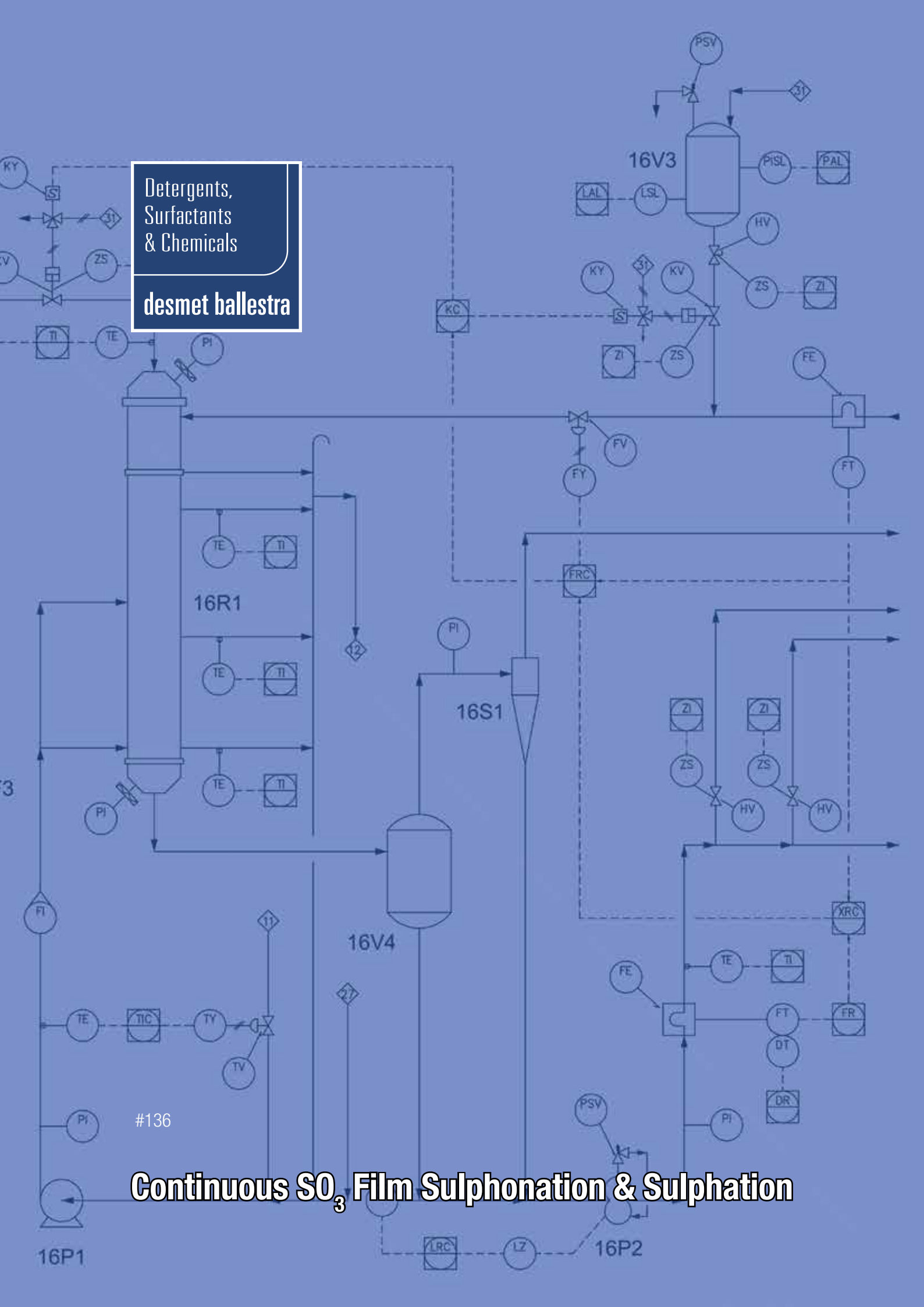


Detergents,  
Surfactants  
& Chemicals

desmet ballestra



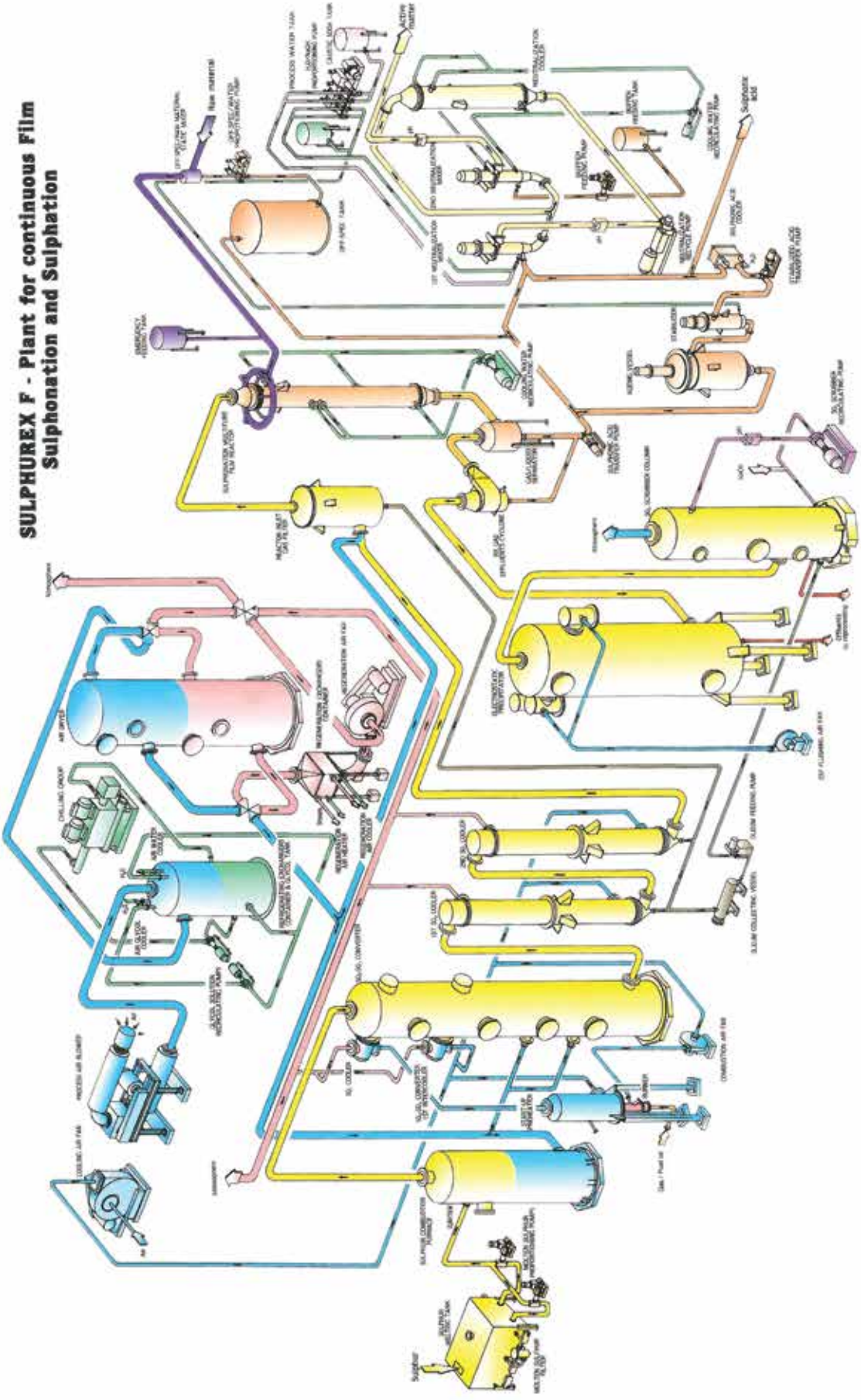
## Continuous SO<sub>3</sub> Film Sulphonation & Sulphation

#136

16P1

16P2

**SULPHUREX F - Plant for continuous Film Sulphonation and Sulphation**



# Introduction

Since its foundation in 1960, Desmet Ballestra SpA is the world leader in the design and supply of Chemical plants for Surfactants and Detergents production.

Desmet Ballestra is the technology supplier of all major surfactants and detergents manufacturers worldwide and in its more than 55 years activity has implemented more than 1800 plants in over 120 countries.

Desmet Ballestra's **film sulphonation technology** presents outstanding advantages over the competing processes, namely:

- excellent performances in terms of product quality specially when sensible raw materials are processed
- exceptionally good conversion rate and colour with maximum achievable absorption of SO<sub>3</sub> gas
- simpler construction which entails easier maintenance
- lower operating pressure
- no external cooling requirements and no need of chilling water equipment
- extra low energy consumption

Desmet Ballestra has implemented the world's largest sulphonation plants (24000 Kg/h as 100% active surfactant) and is constantly optimizing its process through the continuous work of the R&D Centre.

Particularly Desmet Ballestra has developed proprietary Know How and processes for surfactant upgrading downstream the sulphonation process, i.e.: Dioxane removal and Drying process for pure dry surfactant production.

