Core | Extraction

General Operating Guidelines v1.2

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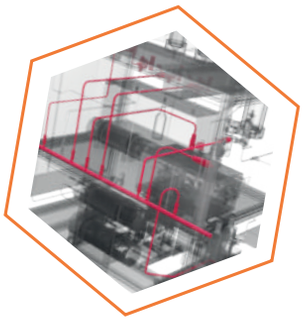
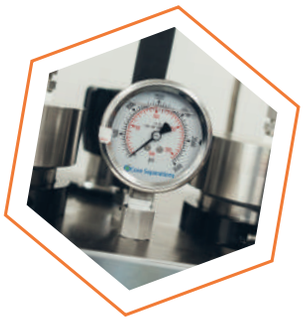
SciMed™

Core Separations

Contents





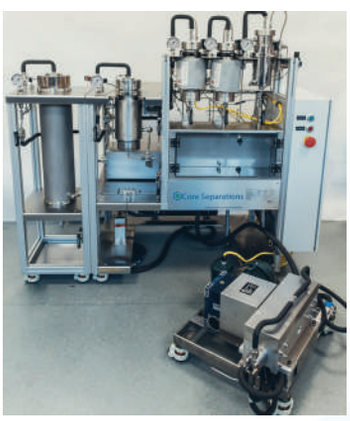
01. System

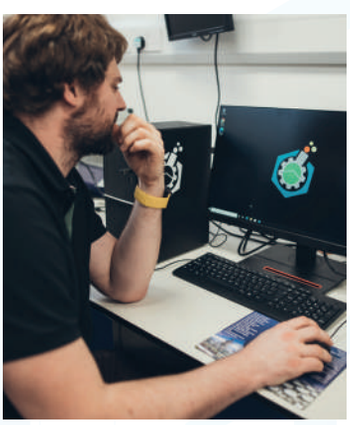
Supercritical Fluid Extraction (SFE) is commonly used to extract compounds from solid botanical material using a supercritical fluid. The supercritical CO_2 can be modified using a co-solvent (usually methanol or ethanol), to help change the polarity of the mobile phase. Extraction conditions for supercritical CO_2 are above the critical temperature of 31 °C and the critical pressure of 74 bar.

These are examples of frequently used applications of bulk-scale supercritical fluid extraction:

- Extraction of liquids and materials from a solid or semisolid sample (for example, tea extracts, nut-oil extracts).
- Extraction processes isolating food and botanical compounds that require a clean and safe extraction process, which does not leave any residual materials in the collected product.

Supercritical fluid extraction separates and fractionates components according to polarity, log P (the ratio of concentrations of a compound in the two phases of a mixture of two immiscible solvents at equilibrium), and molecular weight. These properties render supercritical fluid extraction predictable and scalable from analytical to process applications. When using a high-pressure separator, such as those provided with a Core Separations SFE system, you can manipulate the pressure and temperature of the supercritical CO_2 to selectively extract and collect the desired material.





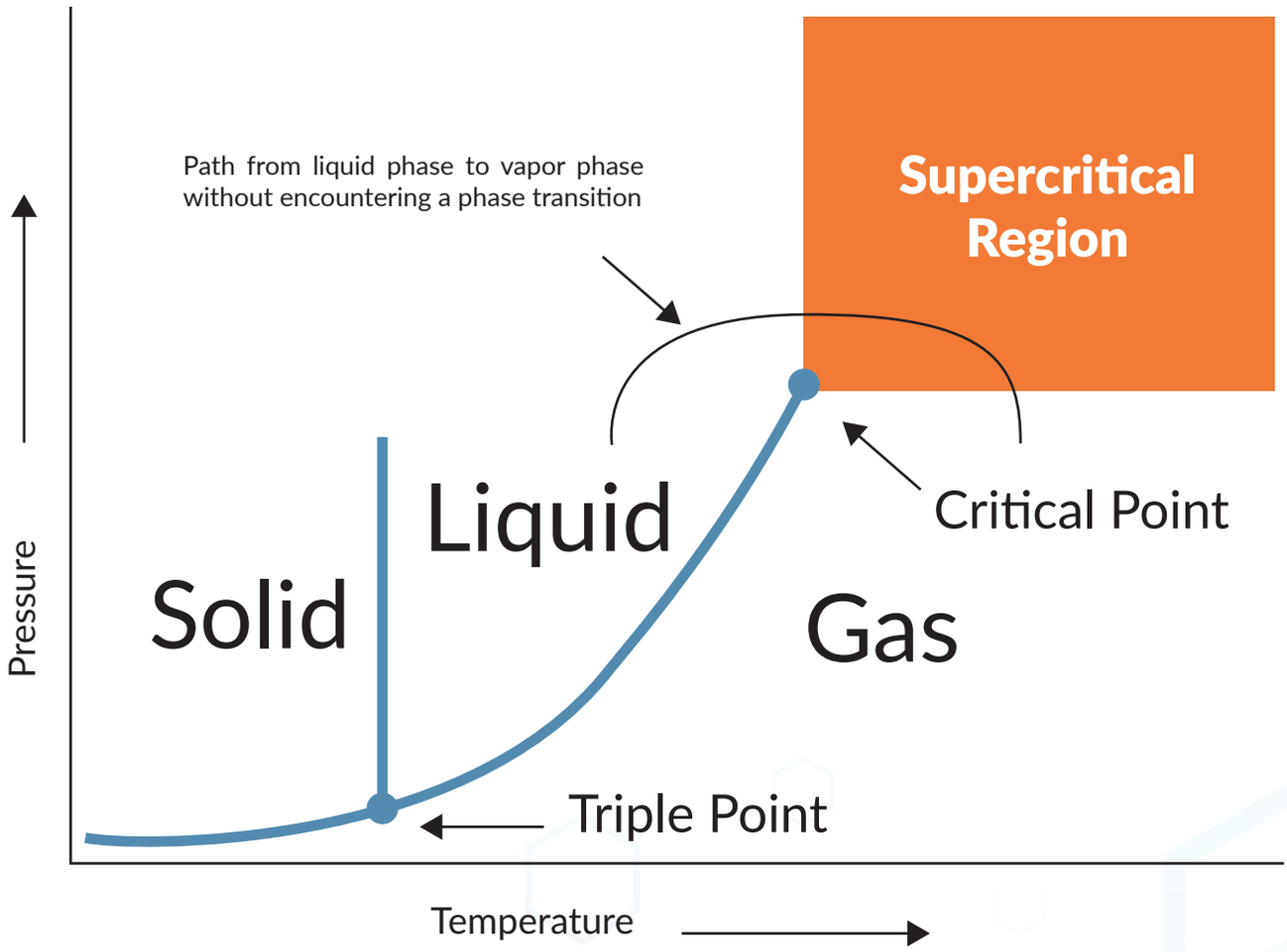
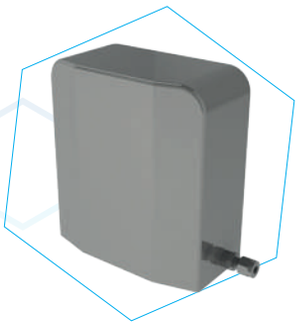


Figure 1–1: Supercritical CO_2 is tuneable without changing phases

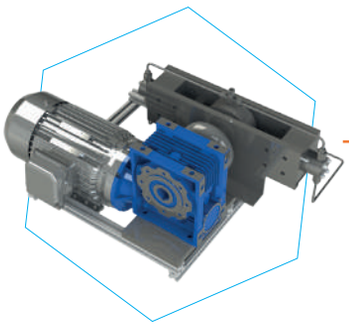
During extraction and collection, by raising the pressure of the supercritical CO_2 flow in the system, you increase a system's capability to extract polar compounds. Adding a co-solvent, such as ethanol, additionally increases this capability to collect more polar compounds.

02. Components



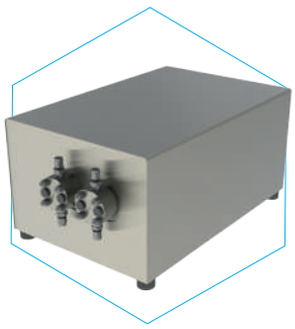
Flow Meter

The mass flow meter, which measures the liquefied carbon dioxide mass input to the high-pressure CO_2 pump. The feedback from the mass flow meter controls the pump and maintains an accurate and consistent CO_2 flow rate.



P-500 high-pressure pump (CO_2)

The high-pressure CO_2 pump, delivers the liquefied carbon dioxide to the system. Its key features are: dual pump heads with cooling chambers for removing the heat of compression, which stops the CO_2 from converting to a gas as it is drawn into the pump. A pressure relief safety device is installed, to prevent high-pressure damage to pump components.



P50 High Pressure Pump (co-solvent)

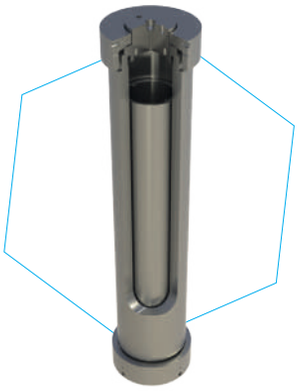
The co-solvent pump, delivers co-solvent modifier into the system. As with the CO_2 pump its key features are dual pump heads to aid in pulseless flow.

Cooling

The cooling-bath, provides a continuous supply of fluid at a constant temperature and volume. Its primary function is to circulate temperature-controlled coolant through HE1 and the high-pressure CO_2 pump heads, and also to cool and maintain the liquid state of the inlet CO_2, removing the heat of compression caused by pump operation. For product details and operating instructions, refer to the chiller unit's operator's guide.

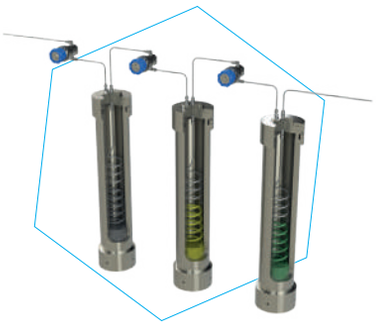
Heat exchanger

Heats or cools the mobile phase to a method-specified temperature before the flow enters the high-pressure extraction vessel or the pump.



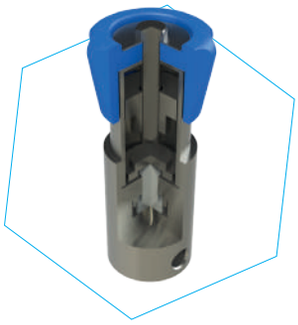
Extraction Vessels

The high-pressure extraction assembly, comprising one or two, stainless steel extraction vessels of 1-L and 5-L volume, which are equipped with hand tight caps for simple opening and closing. The caps seal is a PTFE "C" cup, which is fitted with a spring that is energised under pressure, forcing the seal's inner lip to contact the threaded cap. The seal's outer lip contacts the inner-vessel wall, forming a pressure seal. The caps at each end of the vessel, along with a seal, have a frit assembly, to provide even distribution of fluid during introduction. Each vessel has a pressure gauge which is used for monitoring the internal pressure within the vessel's chamber. A liquid jacket encases the vessel and maintains the supercritical temperature of the mobile phase within the sample matrix.



