



ExtraLab SFE 100 mL

ExtraLab 100 ml with options

Full integrated system for supercritical CO₂ and subcritical water applications
Lower CO₂ consumption
More options for greater flexibility

The ExtraLab 100 is versatile “multi-solvent” extraction equipment designed to perform several applications :

-  **Extraction with Supercritical CO₂ at conditions up to 700 bars/ 150°C** (1000 bar in option)
-  High pressure and **Subcritical water** extraction up to 400 bar and 250 °C
-  **Reaction / Synthesis** under subcritical & supercritical conditions (water or CO₂)
-  **Sterilization**
-  **Drying** of polymers or innovative materials (aerogels)

EXTRATEX S.F.I

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SCOP SARL au capital variable - RCS Nancy 827 517 194



Main advantages:

- + Very versatile solution (Supercritical CO₂ & subcritical water application)
- + Wide range of process conditions (0-1000 bar / 0-250°C / 0-100 g/min)
- + Only small quantities of product necessary (analytical studies)
- + Easy & user friendly operation
- + Small footprint (Benchtop system ideal for laboratory use)
- + Low CO₂ consumption due to recycling
- + Datalogging of all process parameters
- + High precision

EXTRACTION PROCESS PRINCIPLE

Liquid carbon dioxide from the storage cylinder is delivered to the condenser.

Cooled liquid CO₂ is pumped with the HP pump and pressure is raised up to extraction pressure. High-pressure CO₂ is then heated in the heat exchanger to achieve supercritical conditions.

The CO₂ properties can be modified with the co-solvent pumped before the heat exchanger with a co-solvent pump (if existing).

The reaction or the extraction takes place in the reaction / extraction vessel that is maintained at constant pressure and temperature. A back pressure regulator enable pressure control and depressurization of the CO₂. Depressurized CO₂ enables to separate extract and gaseous CO₂. Products are collected in the separators and CO₂ is either vented or re-circulated.

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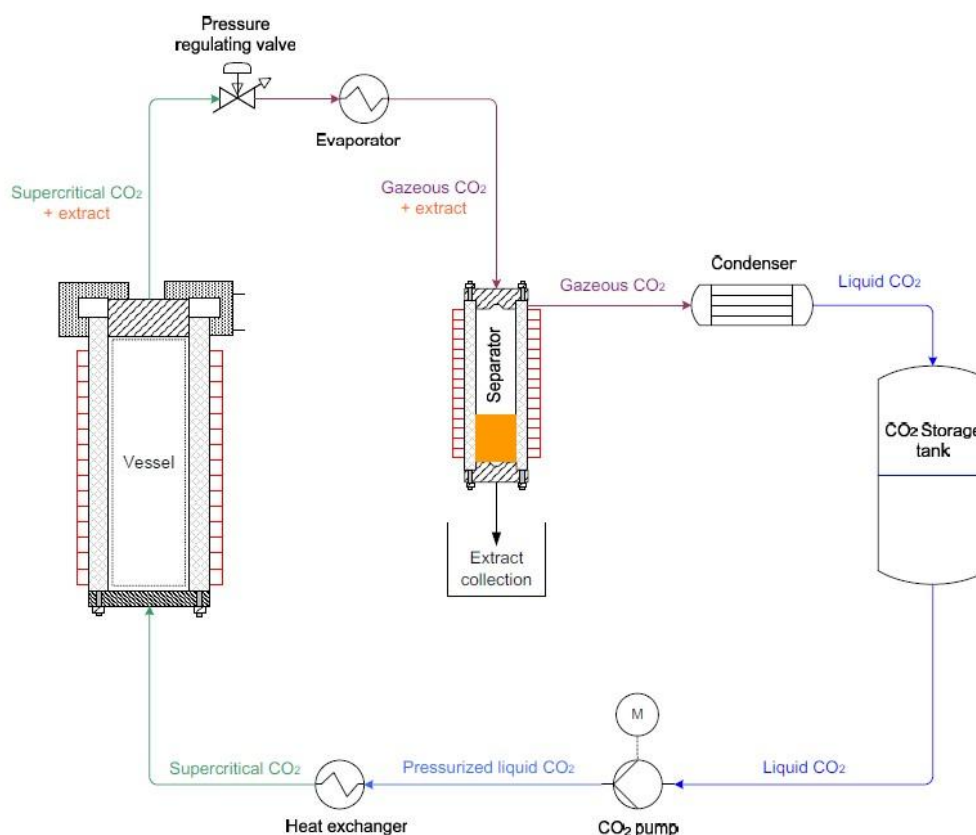
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Simplified principle of Supercritical Fluid Extraction

