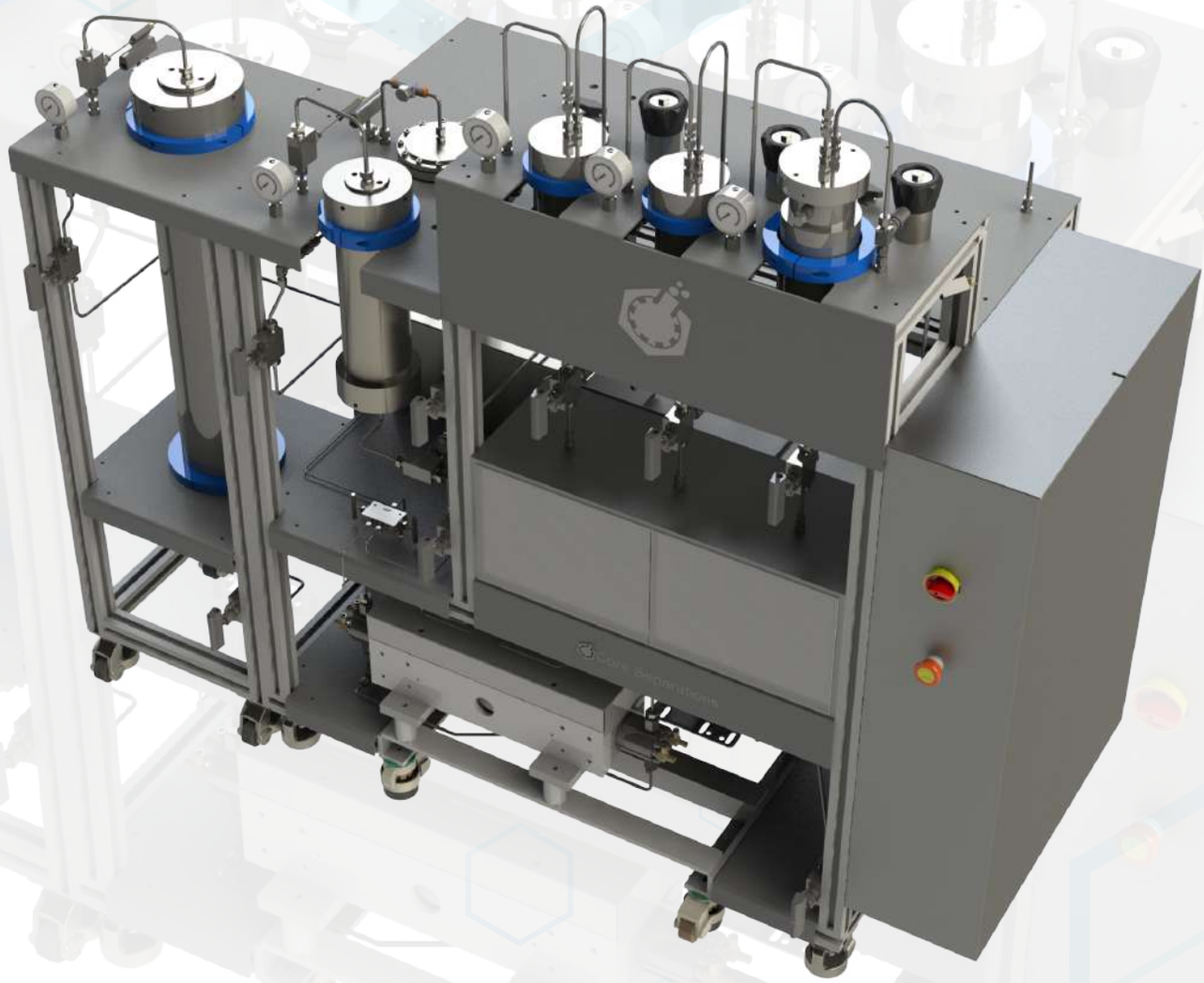


Core | **Extraction**

SFX1L Specification 2022©

SciMED Core Separations

What is CO_2 Processing

Most people are familiar with CO_2 presenting as three states of matter: solid, liquid and gas. These states are dependent on the temperature and pressure of the CO_2. In its natural state, CO_2 is most abundant as a gas making up around 0.04 % of the earth's atmosphere. However, by altering the ambient conditions we can transform CO_2 into either a liquid or a solid.

A phase diagram can be used to determine the state at which CO_2 exists at both a defined temperature and pressure (see figure 1-1). For CO_2 we see two intersect points on the phase diagram, the triple point and the critical point.

The triple point is where the three states of matter (solid, liquid and gas) co-exist in equilibrium. For CO_2 the triple point is 5.1 bar and -56 °C. Any change from these conditions alters the state of matter in favour of one of these forms.