Collection Vessels (Cyclones)

The high-pressure collection assembly, comprising of upto three stainless steel, collection-separation cyclones that are connected in a serial flow, and serve as fraction collectors. When the MBPR's are properly set, compounds precipitate from the solution as the pressure across the three cyclones drops, and the CO_2 loses its solvating power. The flow path from the final cyclone exits to the vent or an optional CO_2 recycler.



MBPR

The manual back-pressure regulators (MBPR's), which control the working pressure in the cyclones, to prevent product loss during collection due to the rapid expansion of CO_2. The system is configured with one MBPR per collection cyclone. On a system fitted with three collection-separation cyclones, the manual back-pressure regulator of the first cyclone is normally set to the highest pressure. The second and third regulator valves are then set to create a decreasing pressure gradient that reduces the final pressure to near atmospheric conditions (or approximately 55 bar, if using the optional recycler), while enabling you to isolate and collect extracted compounds within the second and third.

03. Introduction

The Core Separations high pressure research systems are designed for applications where CO_2 is compressed and used as an extraction medium. All wetted parts are either 316 stainless steel or 17-4PH stainless steel, with PTFE graphite seals as standard.

Pressure Range
0-10,000 psi

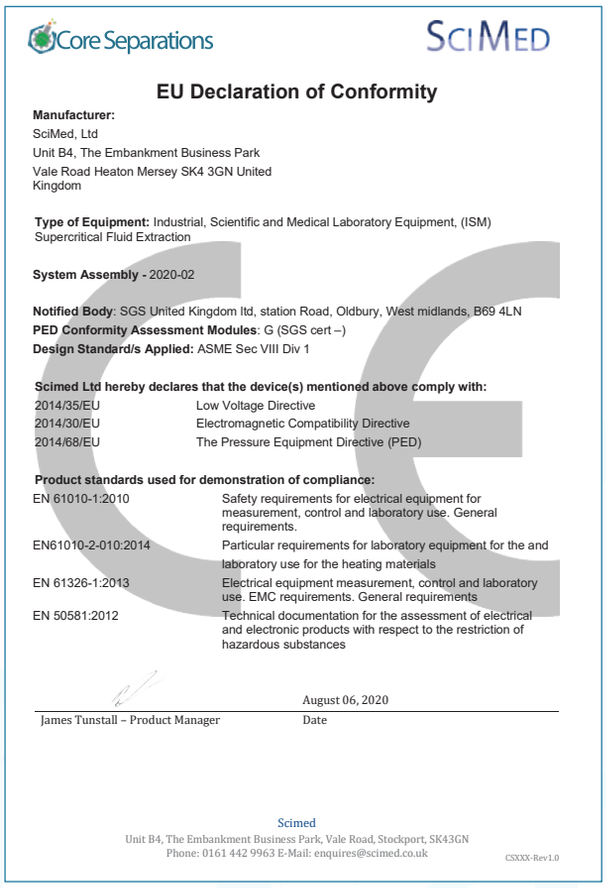
The standard connection is a Swagelok 2-part ferrule for 1/4" O.D. tubing up to 10,000 psi.

The High-Pressure skid is mounted on wheel to allow a degree of flexibility. However, the wheels should be locked off by raising the self-levelling rubber feet before use.

3.1. EC Mark and Certification - Declaration of Conformity

The Core Separations high pressure systems described in this manual are manufactured in compliance with the following Directives.

1. 2014/68/EU (Pressure Directive)
2. 2006/42/EC (Machine Directive)
3. 2014/35/EU (Low Voltage Directive)
4. 2004/108/EC (EMC directive)



Core Separations SciMed

EU Declaration of Conformity

Manufacturer:
SciMed, Ltd
Unit B4, The Embankment Business Park
Vale Road Heaton Mersey SK4 3GN United Kingdom

Type of Equipment: Industrial, Scientific and Medical Laboratory Equipment, (ISM) Supercritical Fluid Extraction

System Assembly - 2020-02

Notified Body: SGS United Kingdom ltd, station Road, Oldbury, West midlands, B69 4LN
PED Conformity Assessment Modules: G (SGS cert –)
Design Standard/s Applied: ASME Sec VIII Div 1

Scimed Ltd hereby declares that the device(s) mentioned above comply with:

2014/35/EU	Low Voltage Directive
2014/30/EU	Electromagnetic Compatibility Directive
2014/68/EU	The Pressure Equipment Directive (PED)

Product standards used for demonstration of compliance:

EN 61010-1:2010	Safety requirements for electrical equipment for measurement, control and laboratory use. General requirements.
EN61010-2-010:2014	Particular requirements for laboratory equipment for the and laboratory use for the heating materials
EN 61326-1:2013	Electrical equipment measurement, control and laboratory use. EMC requirements. General requirements
EN 50581:2012	Technical documentation for the assessment of electrical and electronic products with respect to the restriction of hazardous substances

August 06, 2020
James Tunstall – Product Manager — Date

Scimed
Unit B4, The Embankment Business Park, Vale Road, Stockport, SK43GN
Phone: 0161 442 9963 E-Mail: enquires@scimed.co.uk
CSXXX-Rev1.0

3.2. Guarantee

Core Separations products are under warranty from:

1. Defects in their construction and materials for a period of (1) year from the time they leave the factory.

2. This guarantee is limited to the repair and replacement of parts or products that Core Separations deems were defective at the time of delivery.

3. All the products covered by this limited guarantee must be returned with freight paid for inspection, repair, or replacement by the manufacturer.

4. This limited warranty is the only form of valid guarantee and replaces any other form of explicit or implicit warranty, including any guarantee of fitness for sale or any particular purpose.

5. The manufacturer refuses any such liability with this statement.

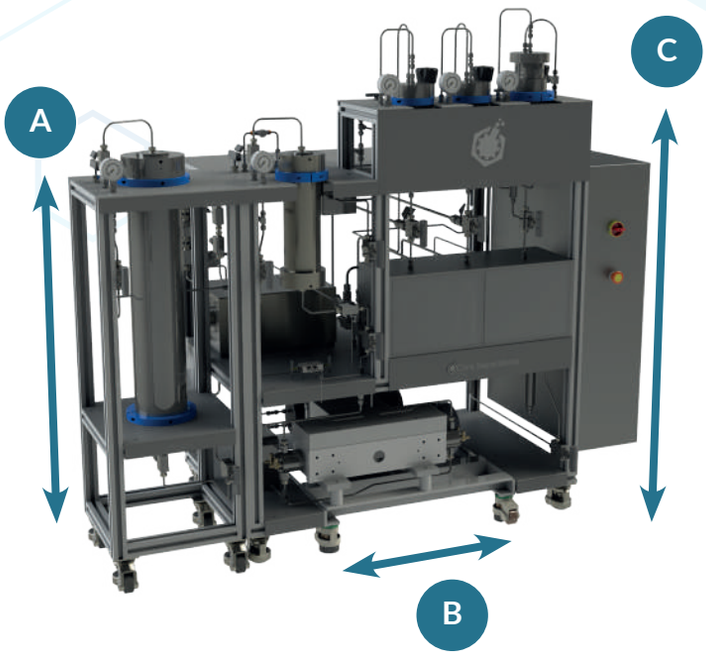
6. Faulty products will only be repaired or replaced according to these terms. Core Separations is not liable for any further loss, damage, or expense, including accidental or indirect damages caused directly or indirectly from the sale or use of these products.

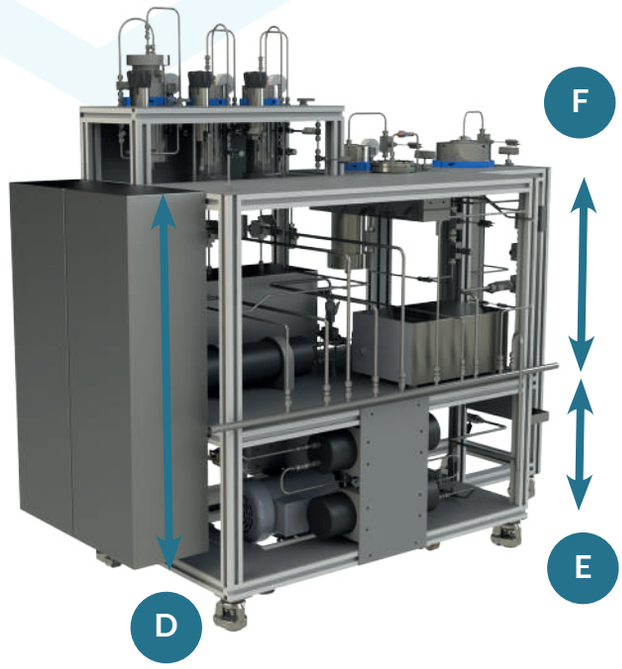
7. Any unauthorised use of spare parts that were not manufactured by Core Separations automatically invalidates this guarantee, which is subject to compliance with the instructions for installation and operation provided. There are no additional guarantees other than the guarantees described above.

3.3. Technical characteristics

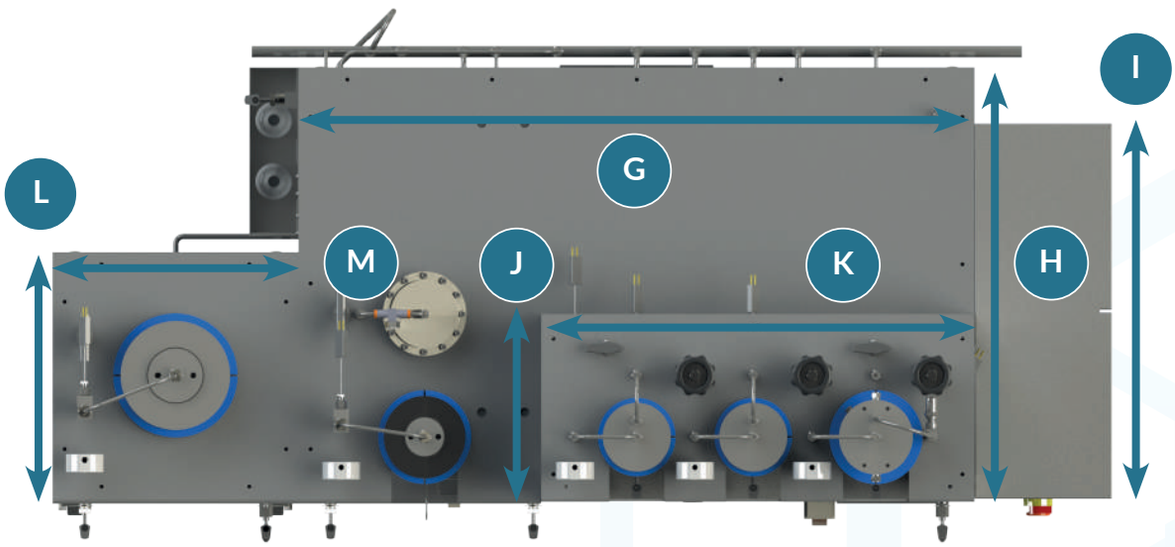
The main dimensions and specifications for the Core | **Extraction** systems are as follows:

A (mm)	B (mm)	C (mm)	D (mm)	E (mm)	F (mm)
1117	650	1325	900	390	594





G (mm)	H (mm)	I (mm)	J (mm)	K (mm)	L (mm)	M (mm)
1100	700	600	300	705	400	400



3.4. Local Conditions

Parameter	Tolerated Values
Room Temperature	From -10 °C to + 50 °C
Storage Temperature	From 0 °C to +50 °C
Humidity	From 20 % to 80 %

The high-pressure skid is mounted on a wheel to allow a degree of flexibility. However, the wheels should be locked off by raising the self-levelling rubber feet before use.