Desmet Ballestra can offer standard plant design for capacities ranging from 1 to 8 ton/hr based on a single production line and customized design for larger capacities.

The mission of Desmet Ballestra is to establish consistent and long term relationships with its customers offering a full range of services and technical support so to enable them to face the increasing challenges of the today's surfactant industry.

*To this aim, Desmet Ballestra is in the position to offer to its customers:*

- Availability of modern and efficient pilot plant facilities where they can test new raw materials, verify performances and easily apply the findings to industrial scale plants with minimum costs
- A very efficient Spare Parts department structured to answer in real time to the customer needs, minimizing maintenance cost and production losses
- A dedicated Technical Assistance department structured to assist customers in troubleshooting, evaluation of plant performances, preparation of plant maintenance plans, personnel training and studies for plant revamping and technological updating



The integration of Ballestra in the Desmet Ballestra Group has substantially enhanced the Ballestra presence in the world markets and its capability to offer cost competitive plants with a strong local back-up management.

**Resources and capabilities**

- 200 employees and full time consultants
- R&D Centre with laboratories and full scale pilot plants
- ISO 9000 Certification
- Up to 250,000 engineering hours per year
- 3D computer design
- Capability to supply all the range of services from feasibility studies to turn-key projects including skid-mounted execution
- Global sourcing of equipment and materials
- Project financing