For example, CO_2 as a liquid exists when the pressure exceeds 5.2 bar at temperatures between -56 °C and 31 °C (these are the temperatures falling between the triple and the critical point - See figure 1-1).

At the critical point we observe a 4th state of matter known as the supercritical region. In this region CO_2 is no longer a gas or a liquid but exhibits properties of both and is known as the supercritical phase. Supercritical CO_2 exhibits some unique properties

1. High densities similar to that observed in liquids
2. Low viscosities near to those of gases
3. Virtually no surface tension.
4. Higher diffusion coefficients than liquids

These properties give an extremely versatile solvent that can be used for a number of applications ranging from extraction of natural materials to chemical reaction.

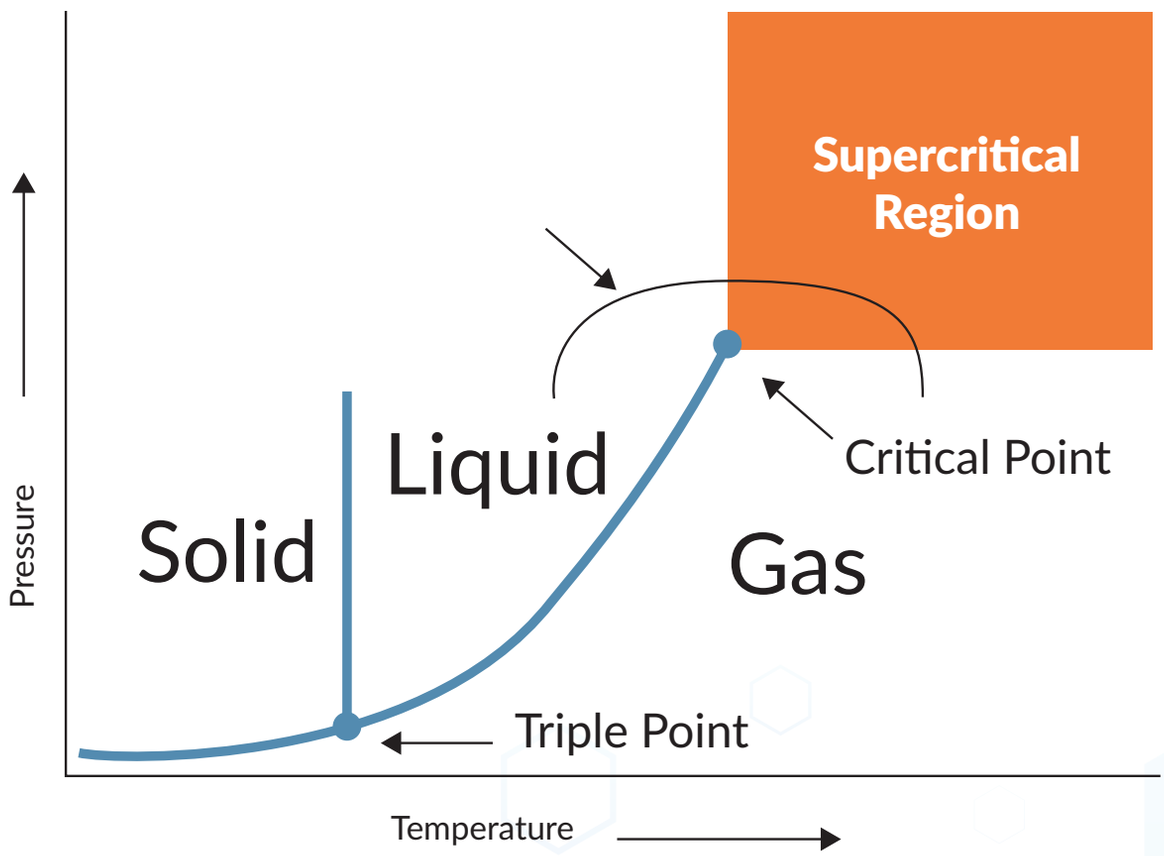


Figure 1–1: Supercritical CO_2 is tuneable without changing phases

Why use Supercritical CO_2

Supercritical Fluid Extraction (SFE) using CO_2 is commonly used to extract compounds from solid botanical materials due to its achievable pressure and low temperature (critical temperature and pressure of 31 °C and 74 bar). It exhibits a number of benefits unique to CO_2 over traditional petrochemical derived alternatives.

Tuneable Density - Supercritical CO_2 occurs when CO_2 is compressed to 74bar @ 31 ° C. This results in a density of around 440 kg/m^3. However, as the pressure and temperature alter the density can increase to over 1000kg/m^3 (density of water). This tuneable density gives CO_2 its selective extraction properties and makes it a very versatile solvent.