3.5. Power Requirements

The main Core | Extraction systems provided for the EU market are designed to use:
Power supply (3 phase). 415V 3~N 50Hz

Depending upon the options chosen the system can require the following amperage/kilowatts (KW)

Component	Amperage (FL) 240 / 415 V	KW
P500 Pump	6 / 3	4.5
P50 (co-solvent Pump)	5.6 / 3	0.72
Pre-heater	8.3 / 2.8	2
Vaporiser	8.3 / 2.8	2
Vessel 1 (5L)	8.3 / 2.8	2
Vessel 2 (1L)	4.2 / 1.4	1
Cyclone 1 (1L)	4.2 / 1.4	1
Cyclone 2 (1L)	4.2 /1.4	1
24VDC PS	3.6 / 2	1.5
Maximum	48.5 / 20.6	15.72

3.6. Vibration

Core Separations systems do not produce hazardous vibrations under normal use, provided the installation and assembly instructions in this document are carried out properly.

3.7. Noise

The equipment was designed and built to reduce the generation of noise at source, as far as its application and method of use allows this. The noise measured is below the minimum level envisaged in current legal standards.

3.8. Heating and Cooling Circuits

The system is designed to reach temperatures up to 150 °C. All the pressure vessels have been insulated to improve heat efficiency and to protect the user from hot surfaces. However, some of the pipework may get hot and therefore signs have been provided to warn the user.

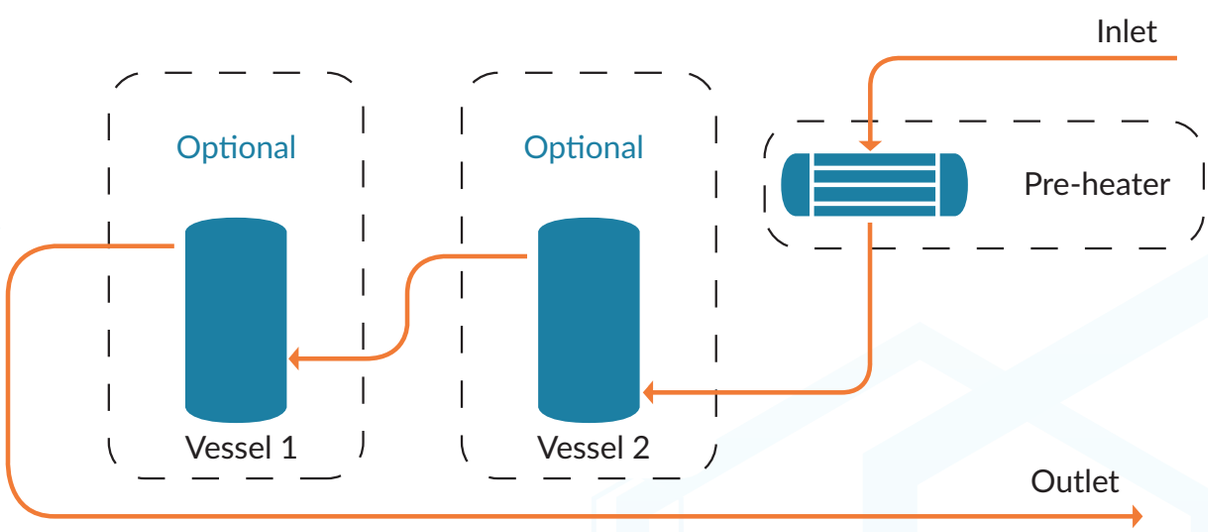
3.8.1. Electric Heating

Electrical heating is common on supercritical extraction systems. If the system specification is supercritical only, the heating will be predominantly electrical. The following devices use electrical heater bands and/or heater cartridges:

1. Pre-heater – The power of this heat exchanger is determined by the flow rate of the CO_2 pump.
2. Extraction vessel – The power of the heater band is determined by the volume of the vessel and the CO_2 flow rate.
3. Vaporiser – The power of this heat exchanger is determined by the flow rate of the CO_2 pump.
4. Cyclones – The power of the heater band is determined by the volume of the vessel and the CO_2 flow rate.

3.8.2. Liquid Heating and Cooling (Subcritical System) – Optional

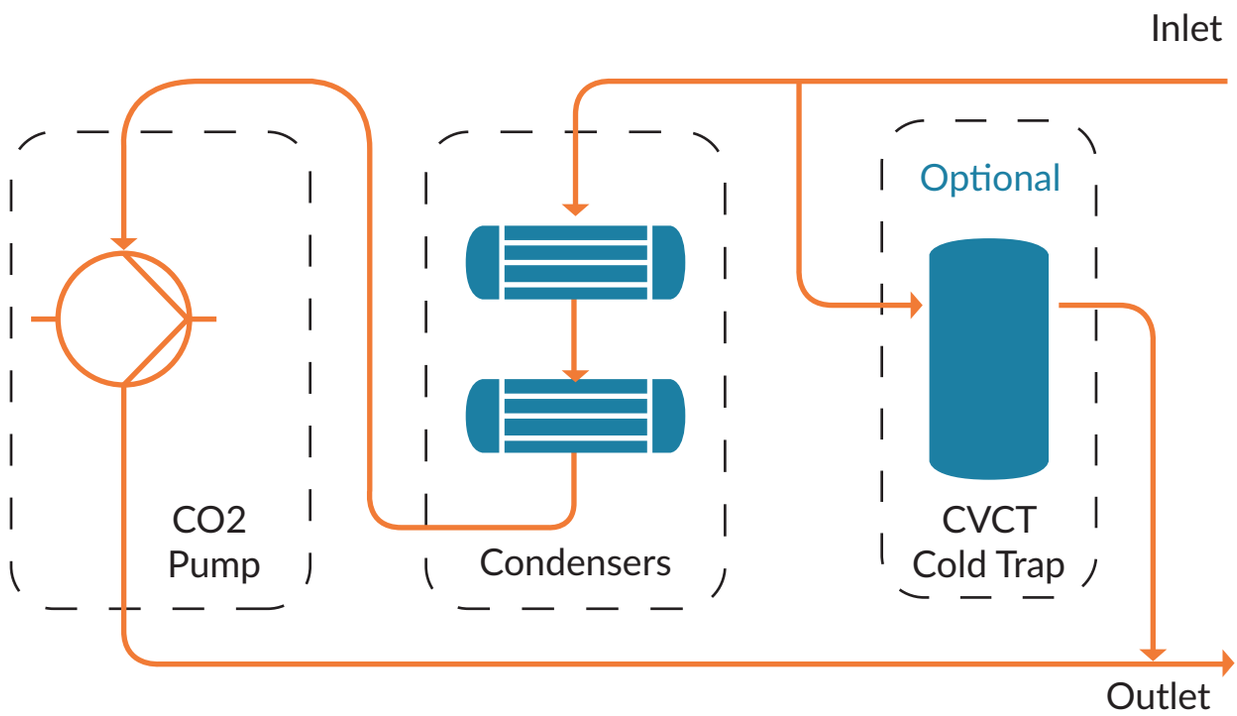
Subcritical CO_2 systems have vessels and heat exchangers which are heated and cooled using a thermal fluid or water glycol mixture. The heating and cooling loop will be connected in series starting in the pre-heater, through Vessel 2 (normally 1 or 0.5 L) and ending in vessel 1 (5 or 10 L).



The specification of the re-circulating unit is dependent on the conditions and the system, so may vary from system to system. In almost all cases the unit will be controlled via the SFX software (See software manual).

3.8.3. Pump Cooling Circuit

A common loop found within all super and subcritical CO_2 systems is the cooling circuit. The closed loop recirculates a water glycol mixture (50/50) around the condenser (heat exchanger) and through the pump head. This is to ensure the incoming liquid CO_2 remains a liquid, and does not enter the gas phase which would reduce the efficiency of the pump.



The high-pressure piston pump contains 4 copper cartridges located in the head of each piston assembly. Its function is to remove the residual heat which is generated through the heat of compression.

Copper cartridges located in the pump heads.

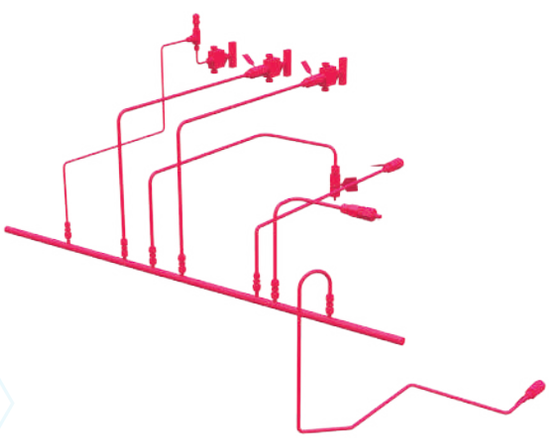
04. Safety

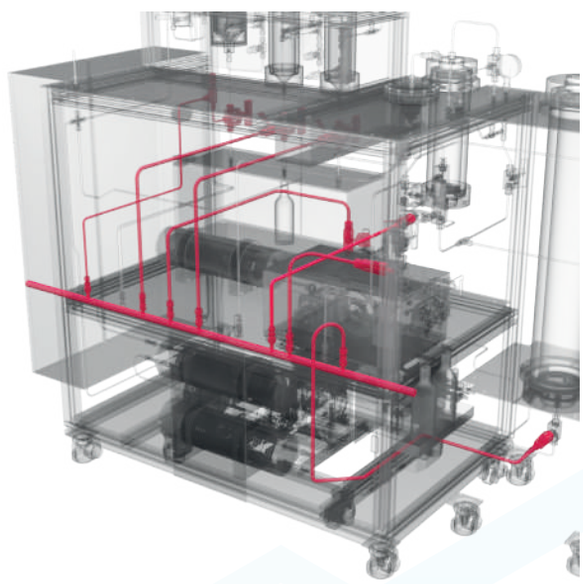
4.1. User Responsibilities

The ultimate responsibility for the safe operation of this system is with the user. The pressure limits of the system MUST be observed and no attempt to increase these limits should be made by altering components, which are not recommended by the manufacturer.