*Sulphonation Plant Panoramic Views*





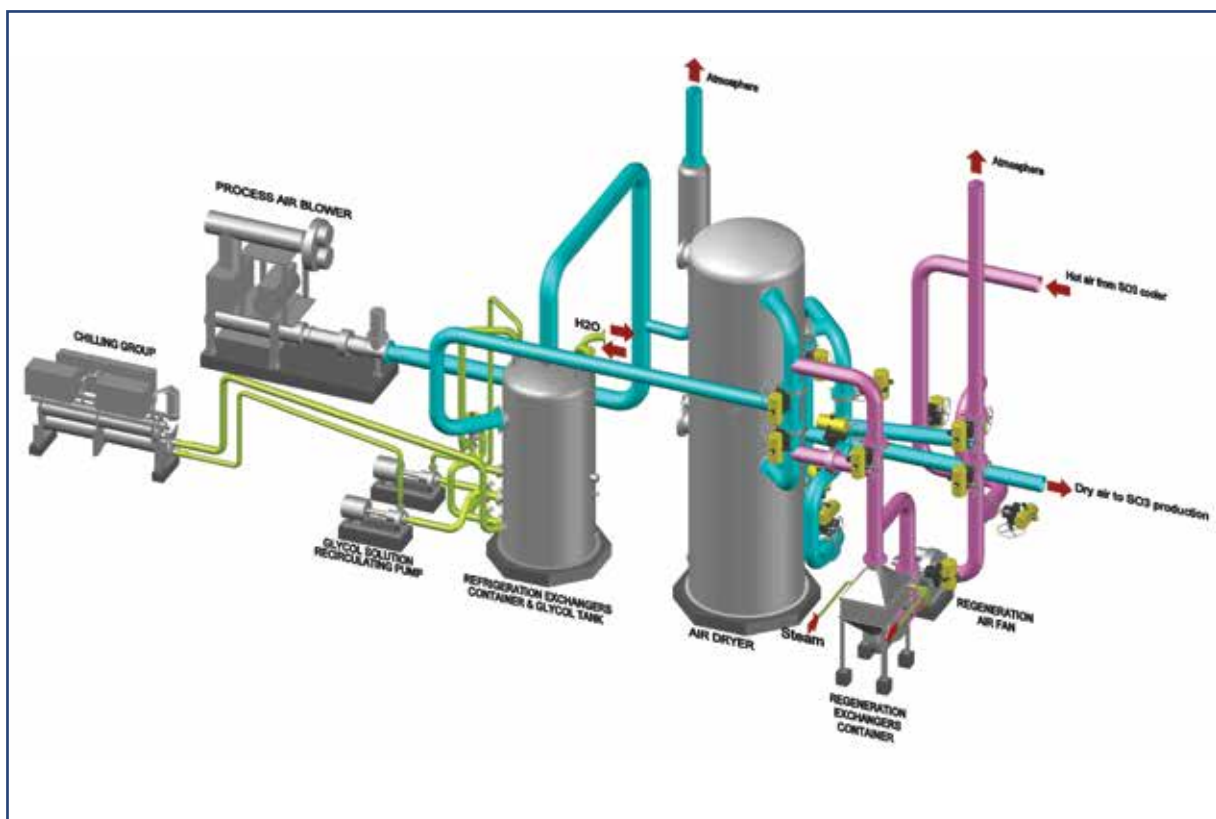
# Process Air Drying

The atmospheric air is compressed up to 0,7 barg as requested by the downstream sulphonation process. The compressed air is dried

by refrigeration and condensation of the water contained in the atmospheric air and subsequent absorption on a dessicant bed.

*Main features of the unit:*

- ➔ Low operating pressure (0,7 barg max.)
- ➔ Process Air Dew Point lower than -70°C
- ➔ Fully automatic regeneration of absorbent medium (silicagel/ $\text{Al}_2\text{O}_3$ )
- ➔ Low energy consumption due to the recovery of the hot air from  $\text{SO}_3$  production unit for absorbent medium regeneration
- ➔ High reliability and efficiency







*Air Drying and regeneration circuit*

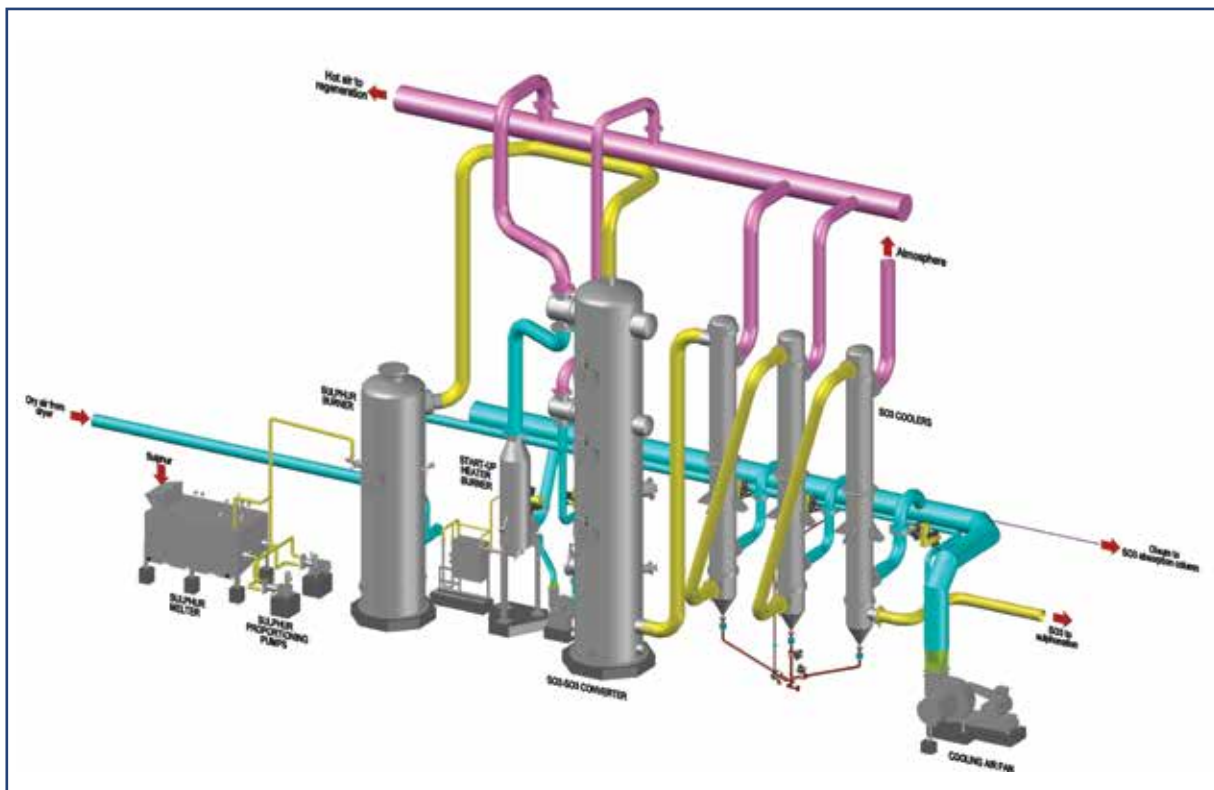
# SO<sub>3</sub> Production

Liquid sulphur is burned with the process air in a special furnace to produce a gas containing 7% by volume of SO<sub>2</sub>. The SO<sub>2</sub> is then converted to SO<sub>3</sub> in a special conversion tower equipped with