Technical Characteristics

EXTRACTION VESSEL	
Capacity	100mL
Max. Working Pressure (PS)	700 bar (1000 bar in option)
Max. Working Temperature (TS)	0/+250°C
Material	Stainless steel 1.4404-316L or 1.4542
Closing system	Simple screwed top & bottom lid Easy and quick handle operation
Sealing	PTFE lip seal with stainless steel spring
Heating system	Electrical oven

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The extraction vessel is equipped by:

- ✚ A screwed bottom and top lid, easily dismantable by hand
- ✚ 2 filters to contain the feed into the extraction vessel
- ✚ Ingenious design to percolate perfectly the feed
- ✚ A heat exchanger placed inside the oven, before the extraction vessel, to pre-heat the fluid entering inside the vessel.



Example of 100ml vessel inside oven

CO2 PUMP	
Fluid	Liquid CO ₂
Max. Discharge Pressure	700 or 1000 bar operation
Max. Flowrate	50 ml/min at 1000 bar or 100 ml/min at 350 bar
Material	Stainless steel 1.4404-316L

The extraction vessel is connected to a CO₂ pump (able to pump water on water configuration) and optionally to a co-solvent pump in addition.

- ✚ Volumetric piston pumps with jacketed head(s) for cooling equipped with multiheads

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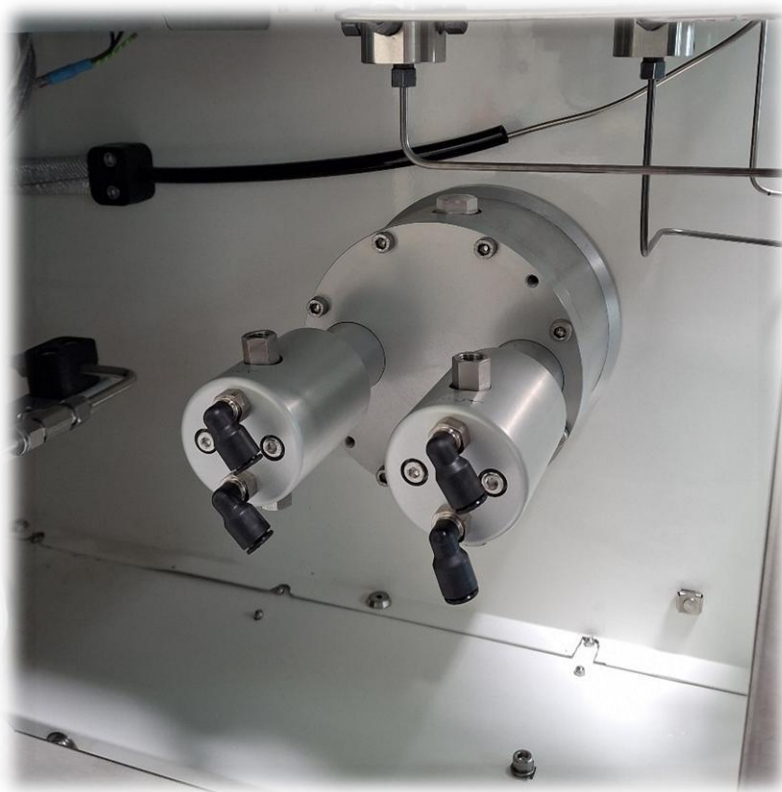
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- ✚ Innovative high pressure pump head with 2 heads for maximum operational reliability
- ✚ **High performance** from low to high flowrates
- ✚ **Very low maintenance cost** and long service intervals
- ✚ CO₂ flow rate is controlled automatically with a frequency drive and a Coriolis mass flowmeter that gives a precise flowrate of the fluid at the inlet of the extractor.
- ✚ Connected to a condenser and sub-cooler to efficiently pump liquid CO₂ from recycling line



ExtraPump Lab

CO-SOLVENT PUMP (IN OPTION)

Fluid	Liquid solvent
Max. Discharge Pressure	400 bar
Max. Flowrate	10 mL/min at 400 bar
Material	Stainless steel 1.4404-316L

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A co-solvent pump can be added in option in order to add a co-solvent (such as ethanol) in the extraction solvent.

We supply a reliable HPLC piston pump with low volume dampener to reduce pulsation.

Pressure control

Pressure is precisely controlled with a back-pressure regulator. In standard configuration, the pressure is adjusted manually.

