# Multi-Purpose Pilot Units for Supercritical Fluid Extraction of Liquids and Solids

High-pressure thermocouple for internal temperature measurement

Fluid cyclone with heating jacket (PID temperature control)

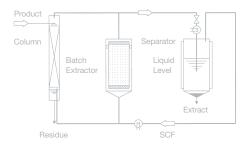
Collecting basket for extract fraction (incl. cooling jacket)

Sampling valve

Lifting device for collecting basket



Option «Fractionated Separation» with highperformance fluid cyclone. Collecting basket is equipped with a lifting device for easy handling.



Simplified process flow diagram for multi-purpose supercritical extraction pilot unit with closed  $\mbox{CO}_2$  cycle.

### Standard Design

Max. operating pressure	300 bar	500, 700 bar
Max. operating temperature	80°C	120, 150, 200°C
CO <sub>2</sub> flow	18 l/h	10, 30, 50, 100 l/h
Extractor capacity	1 litre	2, 4, 6, 10, 20 litres
Column interior diameter	Ø 38 mm	Ø 50, 65, 90 mm
Column internal length	2 m	3, 4, 5 m
Liquid capacity	2 l/h	4, 10, 18 l/h

Options

## Applications with solid raw material

- Production of natural extracts, e.g. hops, caffeine, spices
- Production of active agents for pharmaceuticals and cosmetics
- Degreasing of catalysts, microchips, medical implants
- Extraction of monomers from polymers
- Production of essential oils from blossoms, leaves and roots
- Regeneration of molecular sieves
- Decontamination of soils
- Production of natural colours e.g. oleoresins, carotene, bixins
- Conservation of antique books or wooden sculptures

## Applications with liquid raw material

- Fractionated separation of oils and waxes
- Separation of polyunsaturated fatty acids (PUFAs)
- Removal of cholesterol from dairy products
- Refining of lecithin
- Non-alcoholic wine and beer

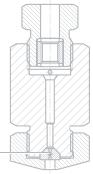
## **Related applications**

- Impregnation of textiles and wood products
- Dying of textile fibres
- Tobacco expansion



Multi-purpose SFE pilot unit for solid and liquid raw materials built for 500 bar max. operating conditions designed for research (Russia). The system includes the options fractionated separation with fluid cyclone, modifier system and Coriolis mass flowmeter.

# Multi-Purpose Pilot Units for Micronization and Spray Drying (RESS/GAS)



Diamond nozzle

Nozzle holder with diamond nozzle. Diamond nozzles are easily exchangeable. Orifice range 15–300 µm.



Spray generated by diamond nozzle (picture courtesy of Fraunhofer Institute, Germany)

### RESS

Rapid Expansion of Supercritical Solutions: An active agent is dissolved in a supercritical solvent and expanded through a spray nozzle. Solvent power is lost rapidly and solid particles fall out.

## GAS

Gas Anti-Solvent Recrystallization: An active agent is dissolved using a conventional liquid solvent and sprayed through a spray nozzle together with a highpressure gas. The gas decreases the solvent power and extracts the solvent during the expansion. Small particles are generated.

#### **Standard Design**

Max. operating pressure	300 k
Max. operating temperature	80°C
CO <sub>2</sub> flow	18 l/ŀ
Column interior diameter	Ø 90
Column internal length	1 m

300 bar 80°C 18 l/h Ø 90 mm 1 m **Options** 500 bar 120, 150, 200°C 10, 30, 50, 100 l/h Ø 60, 110, 160 mm 0.4, 2 m

High-pressure micronization (RESS/GAS) is used to generate very fine and uniform powders or well defined geometries of solid particles by expansion of a highpressure fluid.

## Advantages

- Production of fine powders (nano-/microscale)
- Production of uniform powders
- Shape and size of crystals changeable in a wide range by modifying the process parameters
- Fine-tuning of particle-size distribution

### Features

- For rapid expansion of supercritical solutions and gas anti-solvent recrystallization
- Closed CO<sub>2</sub> cycle
- Contaminant-free recirculation of supercritical solvent
- High-performance separation step

#### Options

- Semi-continuous discharging of solid products
- Diamond nozzle sets with different nozzle geometries
- Data acquisition system
- Process control and batch documentation with programmable logic controller (PLC)

# Applications

- Formulation of pharmaceutical products
- Enrobing of active agents
- Production of colour pigments



Multi-purpose unit for micronization using RESS or GAS techniques as well as for supercritical fluid extraction (France). Unit is equipped with a diamond nozzle and a motor-driven pump for pulsation-free spraying.

