



# **EFFICIENT.**

**CONCENTRATION, PURIFICATION AND  
RECYCLING OF  $H_2SO_4$  AND  $HNO_3$ .**



# POTENT TECHNOLOGY FOR THE CONCENTRATION OF H<sub>2</sub>SO<sub>4</sub>.

## **Economically viable, environmentally sound.**

Nowadays more and more spent acid is being recycled – for environmental reasons, but also because it makes sound economic sense. Bertrams Chemical Plants Ltd. offers sulphuric acid recycling systems for a variety of applications combining acid purification and concentration.

## **Reliability for years to come.**

Bertrams Chemical Plants Ltd. invests its long years of experience and its wealth of expertise in the recycling of sulphuric acid. Plant reliability and quality are accorded the highest priority during the design, development and operational phases. This is our guiding principle when we offer complete systems customized to suit the individual requirements of the user.

- Technology tried and tested over many years of operation
- Customized plants with optimized processes and economics
- Carefully selected construction materials eliminate corrosion and offer high protection against failure
- Emissions reduced to a minimum
- Very low acid losses
- NO<sub>x</sub> absorption units

## **Applications for Bertrams H<sub>2</sub>SO<sub>4</sub> plants.**

- Reconciliation and, when needed, purification of spent sulphuric acid for reuse in the same process, e.g. chlorine drying and the manufacture of organic intermediates
- Reconciliation of clean spent acid for subsequent use in other processes, e.g. fertilizer production
- Preconcentration of the spent sulphuric acid feed to a thermal decomposition plant for the total regeneration of fresh acid

## **Typical sources of spent acid are:**

- Nitration processes such as production of polyurethane intermediates (DNT/TDI & NB/MDI) and production of organic chemicals (aromatics, aliphatics and derivatives)
- Nitration of inorganic metals such as nickel-cobalt, bismuth, etc.
- Production of explosives for both civil use (Pentrite, nitrocellulose, etc.) or military use (NG, NC, PETN, TNT, RDX, T4, NIGU)
- Production of industrial nitrocellulose (NC) for lacquers and/or celluloid
- Manufacture of specialty products for pharmaceuticals (nitro-glycerine, nicotinic acid)
- Production of SBA/MEK
- Production of aramid fibers
- Chlorine drying
- Plastic processing industries: adipic acid, MMA-methylmethacrylate, etc.
- Nitric acid production.

# SULPHURIC ACID: EFFICIENT SOLUTIONS FOR DIVERSE PROCESS PROBLEMS.

## Unit operations.

Sulphuric acid is an important product in the chemical industry: it is a catalytic agent for synthesis processes but also an ideal drying agent for gases such as chlor, bromine, etc. Sulphuric acid will bind water generated by chemical reaction and will improve the chemical equilibrium in the direction of the preferred products.

Bertrams Chemical Plants Ltd. offers a comprehensive range of processes to achieve every desired acid concentration up to 96/97% H<sub>2</sub>SO<sub>4</sub>. The following unit operations are applied to suit the specified duty:

- Preconcentration
- High concentration
- Purification by oxidation of organic impurities
- Offgas treatment to remove NO<sub>x</sub>

## Performance data.

|                    |                  |  |
|--------------------|------------------|--|
| Preconcentration   | 10–85%           | H <sub>2</sub> SO <sub>4</sub>           |
| High concentration | 75–96/97%        | H <sub>2</sub> SO <sub>4</sub>           |
| Capacity           | 1–1,000 tons/day | H <sub>2</sub> SO <sub>4</sub> (as 100%) |

Greater throughputs are possible by using two lines working in parallel. Spent acids often contain organic impurities, whose level has to be reduced to very low values for recycling. Acids with high-boiling organic impurities can be purified by the addition of an oxidizing agent. This purification reaction is integrated in the high concentration stage and takes place concurrently with the evaporation process. A separate purification unit can therefore be dispensed with. This combined treatment process was developed and perfected between 1980 and 1982 and has been successfully implemented at industrial scales.