4 Vanadium based catalyst beds and intercoolers to optimize kinetically and thermodynamically the reaction yield. The SO<sub>3</sub> gas is finally cooled to 50-60°C as requested by the sulphonation process.

*Main features of the unit:*

- Very high reaction yield : 98,5% guaranteed and possibility to achieve higher yield with the use of special Cesium catalyst
- High reliability, safety and long plant lifetime due to the use of air as cooling medium instead of water to minimize corrosion risks
- Special design of sulphur furnace ensuring high operating reliability and easiness
- High energetic efficiency thanks to the use of hot cooling air for silicagel regeneration in the Air Drying Unit
- Very compact design of the conversion tower with internal intercoolers
- Reduced plant start-up time due to a special sulphur furnace which does not require preheating







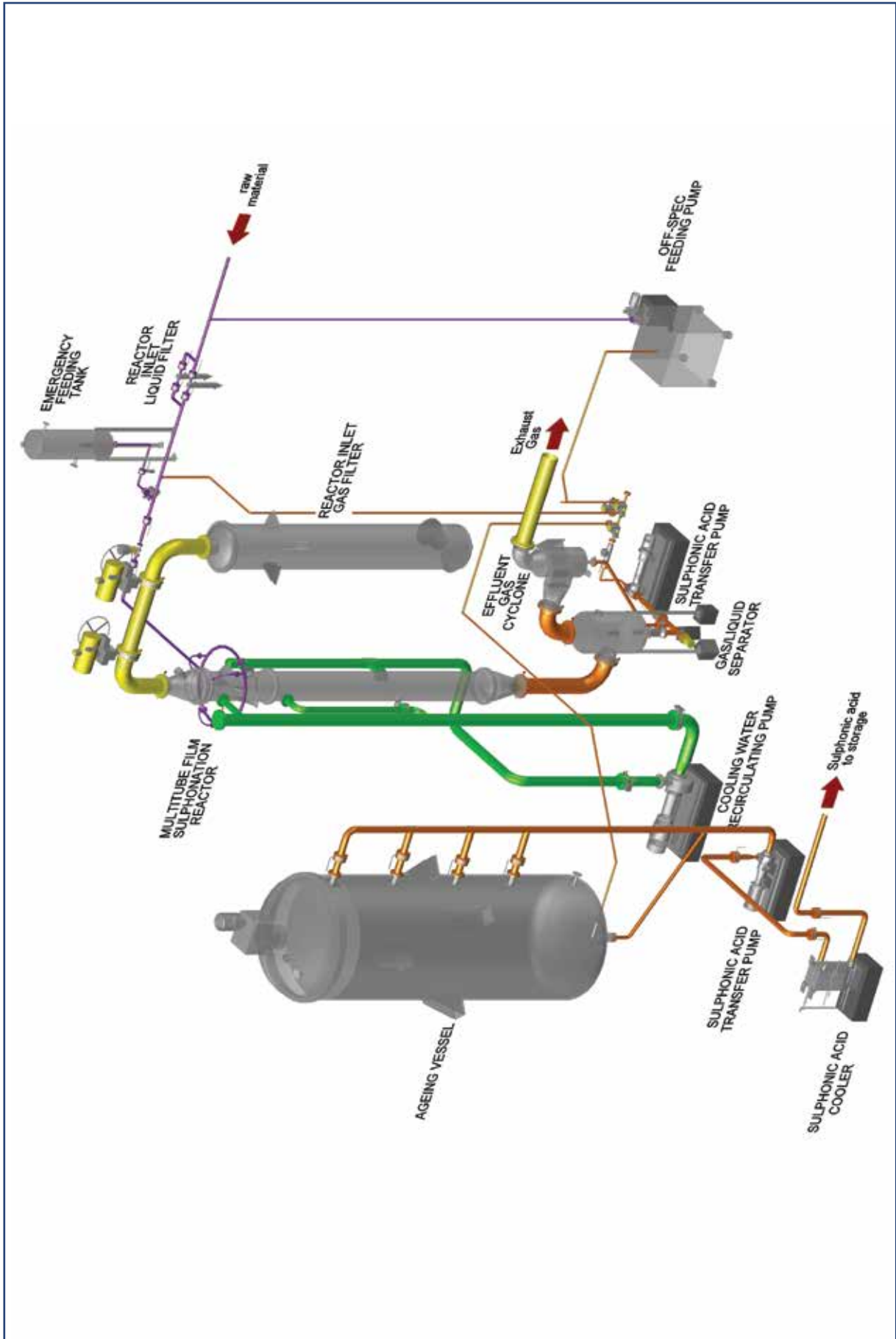
*SO<sub>2</sub>-SO<sub>3</sub> Production Section: Panoramic View*



*SO<sub>2</sub>-SO<sub>3</sub> Converter and SO<sub>3</sub> coolers*



*Sulphur burner*



# Sulphonation Unit

The SO<sub>3</sub> gas outcoming the SO<sub>3</sub> production unit is filtered by a special high efficiency brink filter to eliminate any trace of carried-over oleum. Then is fed to the top of the Multitube Falling Film Sulphonation reactor (MTFR) where also the organic raw material is fed under mass flow control. The reaction of SO<sub>3</sub> gas with the organic raw material takes place in the MTFR.