Installation of the system MUST:

1. Include the use of appropriate safety accessories. If these are provided, they must be installed in accordance with the instructions provided by the manufacturer.
2. The safety accessories should be directed into a safe area. This is commonly achieved by directing the common vent pipe located at the rear of the system, into a defined area away from the operators. Consideration should be taken when defining the safe area. Please consult a Core engineer if required.
3. The system must be immobilised using the self-levelling feet during use.
4. The system should be installed in a well-ventilated lab.





Above shows the approximate locations of the safety devices. Some devices are only present with the optional additions of vessels and pumps.

When operating the system, the user MUST:

1. Inspect the equipment and check that it is in good working order.
2. Ensure all users are adequately trained to use the equipment.
3. Ensure the system alarm limits are not altered to prevent over pressurisation.

4.2. Unpacking the System

To aid in secure transport almost all the components are secured to the skid plates. The P500 or P1K (500g/min / 1kg/min) are supplied on their own trolleys, which wheel in and out of an opening located at the base of the skid.

Installation of the P500 after transport

The pipe work to the pump is removed before packing and transportation, and requires re-installation before the system is re-commissioned. When unpacking the crate, the pump can first be removed from underneath the skid using a forklift, followed by the rest of the system.

4.3. Installation

The system must be installed within a well ventilated space. This is important if using a fluid such as carbon dioxide, as uncontrolled release of the CO_2 into a room could cause asphyxiation.

The area and environment that the high-pressure system operates in must be clearly signposted and prohibited to unauthorised personnel. Prior to starting up the system, the operator or operators are required to check:

1. That the system has the correct power supply.
2. That the electrical parts are correctly and adequately protected.
3. That the high-pressure pipe and fittings do not exhibit signs of abrasion or excessive wear. Any defect, damage or reasonable doubt that might arise before or during the operation must be reported and verified by qualified staff. Should this happen, the system must be stopped immediately, and the pressure brought down to zero.

System should be located close to:

1. Laboratory hoods
2. Exhaust fans
3. Safe outside space

Installation must also include provisions for when a safety device is activated:

1. Installation of extension pipework from the common vent line, directed to a safe discharge area.
2. Appropriate ear protection in case of rupture disk activation, especially for operators standing near the system.

Do not attempt to remove or alter any part of the system, unless instructed in this manual. No unauthorised personnel should tamper with the equipment without contacting the manufacturer first.

4.4. Fittings

The fittings used on this system are a mixture of Swagelok and HIP, and both 1/8" and 1/4" high pressure fittings have been used. The vessels and pumps have been machined using dimensions provided by HIP. Interchanging fittings with other manufacturers is not recommend.

Swagelok Guidelines

https://www.swagelok.com/downloads/webcatalogs/en/ms-13-151.pdfa

HIP

https://www.highpressure.com/products/valves-fittings-tubing/taper-seal-valves-fittingsand-tubing/assembly-procedure/

4.5. Maintenance and Training

It is the responsibility of the user to keep the equipment in good condition. Serious consequences can result from poor maintenance and mis-operation of this extraction system. Frequent checks should be carried out ensuring the equipment is free from:

1. Cracks
2. Corrosion
3. Bulging in the outer wall (Pressure Vessels)

If any of these are observed, stop using the system immediately and contact a Core engineer for advice and further analysis.

We also recommend that all users familiarise themselves with the Swagelok and HIP fittings manual and follow their instructions when assembling the pipe work.

4.6. Personal Protective Equipment (PPE)

As part of the training procedure, we recommend the use of the following PPE when operating the vessels, which should be included in any operating instructions if the vessel is incorporated into an assembly.

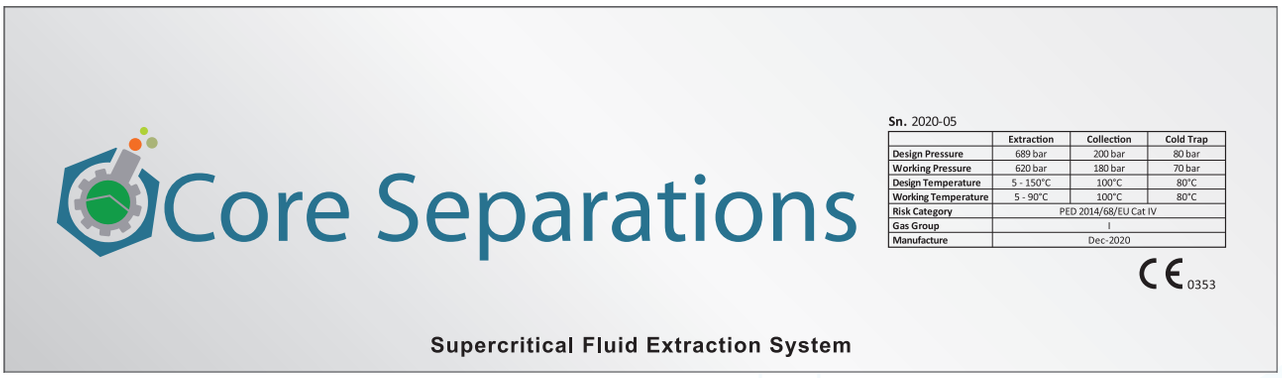
Wear Safety Googles

Wear Gloves

4.7. Product Labels

An exact description of the equipment, serial number, and technical data is included on the system. This allows technical personnel to quickly identify the equipment, allowing for a fast and efficient service (if needed).

The identification data is on the plate attached to the equipment, as shown below:



Under no circumstances should labels be removed from the equipment.

05. Pipework

5.1. Pipe Work on the Extraction Vessels (EV series)

Ensure the system is at atmospheric pressure before proceeding!

This system uses a variety of widely available high-pressure fittings (HIP and Swagelok). It is the user's responsibility to be familiar with the installation and removal of these fittings before attempting to manipulate the system. Manufacturer guidelines for the correct operation of these fittings are found here:

Swagelok Guidelines
https://www.swagelok.com/downloads/webcatalogs/en/ms-13-151.pdf

HIP
https://www.highpressure.com/products/valves-fittings-tubing/taper-seal-valves-fittingsand-tubing/assembly-procedure/

It is important to distinguish the difference between the two types of fittings used. The HIP fittings used on this system can be tightened using the following torque settings:

Fitting Type	Typical Locations	Torque Setting
AF2 (1/8")	Co-solvent valves	Initial Compression – 10 ft/lb Tighten Connection – 25ft/lb
AF4 (1/4")	Main valves, Tees Blocks and Extraction Vessel Heads	Initial Compression – 30 ft/lb Tighten Connection – 50ft/lb

The Swagelok high pressure fittings (and equivalents) use a two-part ferrule system. Creation and tightening of these fittings **DOES NOT** require a torque wrench. Manufacturer instructions should be followed to prevent incorrect installation of these fittings. If in doubt, please contact a Core Separation engineer for advice.

Typical Tools Required

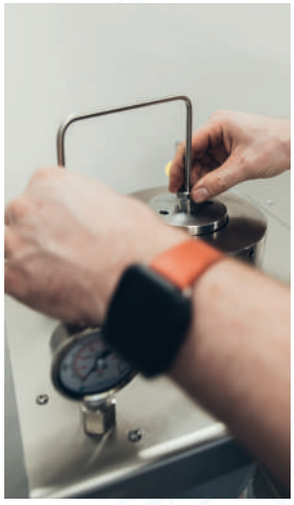
Imperial Spanners: 7/16" (Swagelok (1/8"); 9/16" (Swagelok ¼"); 1/2" (HIP 1/8"); 13/16" (HIP ¼")

Pin Spanner: 1 3/4" (EV500, EV1L), 2 1/4" (EV5L, EV10L)

1	Ensure the system has no pressure before attempting removal of the pipework. **See 08. Venting the system**
2	Brace the thermocouple block with a 1” spanner and loosen the AF4 HIP fitting with a 13/16” spanner. Ensure you take care during this operation not to bend the bend pipework.
3	Using the Pin spanner provided, brace the inner cap of the EV series vessel. Using the 13/16” spanner turn the HIP AF4 Nut counter-clockwise.
4	The pipework can then be removed from the vessel and the vessel heads can be dismantled. **See 06. Opening and Closing the Extraction Vessel (EV series).**

Step 2 – Brace TC block

Step 3 – Brace Inner Cap



Step 4 – Remove Pipework

See Lid Removal

06. Extraction Vessel

6.1. Opening and Closing the Extraction Vessel (EV series)

It is necessary the user becomes familar with opening and closing the vessels between extraction runs, as material needs to be charged and discharged between process runs. It may also be necessary to clean the inside of the vessels to prevent cross contamination when changing materials. The operation of the opening and closing the lids is described below:

1	Ensure the system has no pressure before attempting removal of the pipework.
2	Using the lid bars (3/8”) provided or by hand, unscrew the vessel cap by turning counter-clockwise on vessel.
3	Lift the inner cap from the vessel. This may be tight due to the energised sprung seal.
4	Clean the seal on the cap with a soft cloth. The cloth may be soaked in an appropriate solvent if required. Inspect the seal for cuts or abrasions.
5	If using a basket, pack the basket with the raw material and insert into the vessel.
6	Spray the threads with a dry Teflon lubricant. Install the cap into the top of the vessel by turning clockwise. The cap should be turned, by hand, until the bottom of the cap is in contact with the top of the vessel.
7	Re-attach the pipework

Step 2 – Rotate the cap counter-clockwise

Step 3 – Remove inner cap

Step 4 – Ensure the vessel is clean

6.2. Installing the Extraction Vessel Seals

The extraction vessel uses a high pressure sprung seal to retain the pressure in the vessel. The seal makes contact radially around the vessel wall. Our vessels also have small hole located just above the seal, this is known as a “weep hole”. If the seal begins to fail, the CO_2 will release via this hole indicating the seal requires changing.

1	Installation of the seal depends on whether the filter plates have been installed. See 6.3
2	If the seal retainer plate is installed, then firstly remove this plate by unscrewing the countersunk bolts using a 1/8” Allen key.
3	Remove the old seal by pulling it towards you.
4	Ensure the seal recess is clean before installing a new sprung seal.
5	Press the new seal into position, ensuring the spring is facing the inside of the vessel and NOT towards the head.
6	Re-assemble the seal retainer.

Step 1 – Remove seal retainer

Step 3 – Remove the old seal

Step 5 – Install new seal

Step 6 – Reassemble seal retainer

Ensure the system is at atmospheric pressure before proceeding!

6.3. Installing filter assembly

The extraction vessels can either be filled directly or via a basket. In both cases the filters in the heads must be installed.

1	The filters compromise of 3 items; PTFE O-ring seal, sintered metal plate and a seal retainer.
2	The PTFE O-ring is first placed into the recess of the head.
3	The Sintered disk is then placed on top of the O-ring.
4	The seal retainer is then placed on top of the sintered disk and the 4 countersunk screws are tightened using a 1/8” Allen key until tight.