## Preconcentration systems.

- Concentration range 10–85% H<sub>2</sub>SO<sub>4</sub>
- Evaporation under atmospheric pressure and vacuum
- Single or multistage designs
- Steam as heating medium
- Evaporator types: horizontal, falling film, forced circulation
- Minimum investment costs through optimized process control and selected construction materials
- Low energy consumption through multiple-effect designs
- Low operating costs due to automatic operation under minimum supervision

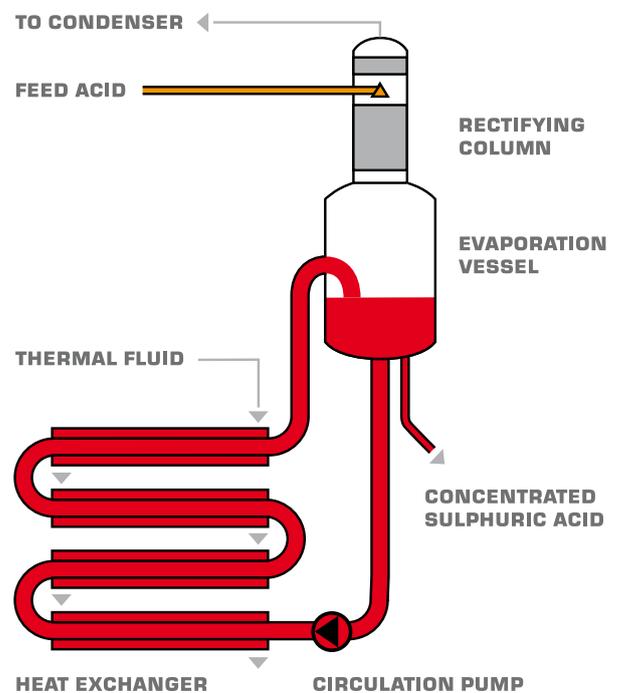


# SULPHURIC ACID: EXPERT IN HIGH CONCENTRATION.

## High concentration systems.

- Concentration range 75–96/97%  $\text{H}_2\text{SO}_4$
- Evaporation under vacuum
- Thermal fluid or steam as heating media
- Steam-heated horizontal evaporator or forced-circulation evaporator in tantalum for final concentrations up to 96%  $\text{H}_2\text{SO}_4$
- Glass-lined steel, forced-circulation evaporator heated by thermal fluid: a system developed and patented by Bertrams with special fail-safe controls for temperatures and process media, for final concentrations up to 96/97%  $\text{H}_2\text{SO}_4$
- Forced-circulation evaporator with integrated purification process for maximum possible decomposition of organics in the long reactor loop
- Excellent corrosion resistance thanks to careful selection of materials
- Avoidance of fouling and scaling due to very smooth glass linings
- Low operating costs: minimum supervision required as operation is automatic

## Basic concept of the final concentrator.



### **Heated vessel concentrators.**

- Suitable for small capacities, typically 1–8 tonnes/day H<sub>2</sub>SO<sub>4</sub> (as 100%)
- Concentration up to 96/97% H<sub>2</sub>SO<sub>4</sub> with use of chilled water
- Electrically heated
- Evaporation under high vacuum
- Single or multistage designs in combination with steam heated evaporators
- Minimum acid losses
- Very simple design with a minimum of system components and inexpensive controls
- Automatic operation under minimum supervision
- Modest spare parts inventory and low maintenance costs



### **Laboratory tests.**

Laboratory tests performed on “as is” spent acids enable an accurate assessment to be made of an individual acid’s characteristics when evaporated under industrial process conditions. They also make it possible to define the quality of the acid product that can be achieved. Bertrams Chemical Plants Ltd. can therefore offer an optimized concept for the proposed plant.

