Reliable scale-up
- More than 30 theoretical stages in one single column
- Standard turndown 1 : 3
- Column diameter: 30 mm - 3.5 m
- Physical properties:
 - Density difference > 40 kg/m³
 - Viscosity < 500 mPas
 - Interfacial tension > 2 mN/m
- Differential phase contact
- Phase ratio up to 70 : 1
- Materials:
 - Stainless steel / higher alloys
 - Special metals, for example, titanium
 - PP / PVDF / PFA
 - Glass-lined shell



CFD analysis of an agitated extraction column compartment

Pilot glass extraction column



Packed Column ECP

Our extraction column of choice for high throughput

Basic and Mechanical Design

The ECP packed column is based on the current state-of-the-art extraction research. The column consists of a packed bed, distributors for the two liquid phases and, optionally, a pulsator.

Packing

The special Sulzer extraction packing reduces the back-mixing of the continuous phase, thus providing nearly plug flow conditions and a narrow droplet size distribution. Both are necessary to achieve high throughput and high efficiency. To further increase the separation performance, additional dual flow plates can be inserted between the packing elements.

Liquid Distributors

In order to create an even flow profile at either end of the packed bed, both liquid phases are distributed over the whole cross sectional area by suitable distributors. The design of the dispersed phase distributor requires special attention, in order to achieve a narrow droplet size distribution.

If the column is operated without energy input (unpulsed), the packed extraction column is a simple item of equipment without any special mechanical features and free of moving parts. Often, however, the liquid has to be pulsed to increase the separation performance and provide a further control parameter for changing operating conditions. Pulsators are typically specified with intensities (frequency

amplitude) of 800 to 1200 mm/min. Such pulsators are available as standard items and are used with our packed extraction columns.

Advantages and Characteristics

Based on the special design of the packing and liquid distributors, the ECP column has a number of advantages:

- High specific throughput resulting in:
 - Small column diameters
 - Revamp options of existing columns to increase capacity
- Capable to deal with challenging physical properties:
 - Low density difference $< 50 \text{ kg/m}^3$
 - Low interfacial tension $< 2 \text{ mN/m}$
 - Tendency to form emulsions
- Reliable scale-up
- Standard turndown 1 : 2
- Column diameter: 50 mm - 5.6 m
- Physical properties:
 - Density difference $> 30 \text{ kg/m}^3$
 - Viscosity up to 100 mPas
 - Interfacial tension $> 1 \text{ mN/m}$
- Materials:
 - Metals
 - PTFE, PVDF, PFA



Packed internals of a liquid-liquid extraction column

Mixer-Settler EMS and Mixer-Settler-Columns ECMS

If you need stage-wise contact but have space constraints

Conventional Mixer-Settler EMS

Mixer-settlers operate with a purely stage-wise contact. After every mixer, there is a settler. Mixer-settlers can be operated in a multistage, countercurrent fashion, which, however, is associated with considerable piping and instrumentation and also requires large areas for installation. Because of these implications, mixer-settlers are only used if process reasons dictate a stage-wise contact.

Conventional mixer-settlers are made either as a box or a tubular type design. The latter is also used for glass equipment and when higher design pressures are required.

The most important element of every mixer is the pump-mix turbine. It has to create the dispersion and the hydrostatic head for transport of the liquid. An additional agitator can be mounted on the same shaft to optimize the dispersion and enhance the efficiency.

Mixer-Settler-Column ECMS

As implied by the name, the mixer-settler-column is a series of mixer-settlers in the form of a column. It consists of a number of stages installed one on top of the other, each hydraulically separated, and each with a mixing and settling zone. With this design, it is possible to eliminate some of the main disadvantages of conventional mixer-settlers, whilst maintaining stage-wise phase contact.

The mixer turbines do not need to transport the liquids from stage to stage, therefore, the rotational speed can be adjusted to achieve optimal droplet sizes. Due to the requirement for settling of the liquid phases in every stage, the specific throughput achievable in a mixer-settler-column is considerably lower in comparison to other extraction columns ($< 10 \text{ m}^3/\text{m}^2\text{h}$). The mechanical design of the mixer-settler-column is comparable to the agitated Kühni column ECR.