# Phase Equilibria Apparatus

Standard Design Max. operating pressure Max. operating temperature Equilibria cell capacity

500 bar 80°C 25 ml **Options** 700, 1000 bar 120, 150, 200°C 50 ml

System for fast determination of solubility data and for the observation of phase behaviour at high-pressure conditions, as base for supercritical fluid extraction processes.

# Advantages

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- Pressure-balanced optical cell allows to take samples without disturbing the equilibrium conditions
- Fast achievement of phase equilibria due to intense stirring
- Sapphire windows for the observation of the phase behaviour in function of pressure and temperature

## Features

- Stirred phase equilibria cell with counterbalance piston
- Camera system with endoscope and cold-light source to represent the inside of the cell on a monitor
- Sampling valves for the upper and the lower phase

#### Options

- External recirculation of liquid and gas phase with high-pressure gear pump
- Larger cell capacities
- Sapphire windows up to optical width of Ø 28 mm



Phase equilibria unit with stirred optical cell and pressure-balanced piston for isobaric sampling. System includes also pressure generation, pressure and temperature control, colour camera with endoscope and cold-light source. Unit is in operation in a pharmaceutical company (Belgium). Electrical heating jacket Magnetic stirrer with heating plate

Phase equilibria cell with pressure-balanced piston.



High-pressure gear pump for option «External recirculation» of upper and lower phase. It allows faster mixing of the phases or pulsation-free feeding to an injection loop for online concentration measurements (SFP, HPLC, etc.).

# **Standard Design**

Max. operating pressure50–1,000 barMax. operating temperature20–500°C

Performing a reaction at supercritical conditions enhances the conversion rates and the space-time yield. Smaller reactors can be used for the same amount of reaction product.

## Advantages

- Improved space-time yield
- High selectivity
- Enhanced conversation rates
- Smaller reactors
- Higher catalytic activity
- Homogeneous reactions because of unlimited solubility of reactants
- Increased catalyst endurance

#### Features

- Fixed-bed tube reactors, slurry reactors, recirculation reactors, down-flow column reactors, Berty-type reactors
- Pressure generation for gases and liquids
- Mass flowmeters for gases and liquids

#### Options

- Data acquisition system
- Process control and batch documentation with programmable logic controller (PLC)
- External recirculation of reaction fluids

## Applications

- Hydrogenations
- Polymerizations
- Isomerizations
- Oxidations
- Catalytic reactions
- Enzymatic reactions
- Synthesis reactions
  - Hydrolysis



Pilot unit built for reaction engineering research (Germany). This unit is equipped with a Berty-reactor for 450°C max. reaction temperature.



Pilot unit designed for catalytic high-pressure reactions for a pharmaceutical company (Switzerland). The two reactors are fixed-bed catalytic reactors which can be operated up-flow, down-flow, in series or parallel.

# Pilot Units for Supercritical Fluid Extraction of Solids

Standard Design	
Max. operating pressure	300 bar
Max. operating temperature	80°C
CO <sub>2</sub> flow	18 l/h
Extractor capacity	1 litre

**Options** 500, 700 bar 120, 150, 200°C 10, 30, 50, 100 l/h 2, 4, 6, 10, 20 litres

Selective separation of valuable thermolabile components from solid and liquid raw materials using a supercritical gas as solvent.

### **Advantages**

- Low-temperature separation produces natural extracts
- Chemically inert and non-toxic solvents leave no residue in the extract
- Solubility variation achieved by changing extraction pressure and temperature
- High selectivity and diffusion rate

# Features

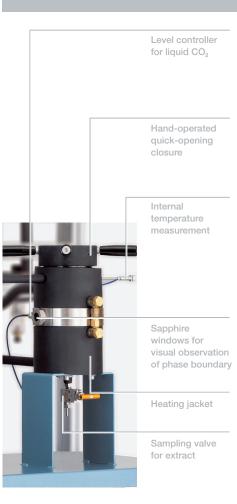
- Closed CO<sub>2</sub> cycle
- Contaminant-free recirculation of supercritical solvent
- High-performance separation step
- Hand-operated quick-opening closures
- Continuous process with liquid raw materials

# Options

- Fractionated separation with fluid cyclone
- Fractionated extraction
- Modifier system
- Multivessel design
- Preparation for retrofit with additional extractors, separators, etc.
- Data acquisition system
- Process control and batch documentation with programmable logic controller (PLC)



Modular SFE pilot unit built for 500 bar max. operating pressure designed for pharmaceutical research (Germany). The system includes the options fractionated separation, modifier system and Coriolis mass flowmeter.



Sophisticated final separator with controlled liquid CO<sub>2</sub> level for the prevention of aerosol forming.