# MIXED ACID (H<sub>2</sub>SO<sub>4</sub>/HNO<sub>3</sub>) TREATMENT.

## Growing competence.

With the acquisition of PIC in 2014, Bertrams Chemical Plants has extended its portfolio in the design and supply of process plants involved in the manufacture of “nitro-derivatives” – from any type of explosive, both military and civil ones, to single and double-base propellants, from specialty nitro-derivatives to some inorganic nitrates, directly from their metals – as well as for the production of the 99% nitric acid itself. Having developed and commissioned multiple specialized treatment processes for spent sulphuric and nitric acid, the know-how and technology acquired enable us to provide safe and reliable plants meeting the latest technology standards.

## Processing of waste acids as an economic recycling to the nitrators.

Nitration syntheses apply so-called “mixed acid”, a mixture of concentrated nitric acid and sulphuric acid. Bertrams Chemical Plants Ltd offers a wide range of separation, purification and concentration process combinations for mixed acids of nitric and sulphuric acids, water and organics.

## The goal of the mixed acid treatment is to:

- Separate nitric and sulphuric acid for their recycling into upstream processes
- Concentrate nitric acid
- Decompose the organics
- Eliminate nitrous acid (HNO<sub>2</sub>)
- Provide safe treatment of explosive spent acids.

Below are the spent acids from nitration processes which can be handled by Bertrams Chemical Plants Ltd. in mixed acid treatment plants:

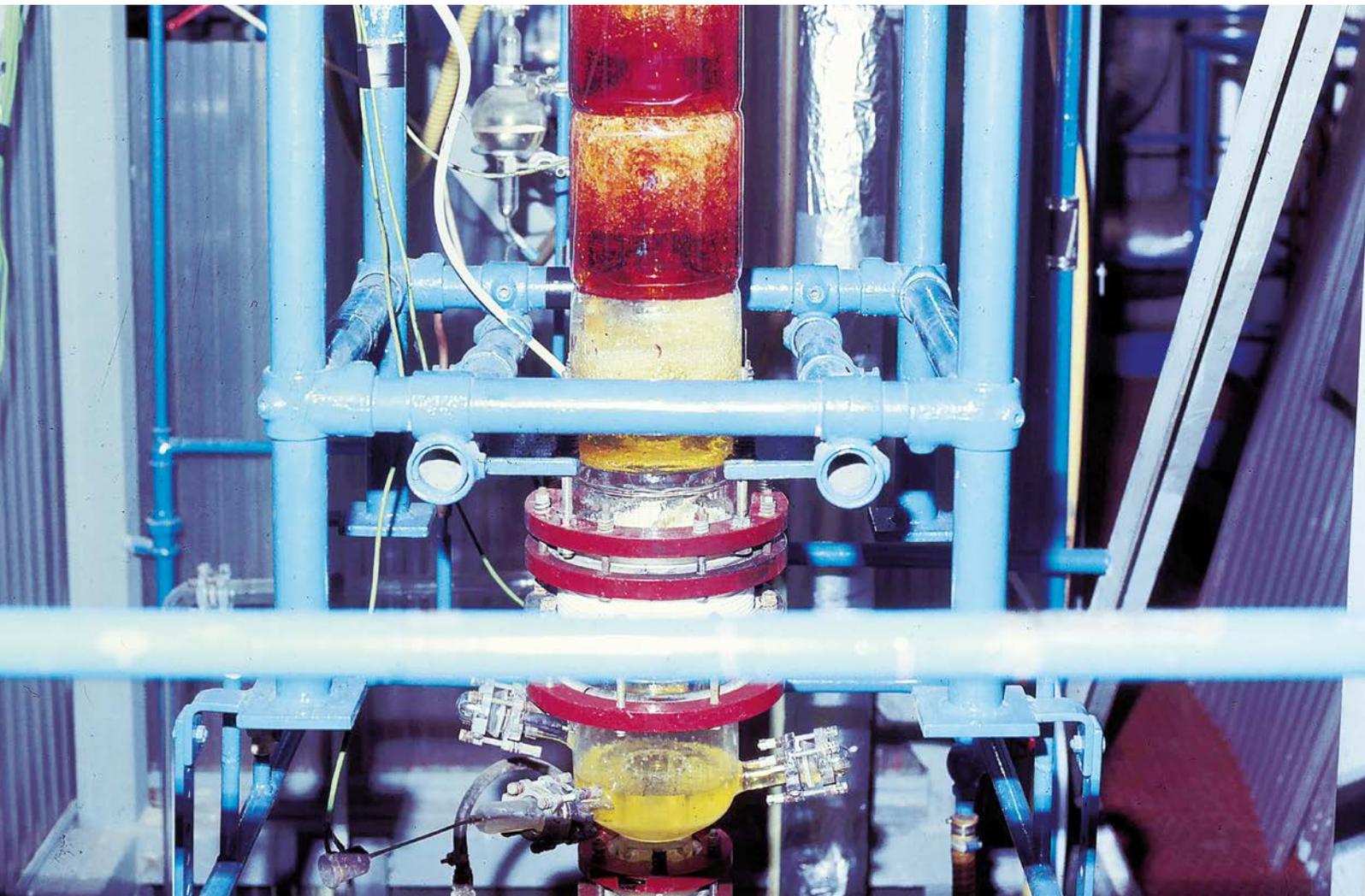
- Nitro ester (NC,NG, EGDN, PETN)
- Nitro-aromatics (i.e TNT, MNT, DNT, picric acid, NB)
- Nitroguanidine
- Nitroamine (RDX, HMX)