Tuneable Polarity - CO_2 is a good extraction solvent for lipophilic and hydrophobic molecules, which is why it is popular in natural product extraction. However, there are times when the product of interest is more polar. The polarity of the CO_2 can be adjusted with the addition of a solvent of higher polarity such as ethanol. Small percentages of more polar solvents can have a significant effect on which components are extracted. It can also help reduce the pressures required to extract components such as polyphenols.

Selective Fractionation - During an extraction, conditions can be adjusted to alter the density of the CO_2 to selectively extract specific components. The same tunability is possible on the collection side.

With a system that has multiple collectors with their own back pressure regulators, the conditions in each separator can be adjusted to achieve a specific density. Selectively precipitating different compounds into each of the separators.

Isolation - When isolating the extract from a CO_2 extraction, it requires depressurisation of the CO_2. This involves a phase change from a supercritical fluid into a gas. This ultimate change in density results in the separation of the dissolved compounds from the CO_2. The CO_2 gas is then able to escape leaving the extract uncontaminated by the extracting fluid.

Recyclable - One of the most powerful aspects of CO_2 as a solvent is witnessed when collecting the product from the separator as it reverts to a gas, so leaving your product uncontaminated. We can also re-use the CO_2 by re-compressing it. The most common method is to drop the pressure of the CO_2 in the final collector to 55 bar (bottle pressure) so that it can be recycled back into a storage tank for reuse.

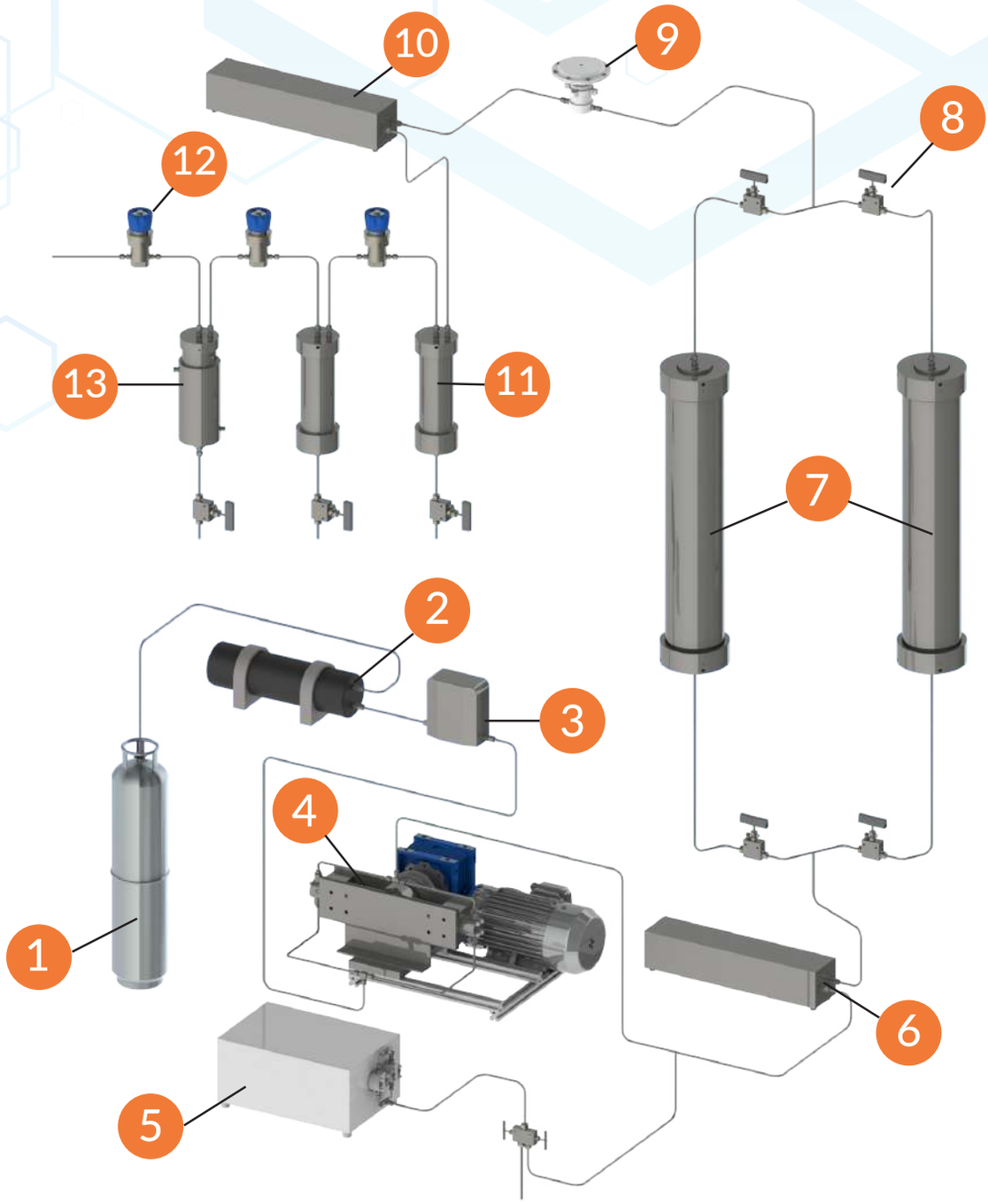
However, this can present some challenges, as materials can carry over and cause blockages. The material that is carried over can also contaminate the extraction process. By understanding the material and process conditions, these effects can be minimised and/or removed.