Head without filter assembly

Step 2 – Place O-ring into the recess

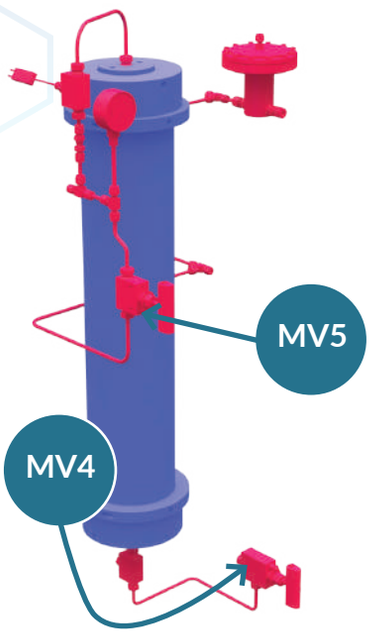
Step 3 – Place sintered disk on O-ring

Step 4 – Tighten seal retainer plate

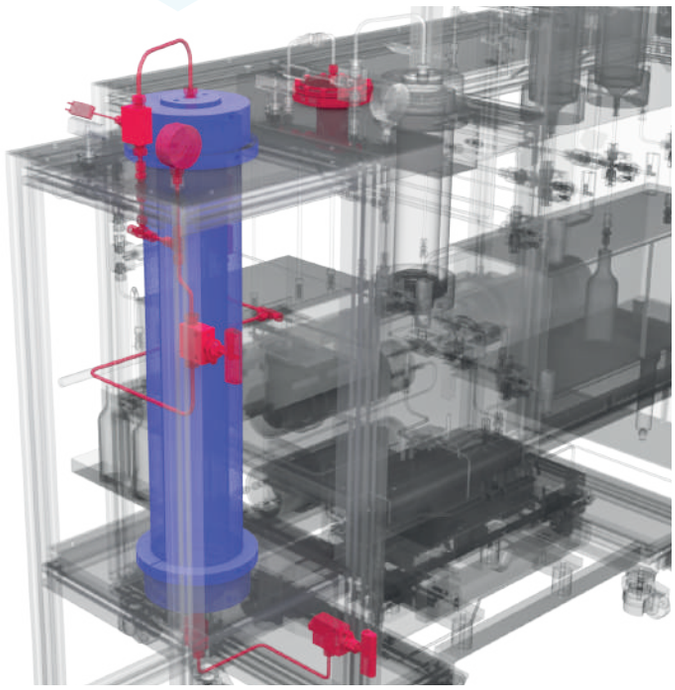
Once the filter plates have been installed, the material can either be packed directly into the vessel or into the basket. Once packed the lid can be re-installed (See 6. Opening and Closing Pressure Vessel).

07. Set-up

7.1. Setting up 5L Extraction Vessel (Optional)



Approximate locations of the inlet and outlet valves for the 5L



Approximate locations of the inlet and outlet valves for the 5L

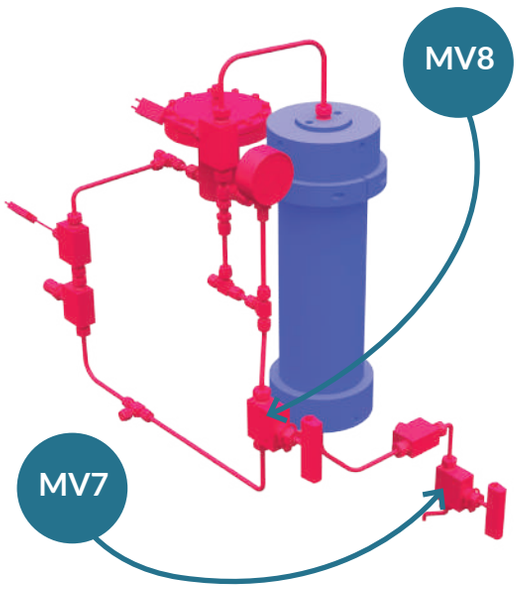
To perform an extraction, the system should be fully assembled with the biomass charged into the extraction vessel. The lids should all be closed, and the pipework assembled. The following valves should be open, unless otherwise stated with the remaining valves closed.

Valve	Description	Position
MV1	CO_2 Inlet (located bottom far right of the system)	Open
MV4	5/10 L Extraction Vessel - V1 Inlet	Open
MV5	5/10 L Extraction Vessel - V1 Outlet	Open
MV12	Vent or Recycle Mode (Process dependent)	Recycle/Vent

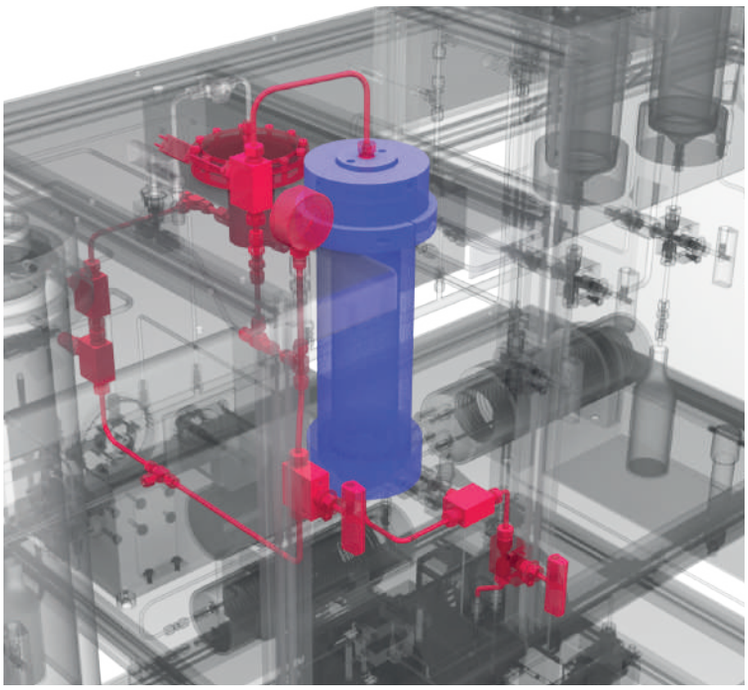
Table 2: 5 L Extraction Vessel V1

Once the position of the valves has been confirmed the system can be started.

7.2. Setting up 1L Extraction Vessel (Optional)



Approximate locations of the Inlet and outlet valves for the 1L



Approximate locations of the inlet and outlet valves for the 1L

To perform an extraction, the system should be fully assembled with the biomass charged into the extraction vessel. The lids should all be closed, and the pipework assembled. The following valves should be open, unless otherwise stated with the remaining valves closed.

Valve	Description	Position
MV1	CO_2 Inlet (located bottom far right of the system)	Open
MV7	0.5/1/2 L Extraction Vessel - V1 Inlet	Open
MV8	0.5/1/2 L Extraction Vessel - V1 Outlet	Open
MV12	Vent or Recycle Mode (Process dependent)	Recycle/Vent

Table 3: 1 L Extraction Vessel V2

Once the position of the valves has been confirmed the system can be started.

08. Venting

Before starting any operation on the system ensure that there is no pressure present in the system.

The system is provided with calibrated pressure gauges (ISO 10012:1013), which are attached to all the pressure vessels. This allows the user to easily determine whether the system still contains pressure.

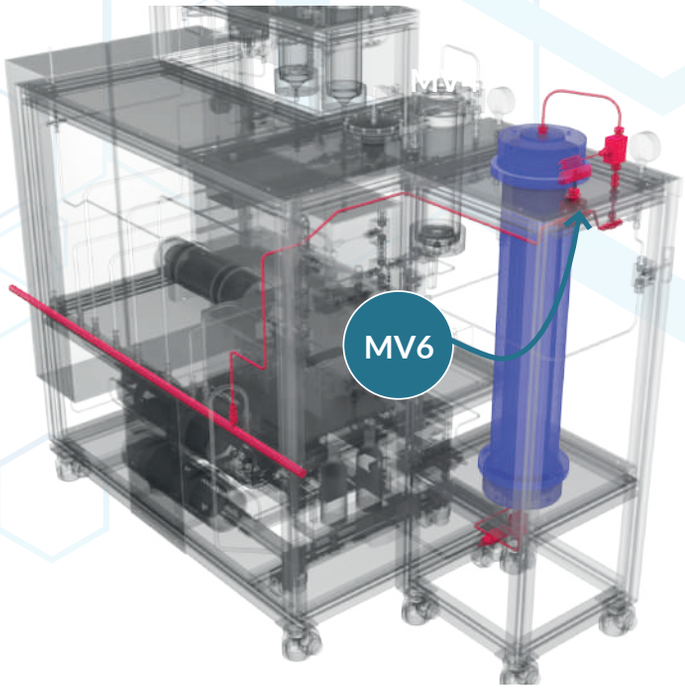
Each extraction vessel has a dedicated vent valve that is connected to the common vent line, located at the back of the system to remove the pressure. The number of vent vales depends on the configuration of the system.

The collection vessels can also be depressurised through the main vent line. The following instructions outline the two operations for removing pressure from these vessels safely.

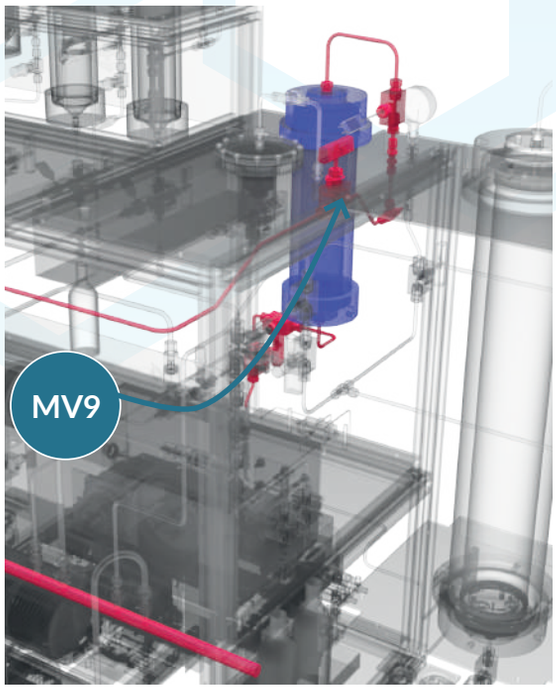
Ensure the main vent line has been directed into a safe and well-ventilated area, before attempting to vent the system.