Advantages and Characteristics

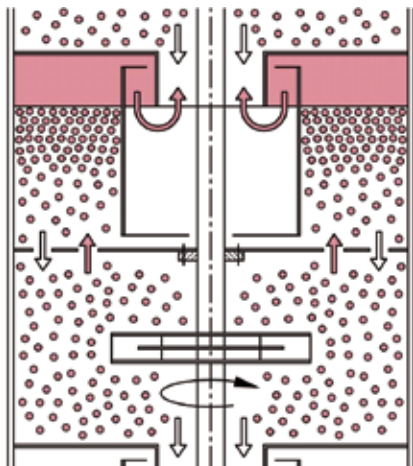
Mixer-settlers EMS have a number of specific advantages and are suitable for:

- Long residence times ($> 15 \text{ min}$):
 - Extraction controlled by residence time
 - Reactive extraction systems
 - Long phase separation times
- Extraction controlled by pH (stage-wise pH adjustment)
- Batch extraction
- Stage-wise phase contact

Specific advantages of the mixer-settler-columns ECMS are:

- Small footprint
- Column like design
- Allows extreme to unlimited phase ratio

Principle of a mixer-settler-column



Examples of mixer-settlers



Selection of Equipment

The right choice of equipment is important for a stable operation and optimal performance

Correct Selection of Equipment

The correct selection of the type of extractor is an important step in every extraction project. Various selection aids, such as decision trees, point score tables, and diagrams can be found in the literature. All extractors have one behavior in common: the efficiency of the extraction is linked to the specific capacity. Basically, the selection of equipment should be made using process and economic criteria. However, this can be difficult at the beginning of a project when without testing the type and size of the extractor required are not known.

Process Related Criteria

The agitated ECR column with its inherent flexibility and wide operating range is the most suitable extractor for the majority of cases. Exceptions and special applications, which require a different type of extractor, can be identified with a few criteria that are easily checked:

- Necessity of stage-wise contact
- Requirement for a residence time > 15 min
- Criteria in favor of a mixer-settler:
 - Density difference < 50 kg/m³
 - Interfacial tension < 2 mN/m
 - Tendency to form emulsions
 - Low number of theoretical stages

- Criteria in favor of a packed column:

- High throughput
- Limited number of stages required
- No moving parts desired

Additional Criteria

When examining economic aspects, reliability is an important factor. Delayed commissioning, non-performance, downtime due to equipment failure lead to loss of production and may bring substantial costs to rectify. Therefore, besides pure technical criteria, the experience and reliability of Sulzer Chemtech as technology partner are important attributes. When cooperating with us, you will find a partner with outstanding test facilities and numerous reference applications in liquid-liquid-extraction in the field. Our state-of-the-art equipment and proven scale-up procedures, in combination with our highly skilled employees will provide you a process solution with guaranteed performance.

Outlook

Research in the field of liquid-liquid extraction continues with great effort. We continue to

innovate our extraction technology, with particular attention to the following two areas: miniplant and modeling.

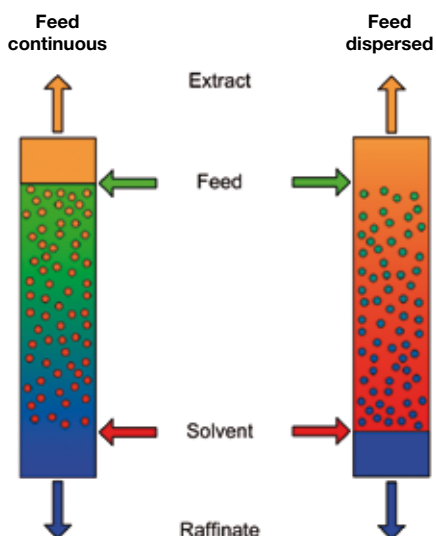
Miniplant and Modeling

The continuous improvement of miniplant technology on the one hand, and the mathematical modeling of extraction columns with population balance or Monte-Carlo methods on the other hand are merging at the same time.

With the help of these complex models and small laboratory scale droplet experiments it is possible to predict the performance of extraction columns in pilot scale in specific cases. This method can be used to reduce the effort in pilot trials as basis for the optimized design of an extraction column and therefore, reduces both costs and time-to-market.

Future developments are expected to extend the application of this method to the scale-up of industrial size columns. It would then only be necessary to establish the model parameters, with the help of small laboratory measuring cells. We intensely participate in and support this field of research within our own test center and by our cooperations with both industrial and university partners.

Sections of pilot columns



Feasibility Study and Trials

Your optimal process solution is guaranteed by our design based on pilot trials in our state-of-the-art test center, and our extensive know-how on our liquid-liquid extraction technology

Optimal Process Solution

In spite of considerable progress in the research and development of liquid-liquid extraction technology, it is still not possible to design an extractor from first principles. Therefore, a systematic test method is very important. We offer you decades of relevant experience to call upon, including the necessary test methods and facilities, incorporating various laboratory and pilot plant equipment.