Environmentally Responsible - Unlike other solvent extraction, CO_2 is recovered from other industrial processes as a by-product. It is purified and stored ready for use in many different processes including supercritical CO_2 extractions. The renewable and abundant nature of CO_2 is one of the most attractive properties when using CO_2 as an alternative solvent, however it also has other benefits:

1. **Non-toxic,**
2. **Non-flammable**
3. **Non-Eco toxic**

This combination of properties makes CO_2 a powerful alternative solvent for industrial processing.

Process Flow Diagram



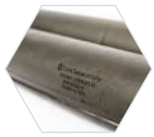
1 CO_2 Cylinder	5 Co-solvent Pump	9 Automatic Back Pressure Regulator
2 Condenser	6 Pre-heater	10 Vaporiser
3 Flow Meter	7 Extraction Vessels	11 Separators
4 CO_2 Pump	8 Inlet and Outlet Valves	12 Manual Back Pressure Regulators
		13 Cold Trap

1

Core | **CO2 Cylinder** (included)

CO2 systems start with a source of liquid CO2 produced as a by-product from other industrial processes. The CO2 is delivered as a liquid and not a gas as the high-pressure pumps are designed to compress liquid into it supercritical state.

2



Core | **Condenser** (included)

Although we use a liquid CO2 feed in our extraction systems, it's important that the incoming CO2 remains liquid. The condenser acts to maintain the incoming temperature of the CO2 ensuring it remains a liquid during the pumping phase. Additional condensers can be added with higher flow rate pumps or the addition of a recycling unit.

3

Core | **Flow Meter** (optional)

Using a mass flow meter corrects the speed of the pump as the density varies, allowing us to accurately deliver the correct mass of CO2 during an extraction. We can observe the in-coming CO2 densities in real time into the system which helps us quickly diagnose any problems with the incoming CO2 supply.

4

Core | **CO2 Pump** (included)

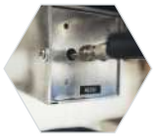
The pump is designed for minimal pulsation using two pistons for an operating pressure range of up to 689 bar. Pump heads are cooled for efficient operation by removing compression heat, using circulating chilled fluid via cooling tubes inserted into machined cavities in the pump head.

5

Core | **Co-Solvent Pump** (optional)

The polarity of the CO2 can be adjusted with the addition of a more polar solvent like ethanol. Small percentages of more polar solvents can have a significant effect on which components are extracted. It can also help reduce the pressures required to extract components such as polyphenols.

6



Core | **Pre-Heater** (included)

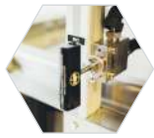
The pre-heater is located just after the pump to control the temperature of the CO2 reaching the extractor. It ensures the CO2 entering the extraction vessel is already at the optimum extraction temperature ensuring a controlled extraction process.

7

Core | **Extraction Vessels** (included)

The double-ended vessel comes with threaded closures for easy, fast and safe opening. Its sealing design allows for hand-tight operation for the end caps that are also fitted with frits to retain feedstock. The vessel is electrically heated with a heat jacket and thermocouple for temperature control.

8



Core | **Inlet and Outlet Valves** (included)

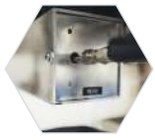
Inlet and Outlet valves – These valves allow the entry and exit of CO2 into the extraction vessels.

9

Core | **Automatic Back Pressure Regulator (ABPR)** (included)

The pneumatically actuated diaphragm acts on the needle assembly within the regulator to automatically regulate the extraction pressure in the system.

10



Core | **Vaporiser** (included)

The Joule-Thomson effect is observed when we go from a high pressure to a low pressure resulting in a drop in temperature. To overcome this, we use a Vaporiser to heat the CO2 exiting the ABPR. The vaporiser also helps to expand the CO2 from its liquid state into a gas in-order to help precipitate the extracted components.

11

Core | **Separator** (1 included)

The mixture leaving the extractor, composed of extract/CO2 and solvent, is depressurised in one or more separators. At each pressure stage, the extract is separated from the CO2 at the desired pressure, before the CO2 is recycled or vented after the last separator.