When using subcritical water extraction, the water fluid is cooled down through a heat exchanger (pipe in pipe) before the back pressure regulator.



Manual back pressure regulator

Option : An automated Back Pressure Regulator can be proposed in option for an automatic control of the extraction pressure. The pressure is controlled from the PLC.

SEPARATION	
Capacity	1 x 50 mL
Max. Working Pressure (PS)	200 bar
Max. Working Temperature (TS)	0/+80°C
Material	Stainless steel 1.4404-316L
Closing system	Screw bottom and top lid
Sealing	O-ring seal
Heating system	Evaporator placed upstream of each separator

One gravity separator (high performance) working under pressures of up to 200 bar allows CO₂ recycling and extract collection. Working with an HP separator allows minimizing solvent consumption, easing the separation, increasing the collection efficiency, and limiting plugging problems.

The enthalpy required for this separation is brought by the evaporator (heat exchanger) placed upstream of the separator in order to vaporize the CO₂ and avoid icing of the extract.

The extract and/or the liquid co-solvents are manually collected at the bottom.

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50mL Gravity separator

Option : A second heated separator or cold trap of capacity 50mL or 100mL can be added as an option to optimize the separation efficiency or to trap volatiles and residual water before CO₂ recycling.

A second Back Pressure Regulator can also be proposed for extract fractionation between the separators.

Option : A sapphire visualization separator of 50mL capacity can be proposed as an option instead of the cyclonic separator for a full visualization of the extract collection.

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50mL Sapphire visualization separator

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Heat exchangers & Recycling line

- + Condenser: The condenser allows liquefying the gaseous CO₂ prior to recycling. It is cooled by an ethylene glycol / water mixture supplied by the chiller.

The recycling line (high pressure line coming from separators outlet to pump inlet) is included as a standard, and integrate a condenser and sub-cooler.

- + Pre-Heater: the heater allows heating the CO₂ after the pump in a range of temperatures from +20 to +100°C. It is electrically heated by heating shells and independently heated from the extractor for an optimum efficiency and versatility.

- + Evaporator: this heater allows evaporating the CO₂ just after the depressurization. It is sized to bring necessary enthalpy. The temperature control range is from +20 to +80°C. It is electrically heated by heating shells

Utilities

- + An air-cooled chiller is supplied to provide the chilled water necessary in the condenser, sub-cooler and in the pump heads. This chiller must be placed outside or in a ventilated area.
- + All heating for heat exchangers vessel, separators are electrical. Extratex develop precise controls for these heating to obtain a quick and precise temperature control. 3 probes are used. A probe in the process fluid, a probe in the vessel metal and a redundant safety probe as imposed by EU standards as well as Extratex safety policy.

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Air-cooled chiller

Operation / Control

The unit is controlled from a touch-screen control panel on the main skid. The control software enables to choose set-points and indicates pressures, temperatures, and flowrates for the different sections.

Valves are manually operated directly on the equipment.

Automatic temperature and pressure safeties guaranty the safe operation at any time. A data acquisition module allowing the storage of operating parameters storage and exporting said parameters to a PC is available.

In this case, the PC-based software allows:

- Display of all parameters on the main screen
- Process parameters remote control (pump, extractor pressure, temperatures)
- Alarm parameters choice, detection and record
- Datalogging, storage of all process parameters on data files
- Curve display of all parameters

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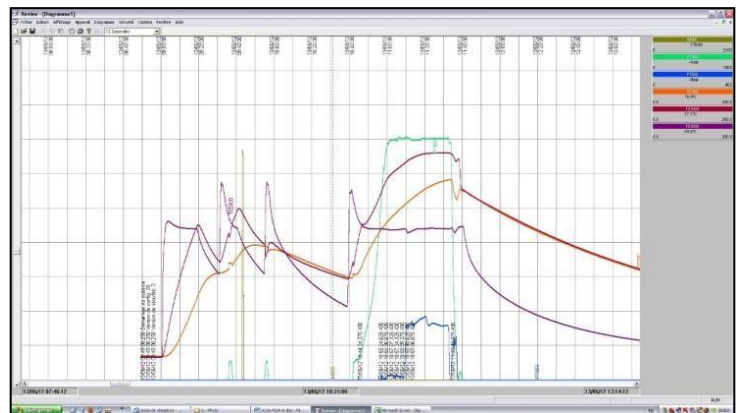
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Parameters controller



Curves and historical display of parameters



Remote control (on mobile or laptop)