The design of an extractor is carried out in four steps:

- Execution of a feasibility study
- Generation of basic data
- Pilot trials
- Scale-up and design

Feasibility Study and Basic Data

The success of an extractive separation step depends largely on finding a suitable extractant. In many cases, based on our experience we are able to propose such a solvent. However, for new processes, a professional and analytical screening of suitable solvents is required. The feasibility of the selected extraction process is then often confirmed in a mini-plant test run.

In the next step, basic data are established which are used as input for subsequent pilot trials and also permit a preliminary sizing of the extractor. These basic data include:

- Mass balance, including concentrations
- Physical properties (density, interfacial tension, viscosity)
- Liquid-liquid equilibrium data

Lab and Pilot Trials

Laboratory or pilot plant trials are necessary to secure the correct design of extraction equipment. We undertake these to establish optimal operating conditions, and, depending on the extractor type, the most favorable geometric parameters (size and type of internals). Successful trials allow us to safely scale-up to an industrial size unit, and to provide you full process guarantees. We are capable of carrying out all testing, including establishing missing physical properties, in our own test center. At this location we have the following test extraction equipment available:

- Extraction columns:
 - Agitated columns with diameter 32, 60, 150 mm
 - Packed columns with diameter 50 and 150 mm
 - Shell: glass
 - Internals: SS316L, hastelloy, titanium, zirconium, PP and PVDF
- Mixer-settlers:
 - Tube type mixer-settlers
 - Total volume per stage: 70 ml – 15 l
 - Material: glass / SS316L

Rental equipment is available for tests on your premises. If required, we can provide you assistance and training for such in-house tests.

The miniplant size extractor ECR 32 is well suited for miniplant piloting of complete processes including recycle streams. This small-scale unit proves the feasibility of the proposed solution and generates preliminary scale-up data while requiring only a minimum amount of feed material.

Sulzer test center in Allschwil, Switzerland



Agitated liquid-liquid extraction column in pilot scale



Design, Fabrication and Delivery of Equipment

Our extensive experience in design and fabrication in our modern workshops ensures top quality extraction units at your service

Design and Scale-up

In the field of liquid-liquid extraction, scaling-up from pilot units to industrial size equipment is an important and critical step. It is not at all straightforward and simple empirical methods will normally fail. Our extensive experience in successful scale-up is supported by our detailed understanding of the fundamental theories of hydrodynamics and mass transfer playing a role in extraction. Our Sulzer extractors have often been the topic of scientific research at various universities.

Installation of an industrial scale ECR extraction column



Manufacturing Service

Every extractor we supply is manufactured under our complete responsibility in modern workshops equipped with state-of-the-art manufacturing tools with certified production processes. Column shells are either manufactured in-house, or by approved and qualified sub-suppliers. The latter approach is followed for very large columns, for glass-lined columns, or when special design codes are to be applied.

GMP and CIP are requirements well-known to us. Design, manufacture and certification of many extractors have been carried out in accordance with these and other standards.

We deliver our extraction technology as a basic design package with state-of-the-art proprietary equipment. In addition, we have a specific long-term experience in fabrication, installation and start-up of our solutions as skid-mounted units.

Advantages of skid-mounted, turn-key plants are:

- Short delivery time through an integrated fast track execution
- Low overall investment costs
- Reduced on-site erection and hook-up time and thus minimal site disruption
- Manufacture of the complete plant under workshop conditions ensuring the highest quality
- Completion of the Factory Acceptance Test (FAT) prior to delivery
- Qualification tests prior to plant delivery

Installation, Commissioning and Start-up

We have a team of highly experienced installation supervisors, who provide construction support services for site-built plants, and for the off-loading and installation of skid-mounted plants. These supervisors liaise closely with your construction manager, safety officer and mechanical contractor, to ensure a safe and trouble-free installation.

Following installation, our process engineers undertake plant commissioning following a structured plan. The commissioning team is usually headed by the process engineer responsible for the plant design. This phase ends with the start-up of the process unit followed by a Site Acceptance Test (SAT). Training of your operators may take place in parallel.

At plant hand-over, you will be provided with final, as-built documentation including operating manuals, final lay-out drawings and certificates. A dedicated after sales group ensures the support for spare parts and services during the whole lifetime of the plant, wherever you are around the globe.

Right from plant start-up you can benefit from your optimal performing plant solution based on our liquid-liquid extraction technology.

Application and Processes

The following examples give you a taste of our collection of successful applications

Sulzer extractors are successfully applied in every segment of the process industry:

- Bulk chemical industry
- Petroleum industry
- Fine chemical industry
- Pharmaceutical industry
- Biotech industry
- Food industry
- Hydrometallurgy

Standard Processes

On the basis of decades of both experience and in-house development, we have acquired our own detailed process know-how for many classic liquid-liquid extraction applications. As a result of this, in some specific applications, our Sulzer extractors are used almost exclusively.