12

Core | **MBPR** (1 included)

By modifying the pressure and temperature in each separator the density can be accurately controlled to favour the precipitation of some components over others. The manual back pressure regulators facilitate the control of the pressure in each of the separators.

13

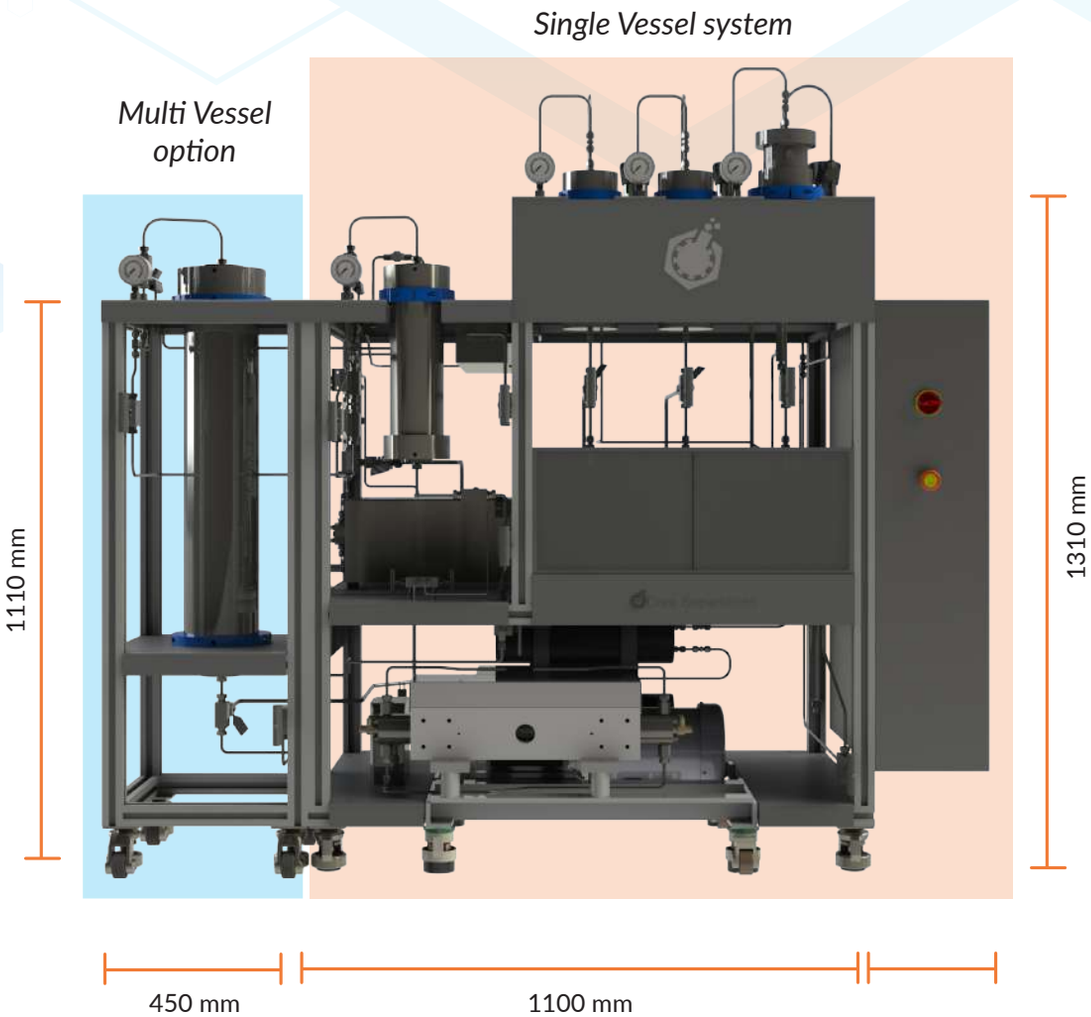
Core | **Cold Trap** (optional)

When looking to collect more volatile compounds such as terpenes, then traditional heated separators can cause these compounds to volatilise and leave with the gas stream. To avoid this a cold trap can be added at the end of an extraction system to help capture these components.