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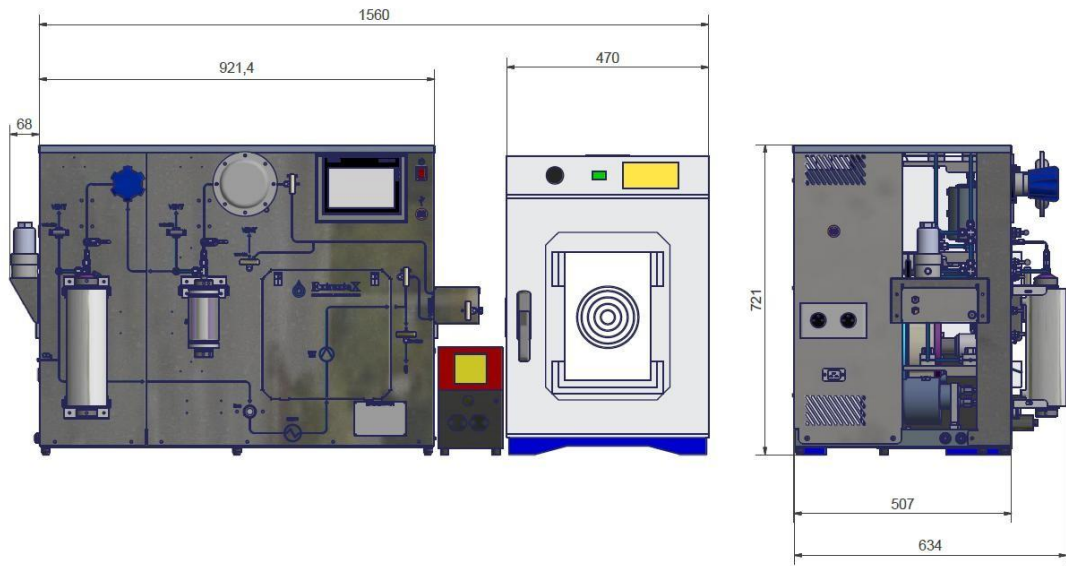
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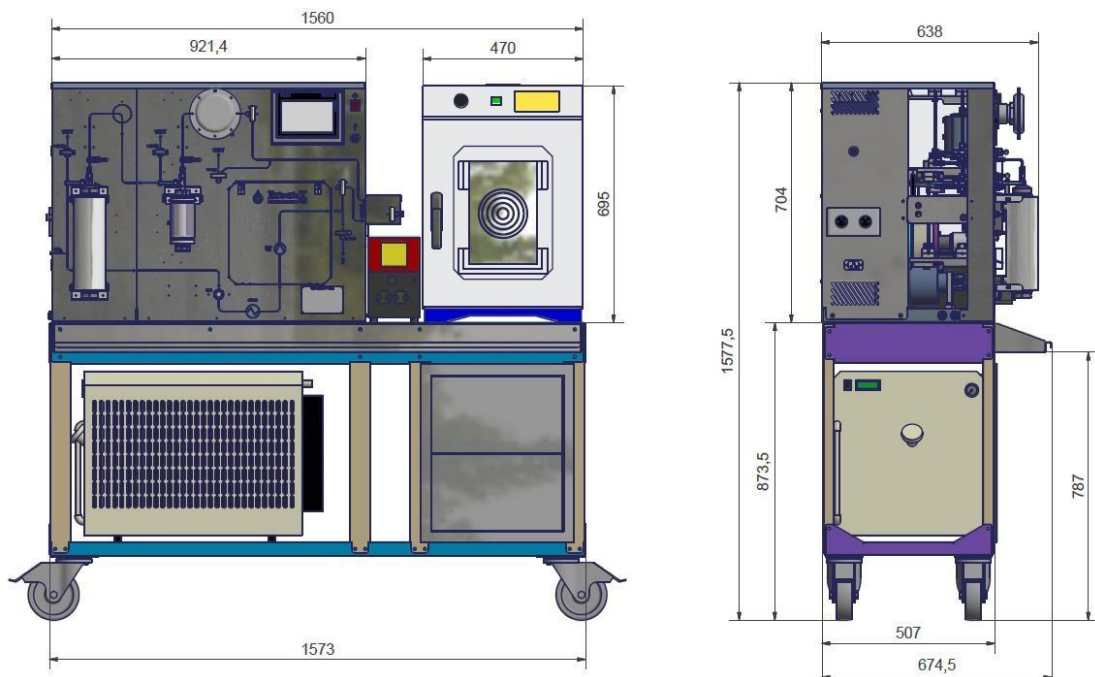
Installation requirements