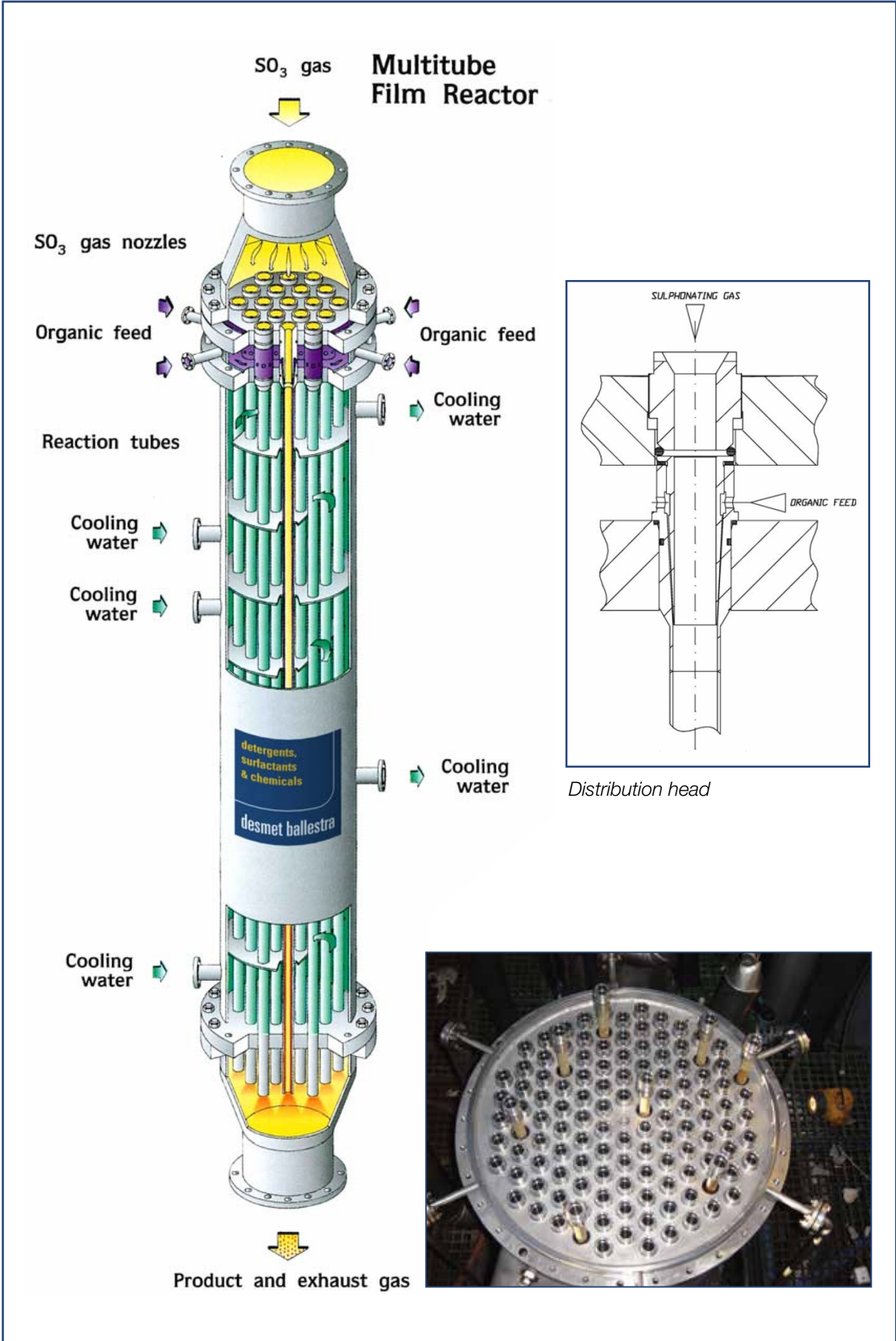
At the outlet of the reactor, the gas/liquid mixture is separated in a special gas/liquid separator and in a downstream gas cyclone. The sulphonic acid is sent to a special ageing/stabilizing unit when Linear Alkylbenzene is processed, or straight to the neutralization unit when Fatty Alcohol or Ethoxylated Fatty Alcohol are processed.

## *Main features of the unit:*

- ➔ Product quality meeting the highest international standards
- ➔ High operating flexibility thanks to the capability to process any raw material used in detergent and surfactant industry
- ➔ High reliability and operation easiness
- ➔ Automatic in-line control of sulphonation degree
- ➔ Low energy demand thanks to the low pressure drop of the MTFR
- ➔ Optimized design achieved through over 300 industrial Film Sulphonation plants implemented and operative worldwide









*Multitube Falling Film Reactor*

### Main features of the Desmet Ballestra's Multitube Film Sulphonation reactor

- ➔ Outstanding performances in terms of product quality
- ➔ Very high heat transfer efficiency that enables an accurate control of the temperature profile in the reactor
- ➔ Low pressure drop (0,3 barg average) resulting in a reduced energy consumption of the whole sulphonation plant
- ➔ Once through design. The sulphonation reaction is accomplished within the residence time of the reactants in the reactor (less than 1 minute) without need of external recycling loop, allowing the production of low colour and low dioxane surfactants. Additionally, this enables the automatic in-line control of the sulphonation degree through the reactor
- ➔ No external cooling requirements and no need of chilling water equipment
- ➔ High flexibility with possibility of product flying change-over with minimum product contamination
- ➔ Operation easiness
- ➔ Easy scale-up of the reactor as all distribution heads and reaction tubes are identical. Scale-up is achieved simply increasing the number of reaction tubes with practically no limitations on reactor size and capacity
- ➔ High reliability and longer lifetime thanks to the use of sophisticated corrosion resistant alloys in critical parts