Dimensions

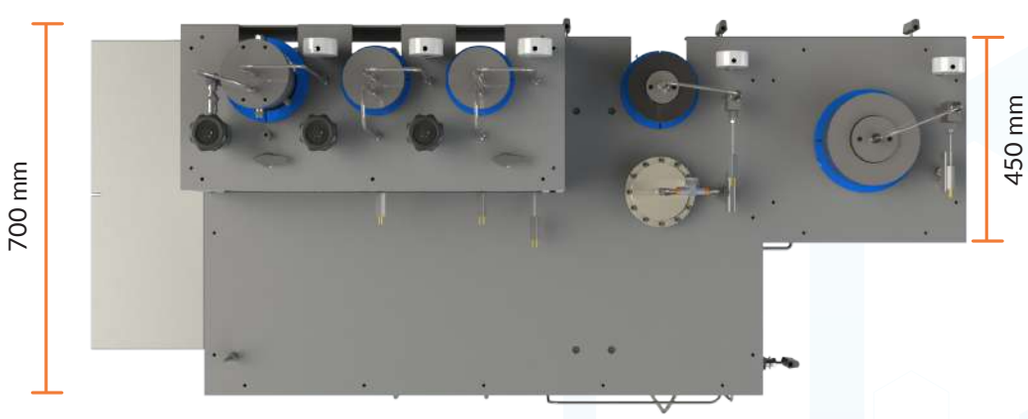
Front View

Multi vessel system



Top View

Multi vessel system



System and component details

Technical Specifications

System's technical specifications	
Extractor volume	1L
Max operating pressure	600 bar (8700 psi, allowance for relief or safety devices)
Max operating temperature	Up to 150°C
Max operating flow CO_2	12 Kg/hr (200 g/min)

Specification and Requirements

Power requirements
415 V (3PH+N+E); upto 64A (depends on heating options)

Pneumatic Air Pressure (bar/psi)
6.9 bar / 100 psi, 1/4" compression inlet

CO_2 Inlet
55 bar, 1/4" compression inlet

Vent Line
3/4" compression inlet

Weight
350/400 kg (depending on options)

Chiller
Required

PC & Monitor
Minimum of 1.5 GHz, 16 GB RAM, 250 GB storage, Ethernet port for control panel, wired or wireless connection for Internet connectivity. Google Chrome browser. Monitor 21" minimum with 1920 x 1080 pixels resolution

SFX1L Specification

Condenser

Maximum operating pressure	350 bar (5100 psi)

Flow Meter (optional)

Standard flow rate	1 to 350 g/min
Maximum operating pressure	200 bar (2900 psi)

High Pressure CO_2 Pump

Standard flow rate	12 Kg/hr (200 g/min)
Maximum operating pressure	600 bar (8700 psi, allowance for relief or safety devices)
Maxiumum design pressure	689 bar (10,000 psi)
Control	Computed Flow Control, Pressure Control, Flow Meter Control (with purchase of Flow Meter Option)

Co-solvent Pump

Standard flow rate	3 Kg/hr (50 g/min)
Maximum operating pressure	600 bar (8700 psi, allowance for relief or safety devices)
Maxiumum design pressure	689 bar (10,000 psi)
Control	Computed Flow Control, Pressure Control, Flow Meter Control (with purchase of Flow Meter Option)

Pre-heater

Maximum operating pressure	689 bar (10,000 psi)

Extraction Vessel

Capacity	1L
Max operating pressure	600 bar (8700 psi), design 689 bar
Max operating temperature	150°C
Temperature Control	Electric Heat Jacket and Thermocouple
Design criteria and certifications	ASME Section VIII, Div. 1; European Pressure Equipment Directive (PED - 2014/68/EU). Note: Actual certificate issued by Notified Body for ASME or PED is at additional cost and listed in options.
Hydrostatic Test Pressure	1.5 X design pressure

Automated Back Pressure Regulator

Standard flow rate	36 Kg/hr (600 g/min);
Maximum operating pressure	600 bar (8700 psi), design 689 bar
Pressure Control	Pressure Sensor

Vaporiser

Maximum operating pressure	515 bar (7500 psi)

Separator

Volume	1 Liter
Max operating pressure	180 bar (2610 psi); Design 200 bar
Max operating temperature	70°C
Control	Pressure control using Manual Back Pressure Regulator, Pressure Guage, Pressure Sensor. Temperature control with electric heat jacket and thermocouple

Extras

Baskets

When performing an extraction, the most convenient accessory is the use of an internal basket. The basket allows you to both load and unload your extraction vessel relatively quickly giving you two major benefits:

i). Quick loading and unloading allows increased turnaround time speeding up the productivity of the system.

ii). Emptying and cleaning the basket can be performed away from the system. This ensures not only that the basket is clean preventing cross contamination, but the system can be running if multiple baskets are in operation.

Order Number	Description
EV500mL-Tech	Extraction Vessel Basket, 500mL vessel threaded end caps 5u frits standard
EVB1L-TEC	Extraction Vessel Basket, 1L vessel threaded end caps 5u frits standard
EVB3L-Tech	Extraction Vessel Basket, 3L vessel threaded end caps 5u frits standard

Fractionation

During an extraction, conditions can be adjusted to alter the density of the CO2 to selectively extract specific components. The same tunability is possible on the collection side.