1500
100
50
200
500
2500
3000
bar
psi
Core Separations

8.1 Venting Extraction Vessels



(If option on system)
5L/10L Extraction Vessel – Vent Valve Location



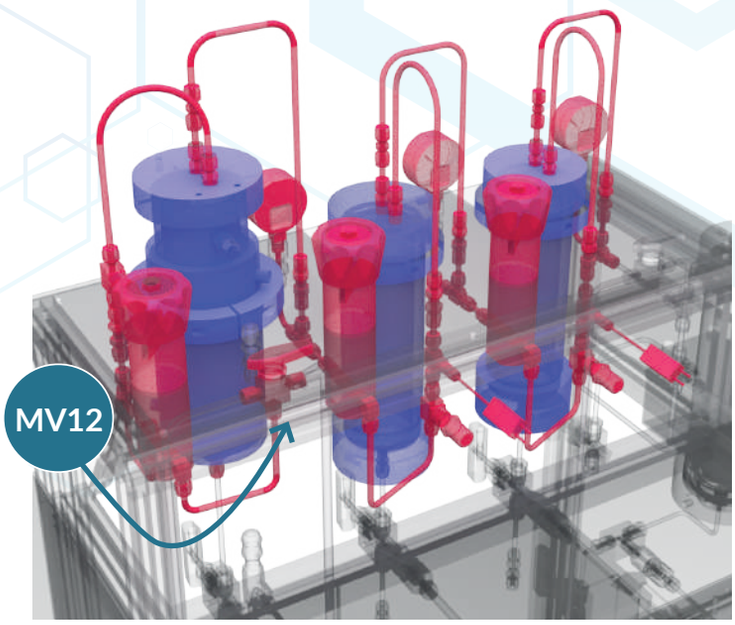
(If option on system)
0.5L/1L/2L Extraction Vessel – Vent Valve Location

The operation is identical for all variants of the Core | **Extraction** systems.

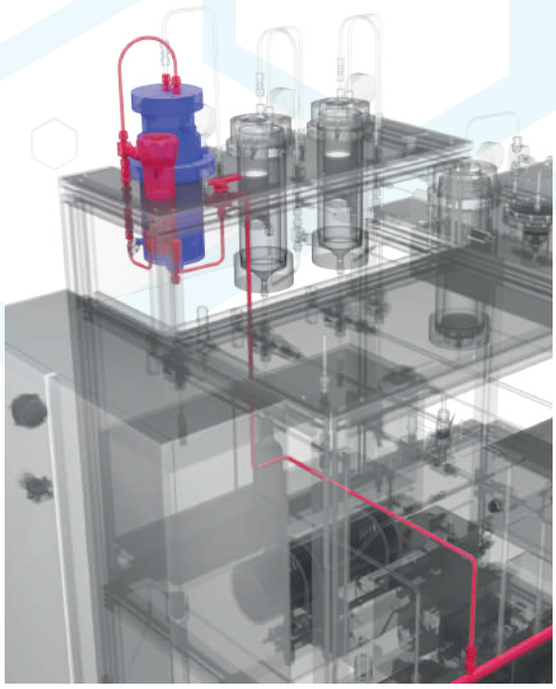
1	To begin draining the pressure from the system, first locate the appropiate vent valve. The approximate locations for these valves are shown in the diagrams above. MV 6 – (5/10L Vent Valve) MV 9 – (0.5/1/2 L Vent Valve)
2	Slowly open the appropriate valve to begin depressurising the vessel. **Note: Depending on which valves are open the vent valve will only drain the vessel it is attached too. Pressure could still remain in the system.**
3	Do not open the vent valve too quick as rapid decompression can damage the system – mainly around the soft seals preventing it from sealing again.
4	Continue to open the vent valve to slow remove all the pressure from the vessel. This can be checked using the pressure gauge located to the left of the vessel.

MV 6 – 5/10L Extraction Vessel Vent Valve

8.2 Venting the Cyclones



(Number of Collectors may vary)
Collector – Manual Back Pressure Regulators approx. Locations



(Number of Collectors may vary)
Collector – Vent/Recycle Valve approx. Location

Unlike the extraction vessels the collectors are connected in series to allow sequential fractionation of the products during the extraction process. They are vented through the last collector, whether that be a one collector system or a multi-collector arrangement. The process ends at a 3-way valve located to the far right of the system behind the final collector (or final cut out if collector not present). The valve allows the system to put into one of two modes.

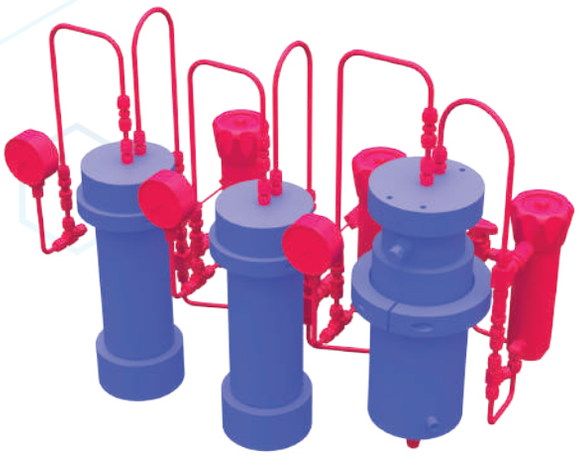
1. Recycle mode – CO_2 is recycled back round the system either *via* a closed loop recycle or *via* a storage tank.
2. Vent Mode – The flow is diverted to the vent line.

The system can be run in vent mode during an extraction to achieve pressures in the last cyclone below 45 bar. However, it is also used to drain the system once the run has been completed.

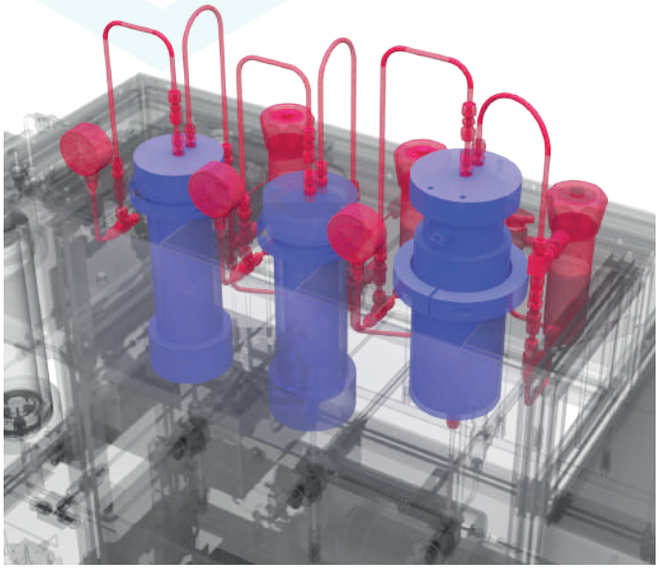
1	To begin draining the pressure from the system, first locate the vent valve. The approximate location this valve is shown in the right-hand diagram above. MV 12 – Recycle/Vent (this will be labelled on the system)
2	First ensure the manual back pressure regulators located at the back of each of the collectors are fully open. **Note: To open the manual back pressure regulators turn the knob counterclockwise.**
3	Turn MV 12 to the vent mode and the pressure will be released via the vent line located at the back of the system.
4	Once the pressure has been removed all the pressure from the vessel the cyclones can be opened. This can be checked using the pressure gauge located to the left of the vessels.

MV 12 – Collector Vent/Recycle Valve

09. Cyclones



Three Collector setup (optional)



Three Collector setup (optional)

Whether the system is set up with two or three collectors the operation is the same.

NOTE: This procedure is for the CS200-1, follow the same procedure for CS200-2, substituting in the appropriate valves.

9.1. Pre-pressurise Valve (MV8)

The pre-pressurisation valve (MV8) is used to pressurise the collectors before starting the system. If the collectors are not pre-pressurised before starting the extraction, then pressure will slowly build during the process. Pre-pressurisation can be used to prevent product carry over.

1	Ensure the vent/Recycle valve (MV12) is set to the correct position for the application.
2	If set to vent, the manual back pressure regulators should be adjusted to the desired pressure. Precise settings will alter depending on the desired flow rate and may require adjustment once the system has been started.
3	If set to recycle, the lowest pressure achievable will be the pressure of the tank. The manual back pressure regulators do not need adjustment to achieve this. **Note: this is normally 50/55 bar.**
4	Once the mode is set, the pre-pressurisation valve (MV8) can be opened.

CS Pre-pressurisation valve

CS Pre-pressurisation valve – Location behind first collector

9.2. Collection

The cyclones should be under pressure (≥10 bar) to aid in draining the material.

After an extraction, the cyclones (CS200-1 and CS200-2) will contain the material which has been extracted. If the material is a liquid it can be drained using the valve on the bottom of the cyclone, MV14 and MV15. Care should be taken when opening the valve, as liquid under pressure may rapidly be ejected.

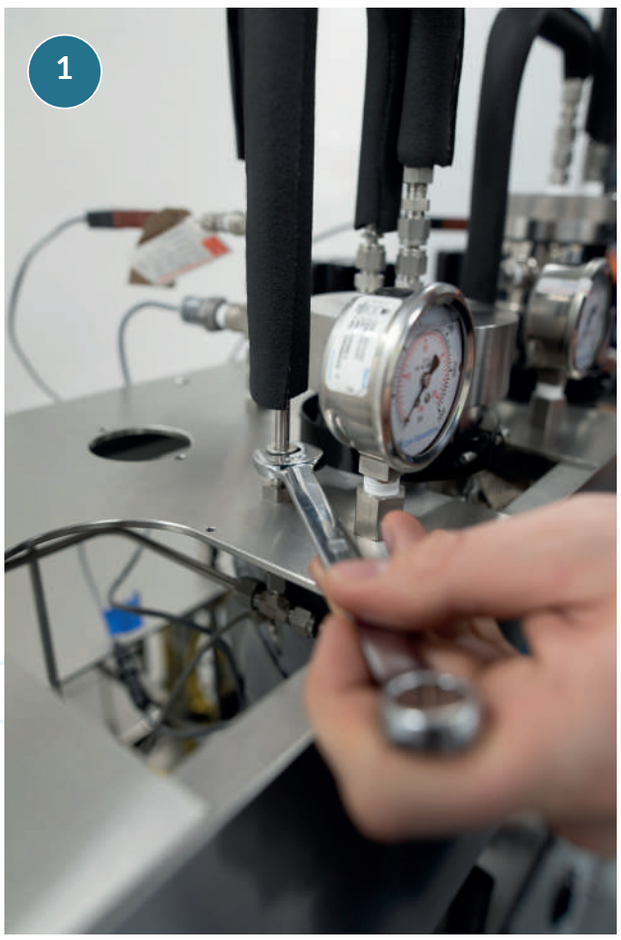
1	Place a suitable container underneath the cyclone outlet located in the vent box.
2	Open the cyclone bleed valve, MV14 (CS200-1) (or MV15 for CS200-2), located on the bottom of the cyclone until all the extracted liquid has been collected.

9.3. Depressurisation

1	To fully depressurise the collectors, ensure the system is off and the pump has stopped.
2	If in recycle mode switch the system to vent.
3	Slowly open the manual back pressure regulators, if they were used to create back pressure in the collectors
4	Once all the pressure has been removed from the collectors, they can be dismantled. This can be checked using the pressure gauges located to the left of each collector.