### PRODUCTS CHARACTERISTICS

RAW MATERIALS	A.M. Conc. %	% Free Oil (*)	% SO <sub>4</sub> (*)	°Klett (**)	1,4 dioxane (*)
Linear Dodecylbenzene	60-75	0,8-1,2	0,5-1,2	10-25	-
Natural Lauryl Alcohol C12-C14 or C12-C18	30-75	0,8-1,5	0,5-1,0	2-5	-
Synthetic Alcohol C13-C15	30-75	0,8-1,5	0,5-1,0	5-10	-
Natural or Synthetic Fatty Alcohol C16-C18	30-65	1,5-2,5	0,7-1,2	20-40	-
Natural or Synthetic Alcohol Ethoxylated with 2 or 3 EO	30-75	1,0-1,5	0,5-1,0	10-15	30 ppm (***)
Alpha-Olefins	30-75	1,2-2,0	0,7-1,2	30-50	-
Methylesters	30-75	1,8-2,5	2-2,5	30 (bleached)	-



(\*) On 100% Active Matter

(\*\*) 5% A.M. solution, 40 mm cell n. 42 filter, unbleached, unless otherwise specified

(\*\*\*) At reactor outlet



# Neutralization Unit

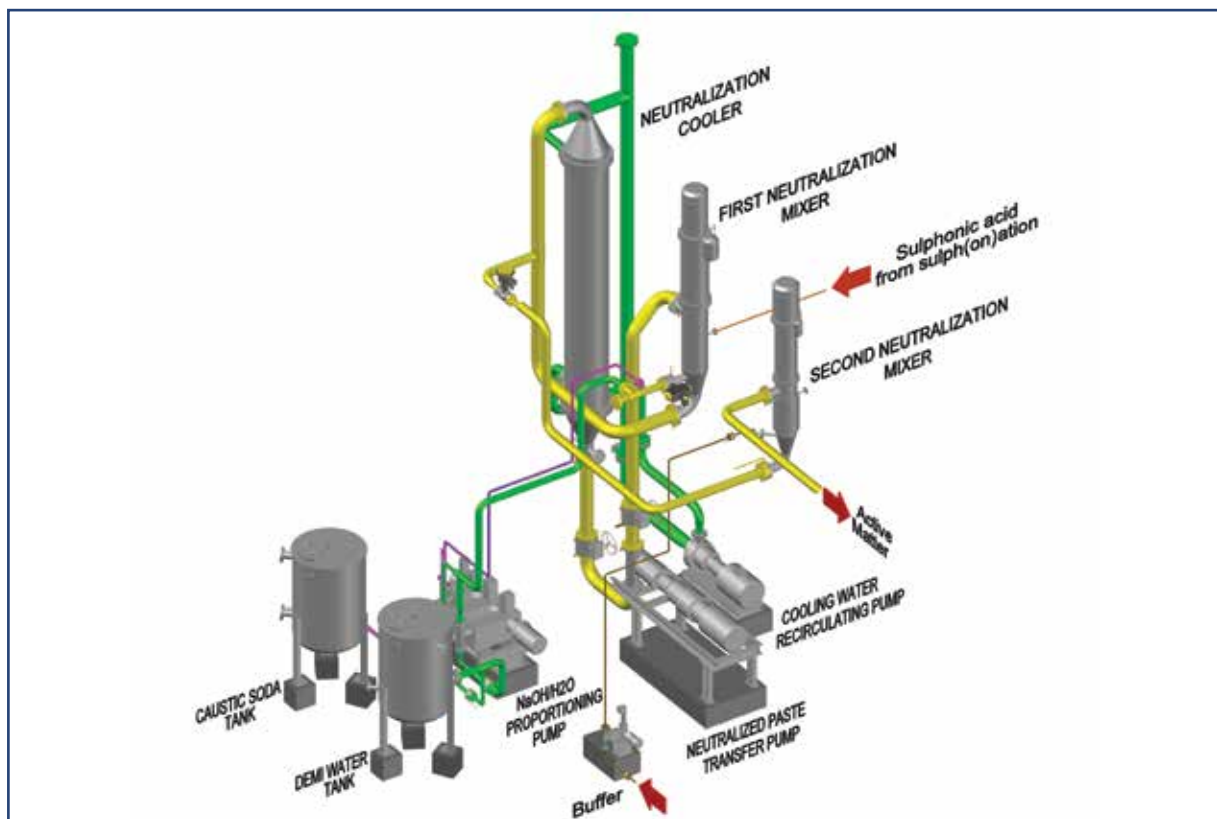
The sulphonic acid outcoming the sulphonation unit is neutralized with caustic soda or an alternative alkaline agent (i.e.: Ammonia) in a special **Double Step Neutralization Unit** or, specifically for Ether Sulphates processing, in a **Vacuum Neutralization Unit** to yield high active neutral paste (70-75%

concentration). In both cases the neutralization process is carried out at high pH values to guarantee the necessary chemical stability to the reaction mass. Final product pH value is adjusted in-line by addition of a buffering agent in a final mixer.