With a system that has multiple collectors with their own back pressure regulators, the conditions in each separator can be adjusted to achieve a specific density. Selectively precipitating different compounds into each of the separators.

Order Number	Description
CS100mL-Sys	Cyclone Separator 100mL, PG, HJ, TC, PT, MBPR, DV, SV
CS250mL-Sys	Cyclone Separator 250mL, PG, HJ, TC, PT, MBPR, DV, SV
CS500mL-Sys	Cyclone Separator 500mL, PG, HJ, TC, PT, MBPR, DV, SV
CS1L-Sys	Cyclone Separator 1L, PG, HJ, TC, PT, MBPR, DV, SV
CVCT1L-Sys	Cold Trap Separator 1L, PG, TC, PT, MBPR, DV, SV

Subcritical

Supercritical systems the CO2 is above the critical point and in subcritical systems is below the critical point. In practical terms a supercritical system operates above 31C and 74 bar with the temperature being the key factor. In most laboratory scale supercritical systems, heating is done electrically. But if you want to go subcritical, in general you are working at temperatures below 31C. This would be limited when using electrical heating. So most subcritical systems use liquids to heat and cool.

Dual Vessel Extraction

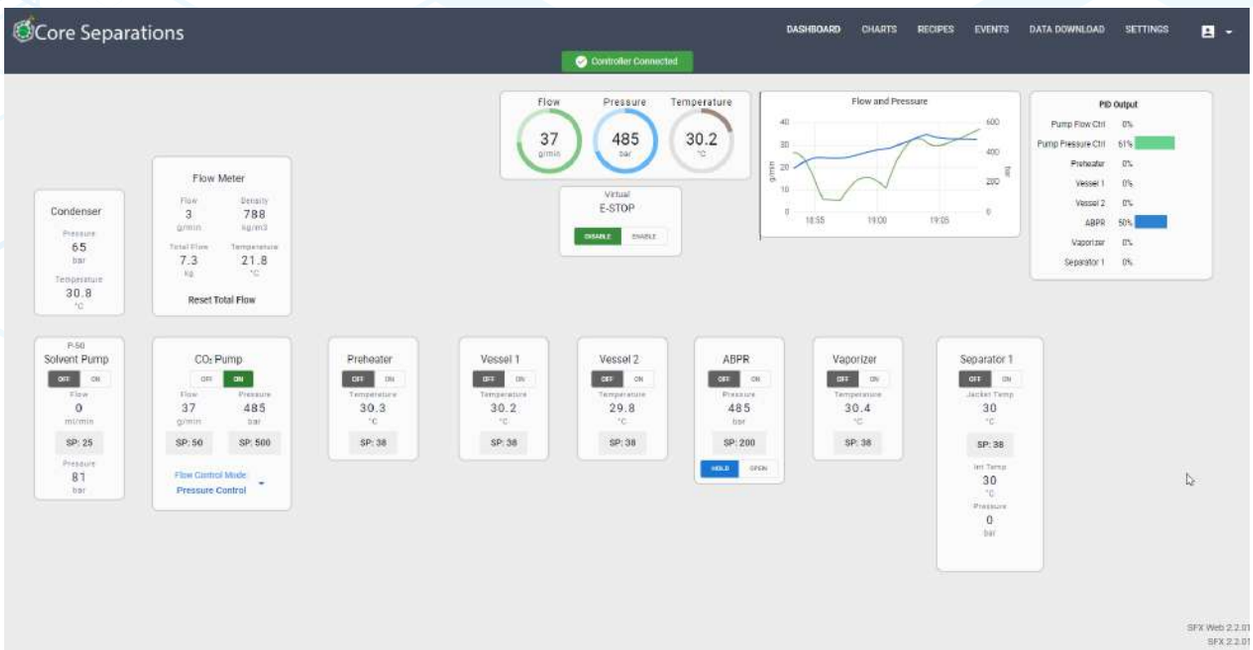
Our systems are designed to be modular and upgradeable. This allows our customers to modify the systems to meet their research needs or processing requirements. Dual extraction vessels offer the flexibility to make the system semi continuous by allowing the user to extract from one vessel while preparing another. Or by varying extraction vessel volumes to allow different scale extractions to be performed, making the system both a versatile research tool and a pilot scale production system.

Multi-Vessel Upgrade

Order Number	Description
EV500-Sys	Extraction Vessel, 500mL w/heating PG, HJ, TC
EV1L-Sys	Extraction Vessel, 1L w/heating PG, HJ, TC
EV3L-Sys	Extraction Vessel, 3L w/heating PG, HJ, TC

SFX Software

Whether you are in a demanding research environment or within a highly regulated cGMP manufacturing facility, our SFX software has been designed from the ground up, to be both a flexible and powerful companion in processing supercritical fluids.