Process skid dimensions:

- Standard configuration : 156 cm x 63 cm x 75 cm (L x D x H)



- Mobile skid configuration : 157 cm x 68 cm x 160 cm (L x D x H)



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Connections:

- + Liquid CO₂: 50 Kg Cylinders equipped with dip tube, liquid CO₂ stored at 50/60 bar.
Connection to the process skid: 1/4" OD (e.g. SWAGELOK fittings)
- + Vent Line: to be connected to outside (atmosphere) – G 1/2" connection
- + Power supply : 1 Phase 230V + Neutral + Earth
- + Air supply 6/7 bars (only needed for automated back pressure regulator option)

Material

All components, vessels, piping, fittings and instrumentation are made in stainless steel 316316L. Gaskets and seals are mainly made from PTFE or EPDM.

Certification

All ExtrateX-SFI equipment are confirmed to the following European Union directives, where applicable:

- 2014/68/EU : PED Directive certification
- 2006/42/EC : European Machinery Directive
- 2014/35/EU : Low Voltage Directive

This equipment is designed and constructed in accordance with the EU safety standard for machine construction. In particular it conforms to the PED 2014/68/UE pertaining to pressure vessels and pressure equipment assemblies. It is also compliant with ASME standard, ASME VIII Div. 1 Computation Code used for the calculation of the Extraction's vessel.

The equipment is certified to be able to handle any fluid including flammable and toxic products which is mandatory for the use of co-solvents (cf. fluid group 1 in PED 2014/68/EU).

All pressure vessels and all heating sources are protected with **redundant** pressure and temperatures safeties: A Pressure Switch at the level of the HP Pump, Temperature Switches for all heating elements and Rupture Disks or Safety Valves depending on the vessel.

The system is **not** Ex proof classified.

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A global hazard analysis is supplied with the system and all safety and operation recommendations are given in full detail.

Training – Technical assistance

Training on installation, good operation, safety, and maintenance is included and will be given at our premises in Extratex facilities, FRANCE (duration: 1 day).

Additional training programs on any topic of SCF Technology as well as on-site training can be agreed upon request.

Documentation

All documentation will be supplied in English. This include:

- Drawings showing equipment layout, component mechanical designs
- P&ID and flowcharts for each process
- Electrical and control wiring diagrams
- Safety and test certificates
- User manual (Installation, operation and maintenance for the equipment)
- Spare parts lists
- Regulatory CE certificates, pressure vessel certification, hazard analysis

This documentation will be supplied printed as well as in electronic form (USB card).

Quality Assurance / Warranty

The project is further guarded by a team of experts for any aspect of the process, like mechanical and electrical safety, process automation and control, eventual chemical and material aspects of the process, and logistical questions.

EXTRATEX guarantees the full functionality of the equipment during one year following the delivery date and shall exchange any defective part (except parts which ought to be changed by way of maintenance) at his costs during this period. Subject to the above, this guarantee does not include or cover consumable products and damages caused by anormal wear and tear , or damages caused by improper handling, storage and/or operation of the equipment carried out by the client.

Extratex' above mentioned guarantees are only given under the conditions that:

- Any modification to Extratex initial design requested by the client must have been expressly approved by Extratex;
- The Client uses the fabrication auxiliaries specified by Extratex (chemicals, water, cleaning agents ...)
- The Installation of the Equipment have been done with the right procedure given by Extratex
- The Equipment is operated by the Client as per all instructions delivered by Extratex (with all the required staff, utilities and chemicals according to Extratex specifications) and all other relevant parameters (temperature, pressure, etc....)

After Sales Services / Maintenance

Extratex proposes curative or preventive maintenance on all our systems, as well as calibrations and certifications. Our after-sales service propose a phone assistance, can repair components in our workshop, or can operate world-wide on-site. We also sell all necessary spare parts.

Extratex technicians can also advise on process improvement thanks to their high knowledge and experience on supercritical processes.

Maintenance contracts

In order to prevent damage to the equipment and downtime, EXTRATEX can offer a maintenance program/package. The maintenance contract usually covers a periodic review of all operational and safety devices (instrumentation calibration, advice and review on the safety systems, proposition of modifications), and replacement of defective parts.



Confidentiality

The present proposal is EXTRATEX' sole property. It shall be considered as EXTRATEX' proprietary confidential information and shall be treated as such.