## Portfolio for various customer requirements.

The following are the process options in our portfolio:

- **Decomposition/stabilizing units** for explosive spent acids
- **Denitrating plants** for regeneration of the spent acid by denitration of the sulphuric acid and production of nitric acid and sulphuric acid solutions at high strength for recycling
- **Sulphuric acid concentration plants** up to 97% concentration
- **Nitric acid concentration units**
- **NO<sub>x</sub> absorption units**





#### **Decomposition system.**

- Pretreatment for stabilization of spent acids from the production of either NG (or other nitro-esters) or PETN for their safe handling
- Decomposition of organic compounds by several methods using thermal or catalytic decomposition depending on the spent acid to be treated
- In combination with a denitrating plant for acids from the production of NG, EGDN, PETN
- Tailor-made decomposer design
- Simple design with maximum heat recovery to reduce steam consumption
- Simple operation

#### **Denitrating plants.**

- Spent sulphuric acid from nitration processes or from the production of nitric acid contains various amounts of nitric acid ( $\text{HNO}_3$ ), nitrous acid ( $\text{HNO}_2$ ) and organic impurities. The denitration of sulphuric acid removes those components and recovers the impurities as nitric acid solution and  $\text{NO}_x$ , which can be converted into nitric acid in the  $\text{NO}_x$  absorption unit.
- Recovery of nitric acid at 98.5–99%  $\text{HNO}_3$
- Minimal amount of nitrous in the concentrated nitric acid
- Highly efficient absorption of  $\text{NO}_x$
- Recovery of spent sulphuric acid with minimum nitric acid content which can be further concentrated to desired concentration
- Choice of excellent corrosion-resistant materials for maximum safety and long-lasting plant life

# ADVANCED TECHNOLOGY FOR HNO<sub>3</sub> CONCENTRATION.

## Unit operation.

Bertrams Chemical Plants Ltd. offers various concentration processes for aqueous nitric acid solutions, as both “virgin” and “spent” nitric acids (even “mixed” with sulphuric acid). The following unit operations are applied to achieve the required concentration:

- Preconcentration by rectification without use of extractive agents to reach concentration a little below the azeotrope which is 68,3% HNO<sub>3</sub>
- High concentration system in an extractive distillation system using as extractive agent either sulphuric acid or magnesium nitrate melt.

NO<sub>x</sub> absorption units as well as sulphuric acid concentration systems complete the technology portfolio.