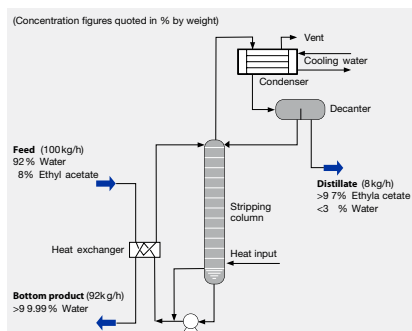
Below we give you an overview of a few typical applications where our extraction solutions are used.

Solvent Recovery

Many standard processes for the separation and recovery of various solvents include extraction:

- Recovery of high-boiling, organic components, in low concentrations, from industrial wastewaters (for example, DMF and phenol)
- Breaking of azeotropes (for example, pyridine/water)
- Removal of water-soluble components from a complex solvent mixture (for example, ethanol, methanol and IPA)

Wastewater stripper with optional decanter including a wastewater example



Wet Purification of Phosphoric Acid

Pure, food-grade phosphoric acid is currently almost exclusively produced using the so-called wet process. This process includes three extraction steps with a scrubbing stage in the middle to remove impurities. Our extensive and specific experience covers a multitude of large columns installed worldwide.

Extraction of Aromatics

Two agitated ECR columns are used in the IFP process for recovery of aromatics (benzene, toluene, xylene) from hydrocarbon mixtures. In the first column of this process the extraction is carried out with DMSO, combined with a backwash. The extreme profiles of flow rate and physical properties require a high flexibility in the

geometry of the extraction column internals.

You can find Sulzer extraction columns in several other processes for removal of polycyclic aromatics out of oil streams, in both production and recycling.

Purification of Organic Acids

Dilute aqueous carboxylic and sulphonic acids can be concentrated and purified with the help of liquid-liquid extraction. A classic example is the recovery of acetic acid from streams with concentrations of 5-50 wt%. Higher acids are recovered by reactive extraction with amines, which extract the acid by forming reactive bonds. An example is the extraction of lactic acid following the fermentation process.

Loading of a Sulzer Chemtech modular system for recovery of solvents and purification



Application and Processes

Client Specific Applications

We jointly develop your process solution and tailor our extraction technology to your needs

Client Specific Applications

Our extractors are widely used and tailor-made to your specific applications. In the majority of cases the optimum process solution was developed in close cooperation with the customer and based on pilot trials. We have an extensive track record of numerous, highly customer specific applications based on our liquid-liquid extraction technology.

The following forms a summary of our reference list of extracted substances.

Acetic acid, acetonitrile, acetyl-para-aminophenol, alkaloids, ammonia, aniline, benzoic acid, caprolactam, catalysts, cresols, diacetone sorbose, dichlorobenzene, diethylaniline, dimethylacetamide, dimethylformamide, dimethyl ketone, dimethyl sulfoxide, ethanol, guaiacol, hydroquinone, isopropanol, lactone, metal ions (like Fe^{3+} , Na^+ , Pd^{2+} , and Au^{3+}), methanol, n-methylpyrrolidone, monoacetonesorbose, nitrobenzene, nitric acid, pesticides, pharmaceuticals, phenol, potassium carbonate, potassium chloride, pyridine, sodium borohydride, sulfate, sulphuric acid, sulphonic acid, tetrahydrofuran, triacetoneamine, vanillin, vitamins, water, wine aroma, wool wax alcohol, xyleneol.

Specific Case Stories

The following case stories demonstrate the variety, complexity and attractiveness of liquid-liquid extraction when applied to specific fields.

Caprolactam

This application presents the successful operation of the agitated Kühni column type ECR in a case with extremely low interfacial tensions in the range of 1–20 mN/m. The total product yield with our process amounts to over 98%.

Pesticides

Pesticides are produced in the agrochemical industry, where our liquid-liquid extraction process has proven application serving two purposes, namely, the treatment of wastewater and the recovery of the chemical compound for re-use.

Used Oil Recycling

Our technology has proven use in extraction of polycyclic aromatics from used engine and lube oil, to improve the color index and oil quality.

Triacetone Amine

A typical example of the application of our extraction process is in the treatment of industrial wastewater with 5% triacetone amine, whereby reductions down to 5 ppm (factor 10'000) can be achieved, with a phase ratio of 35 : 1.