## A. Double Step Neutralization Units

*The main features of the proposed unit are:*

- High shear mixers of Desmet Ballestra proprietary design, ensuring intimate mixing of reactants even at high concentration conditions
- Accurate control of the temperature profile in the loop thanks to the high recycling rate and the low pressure drop cooler so to prevent any risk of product degradation
- Easy in-line transition from low concentration (27-30%) to high concentration (70-75%) paste production
- Accurate control of free alkalinity





*Skid-mounted Double Step Neutralization unit*

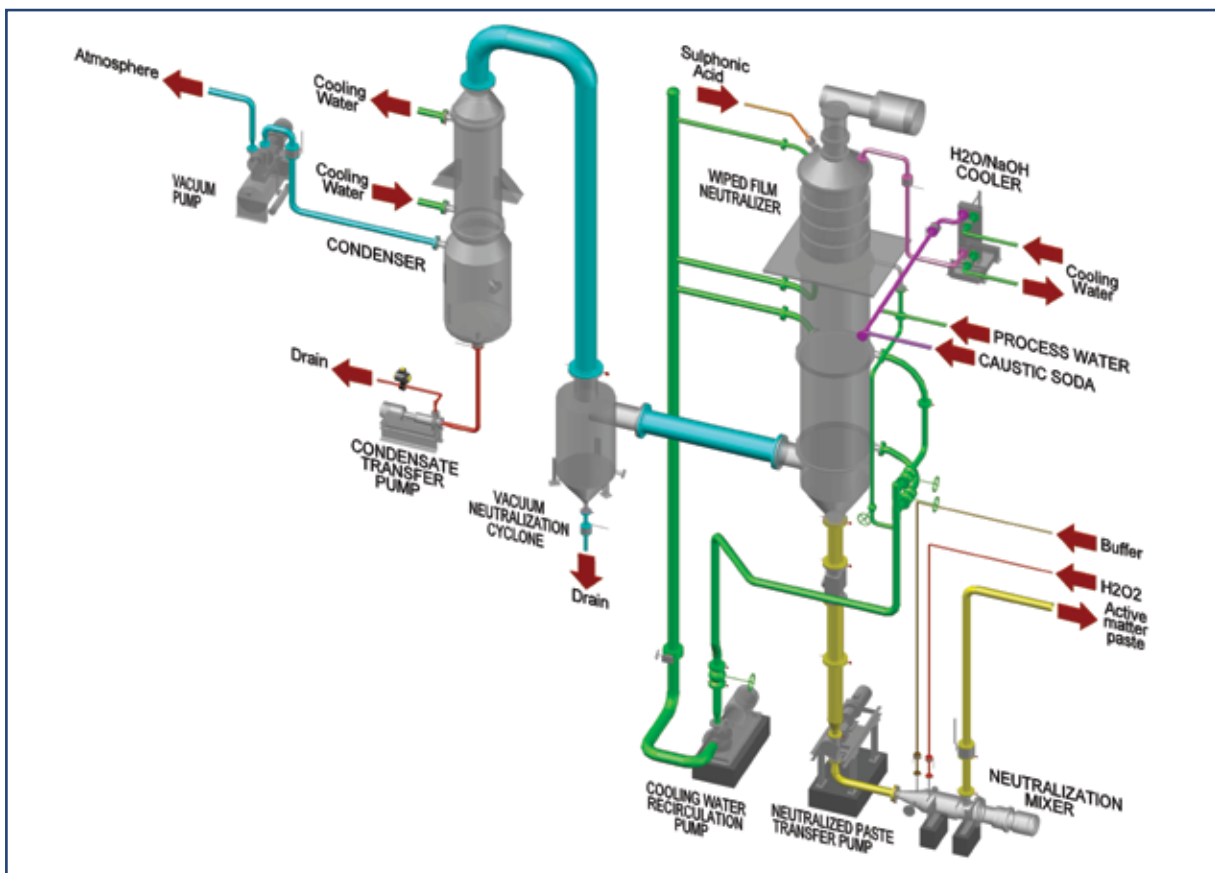
## B. Vacuum Neutralization Unit

Although the «Loop type» Neutralization is suitable for the production of top-quality surfactants, a further improvement in terms of dioxane removal

process flexibility, reduction of energy demand and product quality consistency is obtainable by the «Vacuum type» Neutralization.

*The main features of the proposed unit are:*

- ➔ Once-through operation, requiring minimum hold-up volume in the Neutralizer and avoiding unit filling with pre-neutralized paste at each change-over
- ➔ Efficient Dioxane removal from finished product by evaporation of the azeotropic mixture water/dioxane
- ➔ Possibility to achieve dioxane content in finished product (SLES) below 10 ppm when coupled with the Desmet Ballestra MTRF
- ➔ Product deaeration to increase density and improve appearance (transparency)
- ➔ Mild operating conditions with accurate control of temperature profile and minimum residence time of the product at high temperature so to prevent any risk of hydrolysis
- ➔ No steam/energy consumption as dioxane stripping is achieved using the neutralization heat of reaction
- ➔ High flexibility: capability to process any kind of raw material/product with possibility to easily switch from one raw material to a different one with minimum product contamination







*Vacuum Neutralization unit*



*Vacuum Neutralization unit*



# Exhaust Gas Treatment Unit

The exhaust gas exiting the sulphonation reactors is processed to reduce the level of pollutant to values widely accepted by the most stringent international standards on emissions to atmosphere.

The maximum content of the polluting agents guaranteed at plant stack is the following:

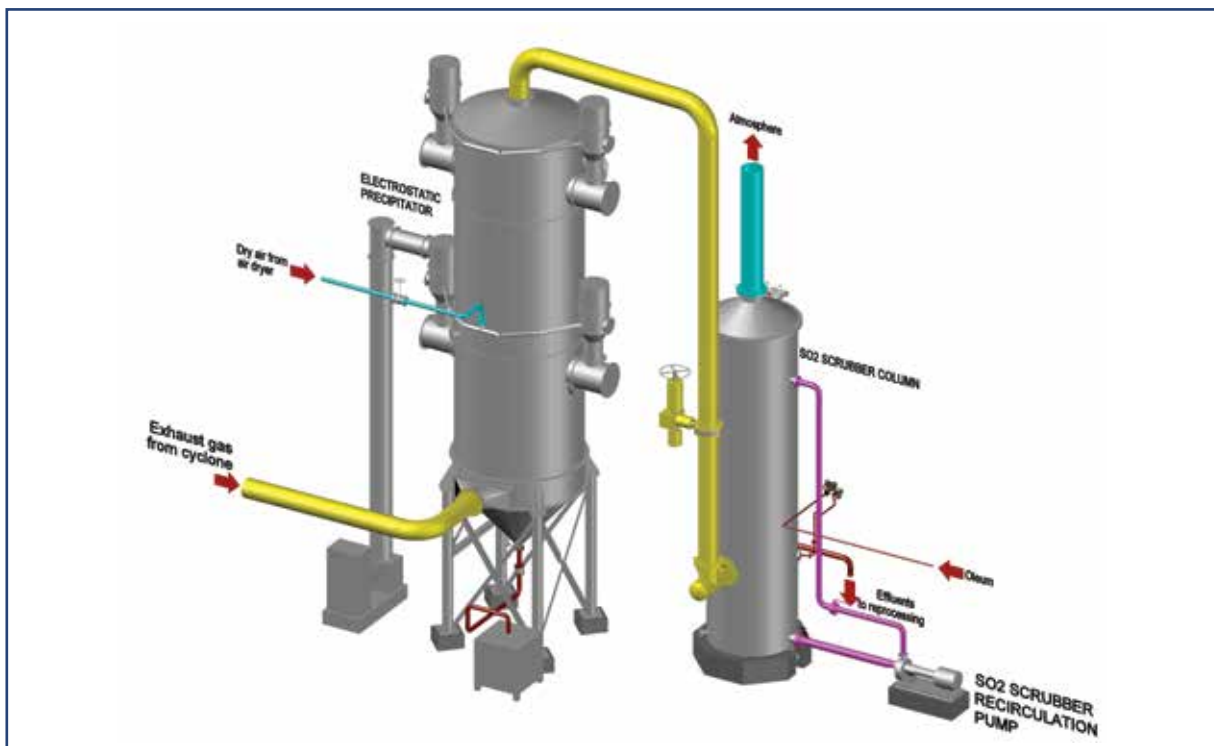
- Organic mist : 25 mg/m<sup>2</sup>
- SO<sub>3</sub> : 15 ppm (vol.)
- SO<sub>2</sub> : 5 ppm (vol.)

The exhaust gas undergoes 2 subsequent steps of treatment before being released to the atmosphere: reduction of organic mist content and final washing with a caustic soda solution to reduce the amount of free SO<sub>2</sub> discharged. This latter step is carried out in a suitable SO<sub>2</sub> scrubber, while for the elimination of the organic mist Desmet Ballestra can offer 2 alternative technological solutions having the same efficiency:

- Electrostatic precipitator (ESP)
- Wet brink filter (NEGT)



*Exhaust Gas Treatment unit based on ESP and SO<sub>2</sub> Scrubber*





### New Generation Electrostatic Precipitator (ESP)

- The latest generation ESP is characterized by:
- fixed electrode rods, granting much longer operation and no risk of rupture
  - upper and lower fixation of the electrode rods, completely avoiding the maintenance problems related to the traditional counter-weights and eliminating the risk of swinging
  - round-shaped tubes bundle, designed to ensure the proper gas speed and having higher tube thickness, so ensuring longer life time
  - integrated blowers for upper insulators flushing, directly installed on top of the machine
  - lower insulators flushing by means of dry process air, so preventing any risk of moisture condensation
  - high voltage transformer, able to work up to 60 kV, providing higher efficiency
  - electronic controller, able to select the best operating conditions in terms of voltage according to the detected electric field



### NEG.T.

The new unit is based on a special Multicartridge High Efficiency brink filter constantly washed with fresh organic raw material.

This new system, while guaranteeing the same performances of the ESP (in terms of cleanness of the gas at the stack outlet), allows the complete recovery of the sulphonic acid carried over by the exhaust gas.

As additional advantage, the NEG.T system enables the recovery, as finished product, of the organic drippings, otherwise normally wasted. For this reason, the system is optimized for processing LABS and MES. The cost related to the disposal of the ESP drippings, which constitutes a problem of increasing importance for the surfactant manufacturers is therefore totally eliminated. This factor constitutes a non-marginal improvement of the whole process economics.

# Active Matter Drying

The unit has been developed with the aim to produce dry anionic surfactants in solid form starting from 70-75% paste, preventing product degradation due to overheating.

The unit is based on the use of the specifically designed vertical wiped film evaporator, characterized by:

- High efficiency in drying with minimum delta T between heating medium and process fluid
- Short and narrowly distributed residence time
- High product homogeneity
- No side reactions
- High achievable dryness of finished product (typical value of residual moisture is 2-3%)
- Easy operation (start-up/shut-down product change-over in few minutes, thanks to minimized residence time)
- High efficiency / reliability of mechanical equipment and process control
- Optimized thermal/energy efficiency

## **Needles:**

- Dry matter content : 97 – 98%
- Bulk density : 500 ± 50 g/l
- Dimensions : Ø : 0,5 ÷ 1,0 mm



In order to preserve the quality of the end product, it is crucial to avoid any build-up of material on the walls of the evaporator. This is prevented by the scraping action assured by the internal mechanical features of the evaporator, consisting of a rotating shaft holding a series of special blades, supported by appropriate frames.

The rotating blades provide for product distribution over a wide surface of the evaporator, as well as for removal of dry material from the walls of the same.

The unit is suitable to produce dry anionic surfactants in needles or powder form having the following characteristics:

## **Powder:**

- Dry matter content: up to 98%
- Bulk density : 550 +/- 50 g/l
- Particle size : 90% in the range 0,1 ÷ 1 mm





*Equipment for Active Matter Drying*





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# Desmet Ballestra

## Detergents, Surfactants & Chemicals



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