## Performance data.

|                    |                |                         |
|--------------------|----------------|-------------------------|
| Preconcentration   | up to 66–67%   | HNO <sub>3</sub>        |
| High concentration | 50%–99%        | HNO <sub>3</sub>        |
| Capacity           | 2–160 tons/day | HNO <sub>3</sub> (100%) |

Much greater throughputs can be achieved on twin or multiple parallel trains for the largest capacities. Bertrams Chemical Plants Ltd.’s plants are very adaptable to varying specifications of the nitric acid feed.



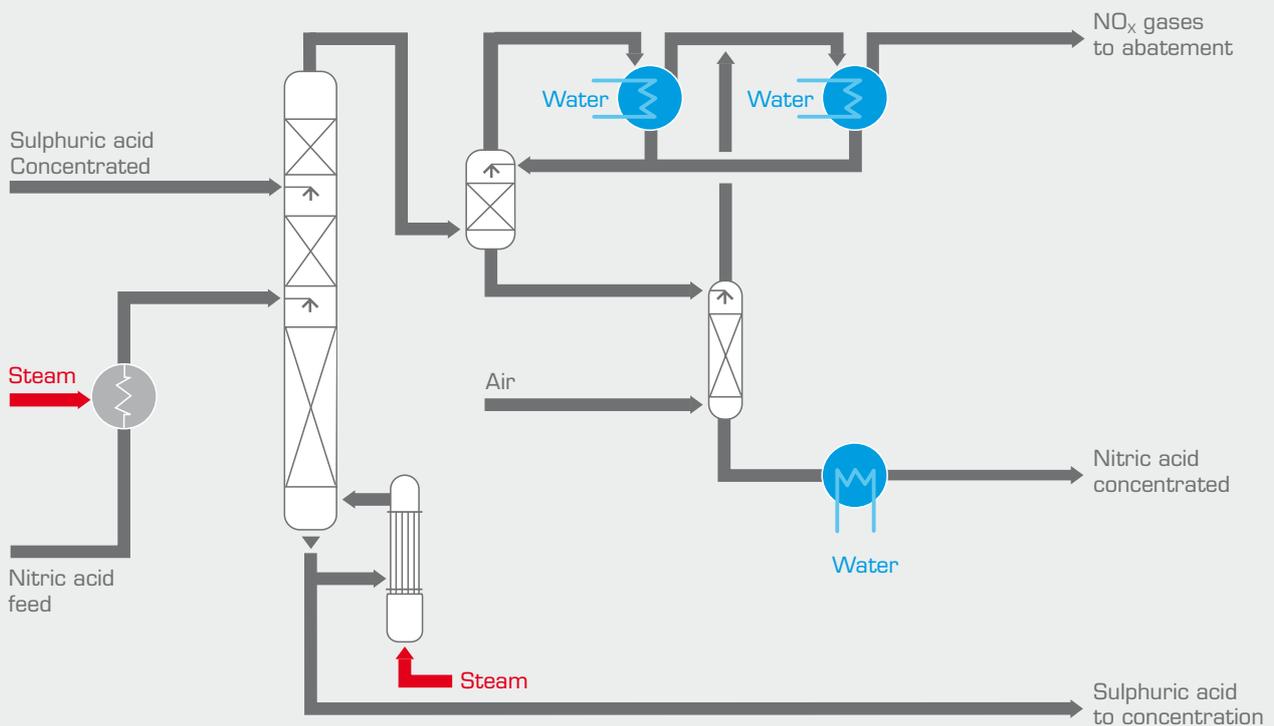
### High concentration nitric acid plants.

Nitric acid is concentrated up to 99%  $\text{HNO}_3$  in an extractive distillation column by means of concentrated sulphuric acid as extractive agent. Sulphuric acid is added to overcome the azeotrope nitric acid-water, modifying its behavior so that nitric acid at high strength can be obtained by distillation. The dilute sulphuric acid stream (typically at 68–71%) coming from the bottom of the extractive distillation column can either be sent for further use, for example in fertilizer production, or can be re-concentrated back in a sulphuric acid concentration plant.  $\text{NO}_x$  containing gases resulting from the

thermal decomposition of  $\text{HNO}_3$  and  $\text{HNO}_2$  are recovered as nitric acid using the  $\text{NO}_x$  absorption unit, resulting in a total nitric acid yield higher than 99.9%. Equipments are made of highly resistant construction materials such as borosilicate glass 3.3, glass lined steel and tantalum for the reboilers.