9.4. Disassembly

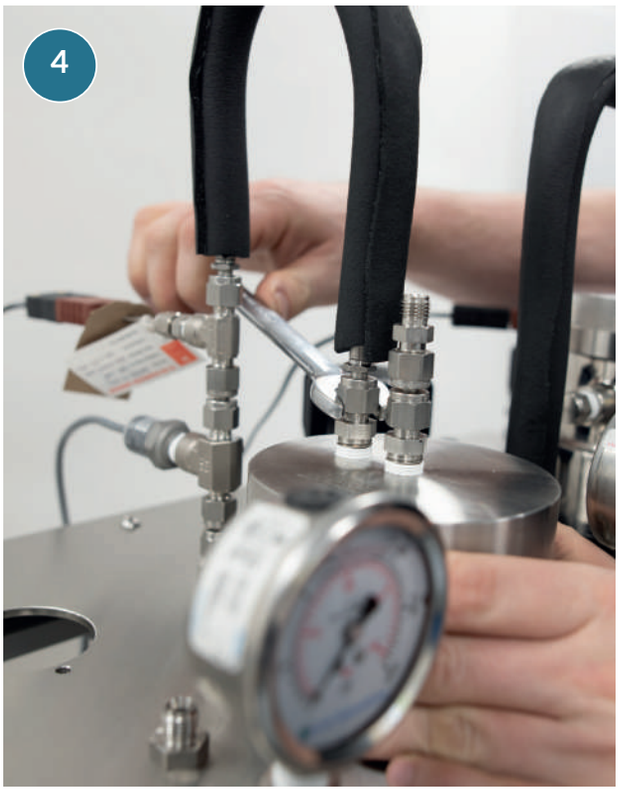
1	Once the collectors have been depressurised, remove the pipe located to the left of the collector using a 9/16” spanner.
2	To remove the central pipe, use a 1/2” spanner to brace the centre of the Swagelok union and 9/16” spanner to loosen the nut.
3	Remove the U-shaped pipe
4	Remove the flexihose located on the outlet (offset port on the head of the collector), take a 9/16” spanner and loosen the nut.
5	The flexihose can be removed by loosening the nut on the connecting fitting.
6	Once the pipes have been removed, the head can be removed using the tommy bars provided, turning the head counterclockwise.
7	When the head is removed, be careful not to damage the connected dip tube or spiral.



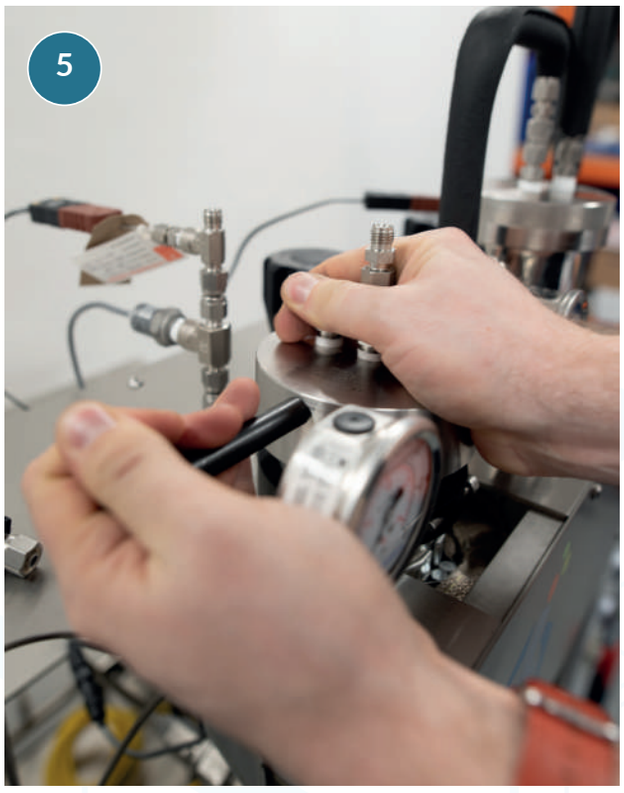
Step 1 – Loosen nut with 9/16” spanner

Step 2 – Loosen central pipe

Step 3 – Remove pipe



Step 4 – Loosen nut on flex hose



Step 3 – Remove pipe

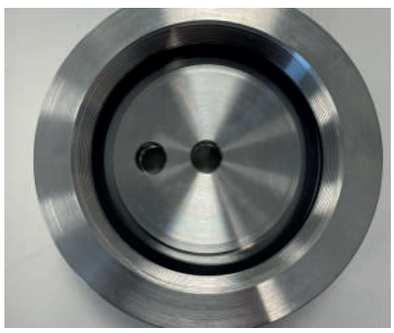
Step 4 – Loosen nut on flex hose

Ensure the main vent line has been directed into a safe and well-ventilated area before attempting to vent the system.

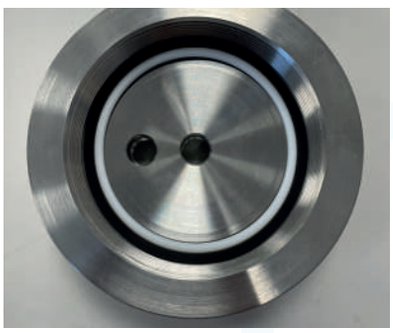
9.5. Seal replacement

9.5.1. Collector

1	If gas escapes from the weep hole located on the head of the cold trap, it may indicate that the seal needs replacing.
2	Once the head is removed, flip it upside down to reveal two seals, a PTFE O-ring, and a backup Peek seal with a square profile
3	The seals can be easily removed using an O-ring pick.



Back-up ring



PTFE O-ring

9.5.2. Cold Trap

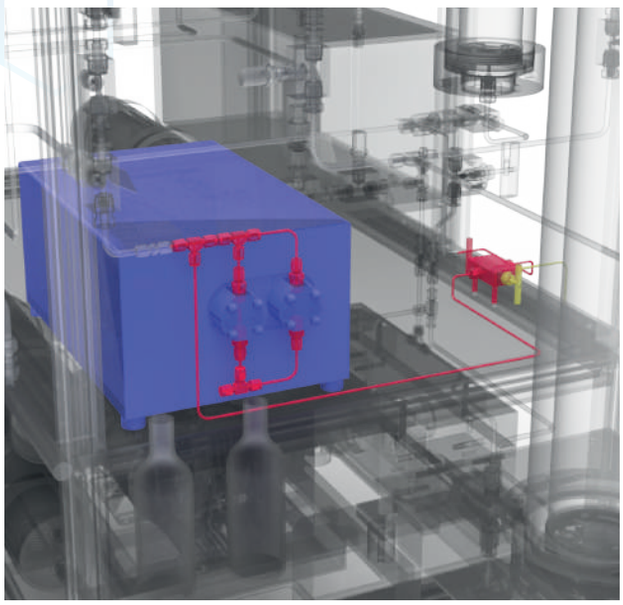
1	If gas escapes from the weep hole located on the head of the cold trap, it may indicate that the seal needs replacing.
2	Using the pin spanner provide (1 1/2”) remove the head, flip it upside down to reveal a PTFE O-ring which is seated into a recessed groove.
3	Using an O-ring pick, remove the O-ring and replace.

Seal cold trap

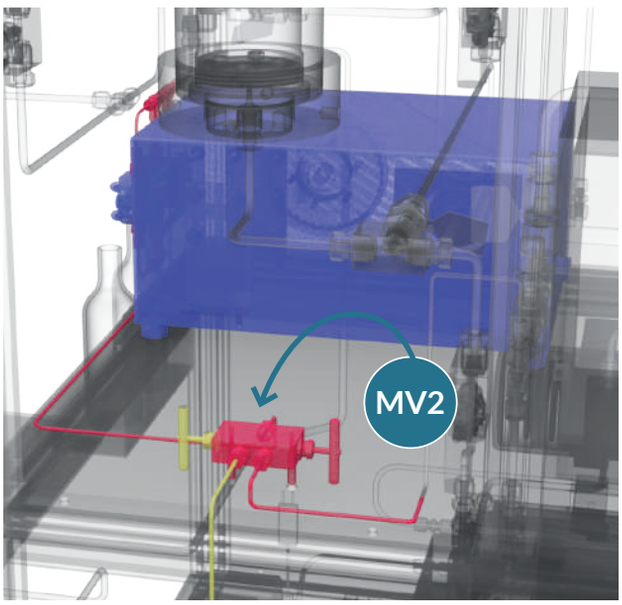
10. Co-solvent

The co-solvent pump has 3 separate options; prime, co-solvent modifier, and co-solvent makeup.

10.1. Prime



Prime valve (MV2) - (optional)

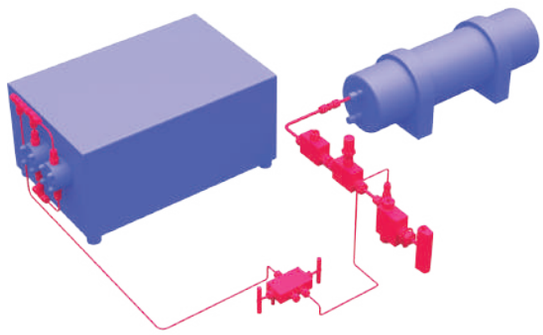


Prime valve (MV2) - (optional)

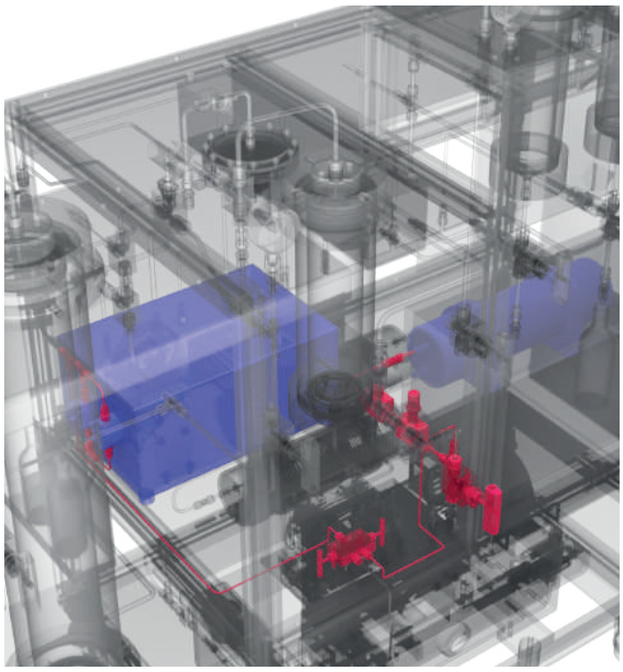
The prime function is used to expel the air from the pipe work or to flush solvent through the delivery lines. MV 2 is a 2-way 2-stem valve, the left-hand valve is used to prime the solvent lines. tt is recognisable because the drainpipe is not connected to the rest of the system, and allows the collection of the solvent into a suitable container, such as a beaker.