Key Features

Dashboard visualisation of key processing parameters

Manual control of key components within the SFX system in real time using APC to accurately control the pressure

Recipe menu allows you to automate a variety of conditions including flow rates, temperatures and pressures over a defined time limit.

Real time data logging and visualisation via Grafana Dashboard

Programmable warning and alarm limits to alert the user that the system conditions are approaching the cut off safety limits.

SQL database logs all the alarms and user activity to aid in fault detection and diagnosis.

Grafana is an open-source platform which offers the ability to efficiently unite a variety of data sources within our system such as flow, pressure and temperature readings. The customisable dashboard helps the data to be easily visualised by the user for effective process monitoring and control.

When dealing with high pressure systems, pressure control is key. Standard control is accomplished using proportional, integral and derivative control (PID). Unsatisfied with the standard level of control, Core Separations developed APC (Advanced Pressure Control). This multilevel PID control achieves superior operational management while maintaining rapid pressure build up.

SQL database to store all data and Metadata. Data are never deleted or removed by these systems. Users can archive and store archives in secure locations. Archives can subsequently be viewed as read-only files on the system. User ID/ Password to track the time, date, and name of the user performing actions. The event is recorded, and appropriate before/after states are recorded.

FAQ on Extraction

One of the more common applications for sub and supercritical CO_2 is the extraction of natural materials. However, CO_2 is a powerful non-polar solvent and can be used to extract a variety of components traditionally extracted using petrochemical derived solvents such as hexane. CO_2 has been used in a number of industries for decades with the most recognisable applications being the decaffeination of coffee, hop oil extraction, defatting cacao and in more recent years, extraction of cannabis. The process involves CO_2 either as a pressurised liquid or in its supercritical state passing over a solid bed of the material, extracting soluble compounds. These then can be collected by precipitating them once the CO_2 is depressurised to a gas.

What the best way to approach a new extraction in CO_2?

There are many ways to approach an extraction. The best way is to look back into the literature and see if anyone has done it before. If not, is there something close. As natural products vary, conditions that worked for one researcher may not be optimal for another. Now, as CO_2 extraction is a combination of both varying pressure and temperature it's best to look at the density that is achieved when you alter these conditions. Then you are looking at only one variable rather than two.

When do I use a co-solvent?

CO_2 is a relatively non-polar solvent and therefore is good at extracting non-polar compounds. So when it comes to the extraction of more polar compounds then it needs a little help. By mixing a small amount of a more polar solvent such as ethanol, you can significantly improve the polarity of your extraction stream!

How much CO_2 do i need per extraction?

This is a factor of residence time and solubility in CO_2. The residence time is the amount of contact the CO_2 has with the compounds you are trying to dissolve. For example, if you know how much CO_2 is required to dissolve 1 g of your substrate you can calculate the amount of CO_2 required for your extraction.

250g of CO_2 for 1g of substrate. If you have 250g of substrate then you need 250g x 250g = 62.5 kg of CO_2.

So the faster I flow the CO_2 into my vessel the faster the extraction is done?

Now it starts to get technical! The velocity of the CO_2 in your extractor can begin to cause issues in your extraction. If the velocity is too high you can get channelling in the material. These channels allow the CO_2 to pass through the material without interacting with it. Residence time of the CO_2 is also important. If the velocity is too high, then the CO_2 doesn't have time to dissolve the compounds you are interested in.

So if channeling affects you extraction, does that mean i need to pack my vessel well?

That's right, if the vessel is half empty then the material floats inside the extractor. The CO_2 can then pass by the material without interacting with it. In reality it will still dissolve some material, but the extraction won't be very efficient.

What is the difference between Subcritical CO_2 and Supercritical CO_2 Extraction?

The main difference is that in supercritical systems the CO_2 is above the critical point and in subcritical systems is below the critical point. In practical terms a supercritical system operates above 31C and 74 bar with the temperature being the key factor. In most laboratory scale supercritical systems, heating is done electrically. But if you want to go subcritical, in general you are working at temperatures below 31C. This would be limited when using electrical heating. So most subcritical systems use liquids to heat and cool.

But why use subcritical conditions? Isn't Supercritical CO_2 better?

As with most things it depends. When doing an extraction there are a number of things to consider. Firstly, when we compress CO_2, we increase its density by forcing the CO_2 molecules closer together. By manipulating this density, we can affect what dissolves in the CO_2. But this is not the whole story..... in liquid extractions the biggest driver is polarity. We vary the solvents to alter the polarity. Different polarities give rise to different solubilities in certain compounds. CO_2 is traditionally considered a non-polar solvent, but there are polarity differences when CO_2 is in its supercritical state versus its liquid state (subcritical or high pressure liquid).

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