The magnesium nitrate melt route is limited to the production of concentrated nitric acid from “virgin” acid. Re-concentration of the magnesium nitrate solution in stainless steel equipments make this an attractive economical route from an operating and investment point of view.

## NITRIC ACID CONCENTRATION FLOWSHEET.



# ENVIRONMENTAL ENGINEERING: NO<sub>x</sub> ABSORPTION UNITS.

## **NO<sub>x</sub> a toxic pollutant but a valuable gas to be recovered as nitric acid.**

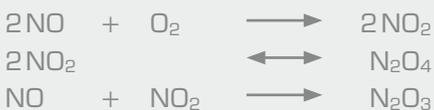
The partial degradation of nitric acid (HNO<sub>3</sub>) used in the nitration process produces emissions often sent to the atmosphere, causing extremely high levels of air pollution. Our technology for the abatement of NO<sub>x</sub> provides air pollution control that is fully integrated with high efficiency nitric acid recovery.

## **The NO<sub>x</sub> absorption process anticipates:**

- The operation under atmospheric pressure or under pressure (up to 6 barg) depending on the NO<sub>x</sub> content of the gases and on the environmental standards to be fulfilled
- A single or multiple effect highly efficient cooled tray absorption tower
- The oxidation/scrubbing of NO<sub>x</sub> by oxidant as air or as hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>)
- The purification of air, complying with statutory emission regulations worldwide.

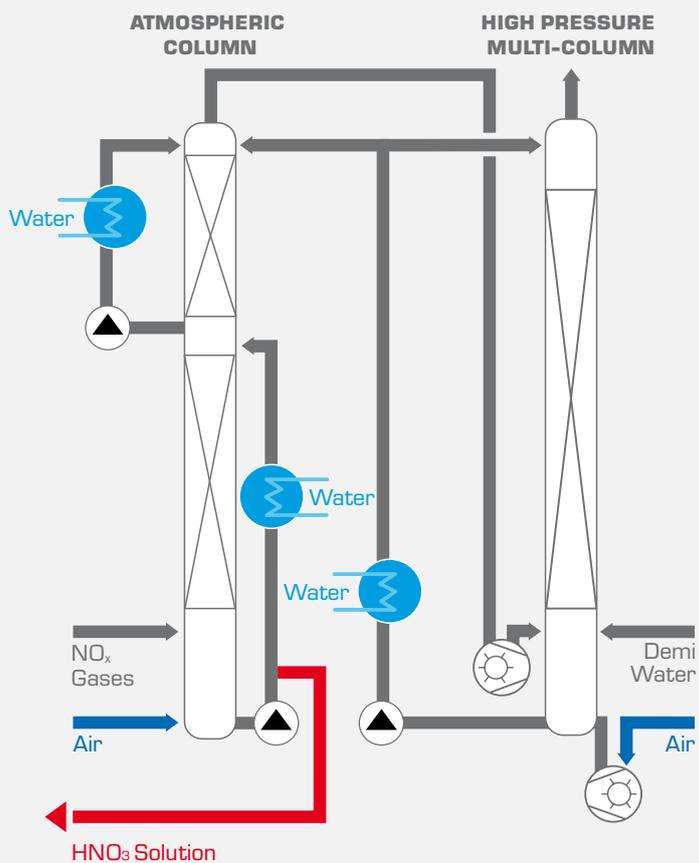
## REACTION MECHANISM.

### GAS PHASE



### LIQUID PHASE





### Technology for clean air:

Simultaneous gas and liquid reactions occur in the pressure absorption column in accordance with the reaction mechanism above. Heat removal due to the exothermic character of the reaction is taken care of by specially designed cooled trays.