Wool Wax Alcohol

Wool wax alcohol can be extracted following the saponification of wool grease. Another typical application in this field is the purification step in the manufacture of lanolin (ingredient in cosmetics) from wool grease.

Separation Based on Polarity

In numerous applications the separation of a polar component, like an alcohol or ketone, out of a non-polar matrix, for example monomer or hydrocarbon, is required. In this so-called washing step a high flexibility and often extreme phase ratio is required.

Removal of Phenol from Industrial Wastewaters

Our extraction process has also proven its use in environmental applications. A typical example in this field is formed by extraction of phenol from industrial wastewater with a heavy oil. The extract is subsequently used as burner fuel, thereby recovering energy in addition.

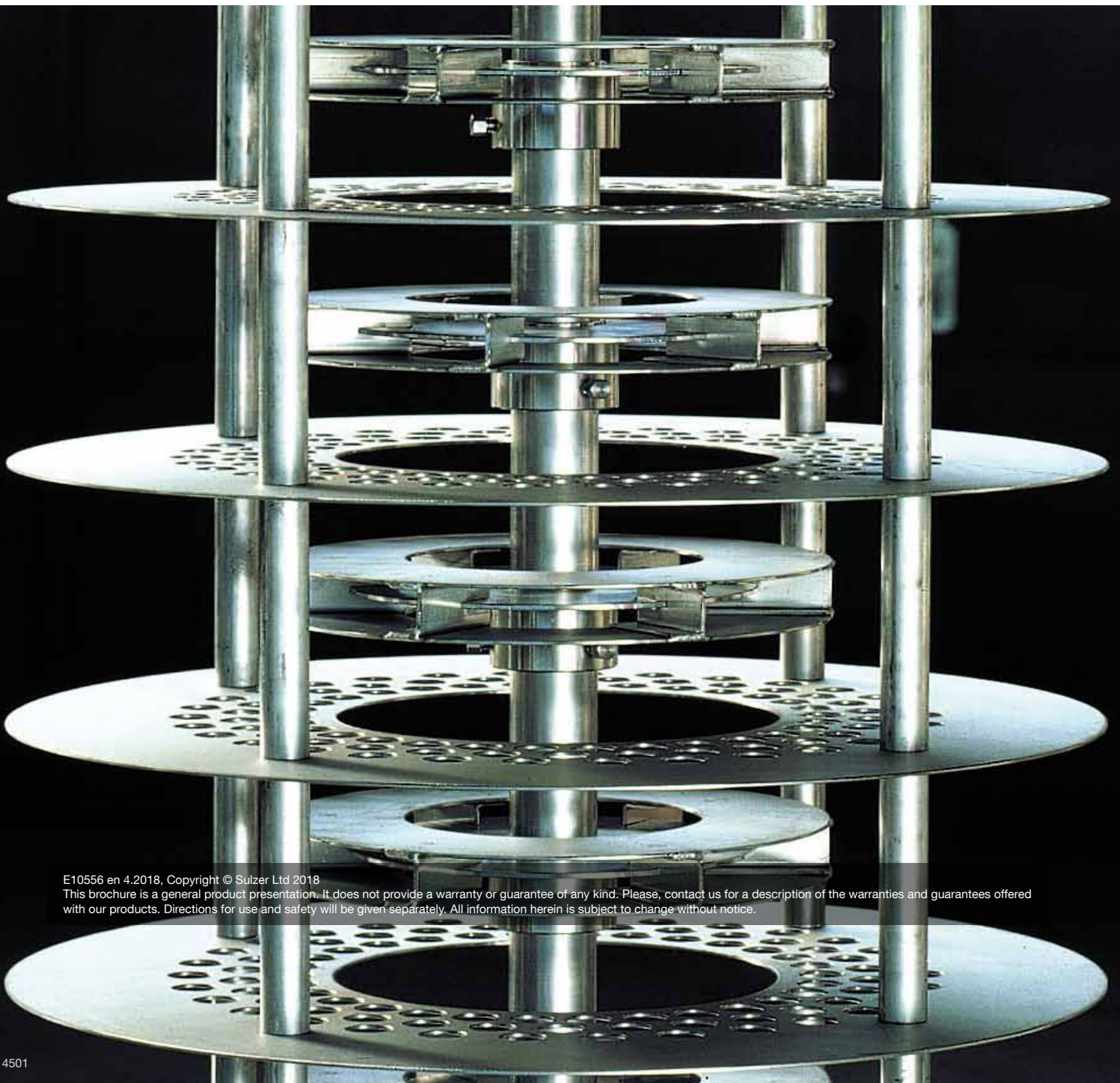
Part of an extraction column on a truck



Glass-lined column shell with PVDF internals



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