1	To prime the pump, place the solvent bottles in the tray below the pump.
2	Open MV2 (left hand valve) and close the right-hand valve.
3	Start the pump from the software, and run the pump until solvent is seen flowing out of the exit tube.
4	Bleed the pipes for a few seconds then stop the pump and close MV2 (left hand side)

10.2. Co-solvent Modifier



Prime valve (MV2) - optional

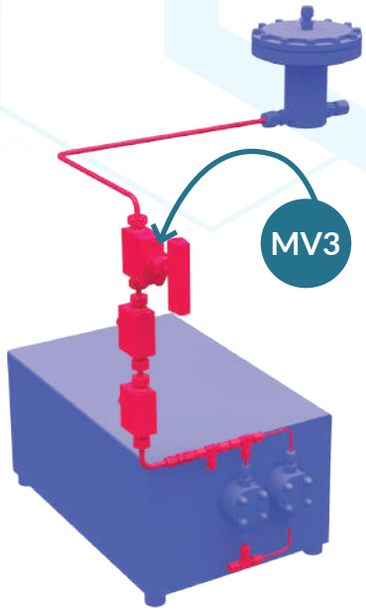


Prime valve (MV2) - optional

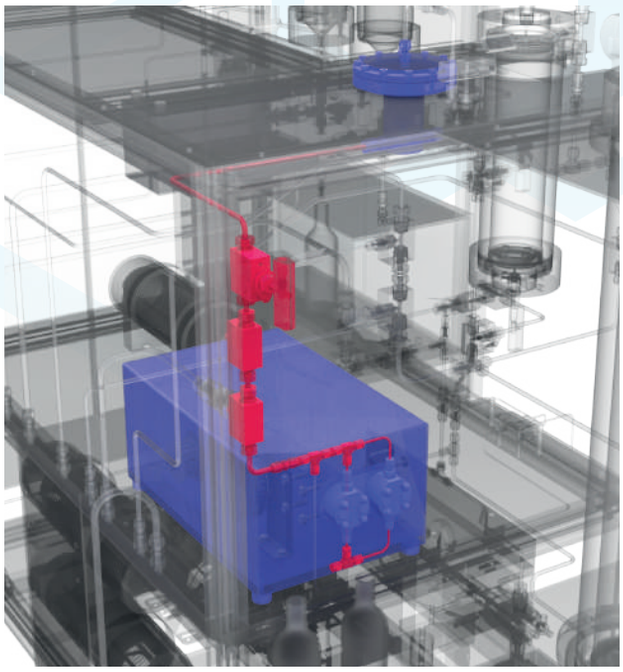
The co-solvent pump can be used to adjust the polarity of the CO_2 being used to extract the biomass. This is accomplished with the addition of a small percentage of a polar solvent, such as ethanol. To make this addition, ensure the pump is primed (See 10.1) with the appropriate solvent before continuing.

1	The addition starts once the system is up to pressure. Ensure valves MV2 and MV3 are closed before you start.
2	Open MV2 (right hand valve), and the pressure on the co-solvent pump (see software) will rise.
3	Start the pump from the software.
4	At the end of the run, stop the pump and close MV2 (right hand side).

10.3. Make-up Pump



Make-up Valve (MV3) - (optional)



Make-up Valve (MV3) - (optional)

During the extraction, material can build up in the vaporiser or on the needle of the ABPR. Solvent can be introduced just before the ABPR, so not to interfere with the extraction (as a modifier), and clean the needle and vaporiser in situ.

1	The addition can be started, before, during or after an extraction run, to clean the ABPR and vaporiser *in situ*.
2	Ensure MV2 is closed before starting the addition.
3	Open MV3 pressure and the co-solvent pump (see software) will rise.
4	Start the pump from the software.
5	At the end of the run, stop the pump and close MV3.

Co-solvent Selection Valve (MV2)

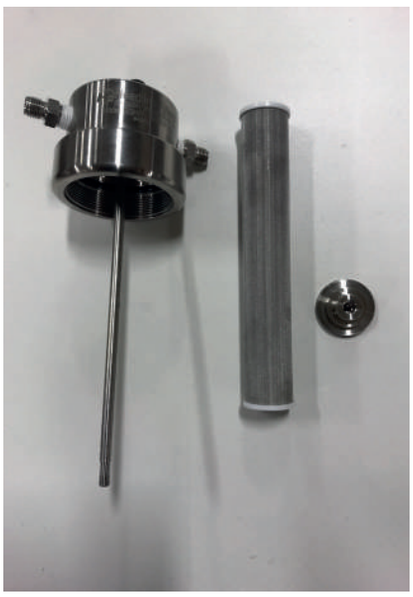
11. Filter

11.1. Cleaning CO_2 Inlet Filter

The CO_2 inlet filter traps any particulates that might be present in the CO_2 bottles or lines. This ensure these particles do not reach the CO_2 pump potenitally damaging the check valves. It also acts to capture any material that may carry over as a result of CO_2 recycling. The following procedure describes how to dismantle and clean the inlet filter:

Headline CO_2 inlet filter

Headline CO_2 inlet filter – Body removed

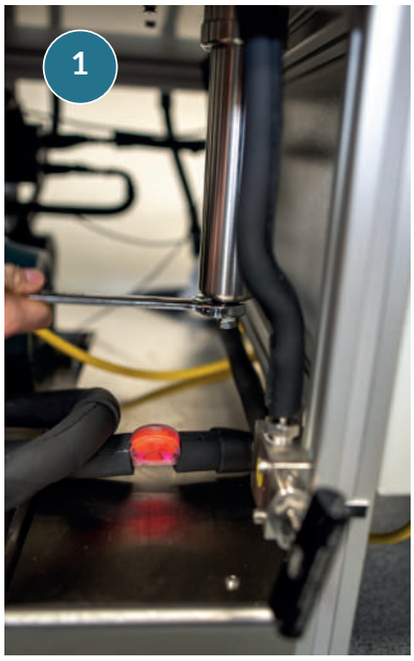


Headline CO_2 inlet filter – Filter mesh removed

1	The CO_2 inlet filter is located on the right hand side of the system, on the lower shelf next to the pump.
2	It can be removed using a 9/16” spanner on the hex nut, located at the bottom of the filter body.
3	Turn the body counter-clockwise to loosen.
4	Unscrew the main body to reveal the filter mesh.
5	To remove the mesh for cleaning, the body needs to be removed to expose a round locknut which clamps the filter to the upper head. This is located at the bottom of the filter mesh.
6	Turn counter-clockwise until the locknut is removed from the thread, which will release the filter.

Manufacturer instructions for servicing the filter can be found from the link below:

www.headlinefilters.com/library/documents/5919a46d7c403-instructionsforheadlinefilterhousings.pdf



Step 1 – Loosen nut with 9/16" spanner

Step 2 – Unscrew body counter-clockwise

Step 3 – Lower body to reveal filter mesh

12. Spare Parts

Vessel 1 – EV5L (5L Extraction Vessel)

Part Number	Description	Quantity
S4375VID	SEAL, CS VESSEL GFP - 5L	2
OT5LCA	O-ring, Teflon 5L Cap Assy	2
FT325-5U-Assy	Frit Assy, 3.25” 5 micron	2
FR-425VID	Frit Retainer, 4.25 VID	2

Basket 1 – EVB5L (5L basket)

Part Number	Description	Quantity
OT1L500MLBA	O-ring, PTFE 1 Litre/500 mL basket	2
FT2709-40u	Frit 2.709” OD 40um	1

Vessel 2 –EV1L (1L Extraction Vessel)

Part Number	Description	Quantity
S300VID	Seal, 3.00” VID CS vessel GFP 500mL-1L	2
OT1L500MLFR	O-ring, Teflon 1L/500mL Cap Assy	2
FT225-5U-Assy	Frit Assy, 2.25 x 0.076” 5 micron	2
FS300	Frit Retainer, 3 VID x 0.075 wide	2

Basket 2 – EVB1L (1L basket)

Part Number	Description	Quantity
OT1L500MLBA	O-ring, PTFE 1 Litre/500 mL basket	2
FT2709-40U	Frit, 2.709” Dia x 0.078” x 40 - 0.5/1L basket	1

Cyclone – CV1L (1L Collection Vessel)

Part Number	Description	Quantity
OringP232	O-ring, CS1L collector Cap PTFE	2
Oring233	O-ring, CS1L Top Cap PTFE	2

Cold Trap – CVCT1L (1L Coldtrap)

Part Number	Description	Quantity
OT1LCT	O-ring, 1L Cold Trap PTFE Face Seal	1

P500 – CO_2 Pump (SFXPC500-689)

Part Number	Description	Quantity
SP050	Seal, Piston 500g/min	2
SBKP050	Seal, Backup Piston 500g/min	2
VCHKP050	Check Valve, P500 Pump	4
SCHKP050	Check Valve Seal P500 pump	8

P50 – CO_2 / Co-solvent Pump (SFXPC50)

Part Number	Description	Quantity
SP316-1	Seal, Piston 50g/min	2
SBKP316-1	Seal, Backup Piston 50g/min	2
VCHKP05	Check Valve, P50 Pump	4
SCHKP05	Check Valve Seal P50 pump	8

ABPR – Automatic Back Pressure Regulator

Part Number	Description	Quantity
SEN-ABPR10K	Sensor Assembly + Seals	1

13. Troubleshooting

Problem	Cause	Solution
Pressure not building within system	Incoming CO_2 not liquid	**Check density if flowmeter installed. If density below 0.85g/cm3 then:** Check chiller working and cooling to below 4 degC. Check CO_2 bottle pressure. This can be checked from the Condenser PT. Ensure this is above 50 bar.
	Check valves in CO_2 pump	Remove and clean valves. Ensure the direction of the check valve is correct before re-installing.
	Pneumatic pressure	Check pneumatic pressure on ABPR is above 8 bar or 100 psi.
	ABPR seat is worn	Service ABPR

Problem	Cause	Solution
Leak on pipework	Swagelok - Loose connection on the compression fitting	Ensure tight. If still doesn't seal inspect 2-part ferrule system once de-pressurised. If no gap between the front and back ferrule joint may need replacing.
	HIP - Loose connection on the compression fitting	Ensure tight. If it still won't seal the joint may need replacing.

Problem	Cause	Solution
Leak on vessel	Piston seal worn	Replace piston seal
Leak on pump	Vessel seal worn	Replace vessel seal

Problem	Cause	Solution
Drop in yield when using basket	Inconsistency in raw material	Check material
	Basket O-rings worn causing CO_2 to bypass the basket	Change O-rings

Problem	Cause	Solution
Co-solvent pump not delivering correct volume	Pump calibrated against water (density of 1g/cm3)	Ensure the density of the solvent being pumped has been accounted for. This can be adjusted *via* the software.
	Co-solvent pump has not been primed	Prime the line using MV2.

Problem	Cause	Solution
CO_2 flow rate does not match setpoint (When flow meter installed)	Condenser pressure is below 50 bar	Change CO_2 bottle
	Condenser temperature above 5 °C	Check chiller is functioning correctly. Dropping the temperature further could help the flow rate.
	Flow meter density below 0.85 g/cm3	Check incoming CO_2 (temperature + pressure)

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Core Separations