With Bertrams Chemical Plants' design of the NO<sub>x</sub> absorption system, using only air and water, NO<sub>x</sub> is recovered in the form of nitric acid solution at 55–60% wt. For gases with high NO<sub>x</sub> content, a high pressure multicolumn system using hydrogen peroxide as an oxygen donor is used to achieve strong environmental standards.

The applications are large and include all industries processing by nitration and emitting atmospheric NO<sub>x</sub> polluted air such as explosives (military and civil), metal pickling, gold and valuable metal processing or manufacture of special batteries.

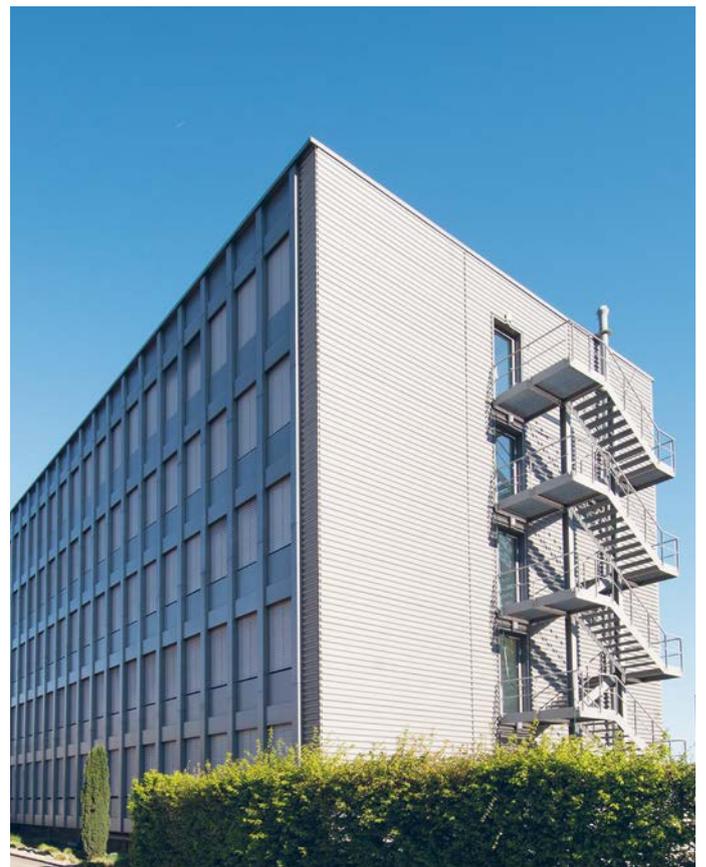
# COMPREHENSIVE RANGE OF SERVICES.

**Right from the very first meeting to sound out ideas you profit from our all-embracing expertise, our quality standards and a comprehensive service program.**

- BASIC ENGINEERING WITH KEY COMPONENTS AND PROCESS GUARANTEE
- DETAIL ENGINEERING
- PROCUREMENT SERVICE
- SKID-MOUNTED SYSTEMS
- TURNKEY PLANTS
- ON-SITE TRAINING
- AFTER-SALES SERVICE
- LONG-TERM GUARANTEE FOR GENUINE SPARE PARTS

## **Constructive relationships with customers.**

The atmosphere of constructive cooperation that we enjoy with our customers results time and again in suggestions being made to improve the efficiency and cost-effectiveness of the plant. If a plant expansion or modification is under discussion, we are also available to help our customers and put forward solutions that are optimized to suit user needs.



**RELIABLE:**

Security and partnership for the most diverse requirements in the chemical industry.

**LEADING THE WAY:**

Plants for the concentration and/or solidification of alkalis in the chlor-alkali industry.

**EFFICIENT:**

Recovery plants for the concentration, purification and treatment of sulphuric acid and nitric acid ( $H_2SO_4/HNO_3$ ).

**COST-EFFECTIVE:**

Wet oxidation units for the treatment of waste-waters that are not readily biodegradable.

**CUSTOMER ORIENTED:**

We sell technology – we deliver satisfaction.

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