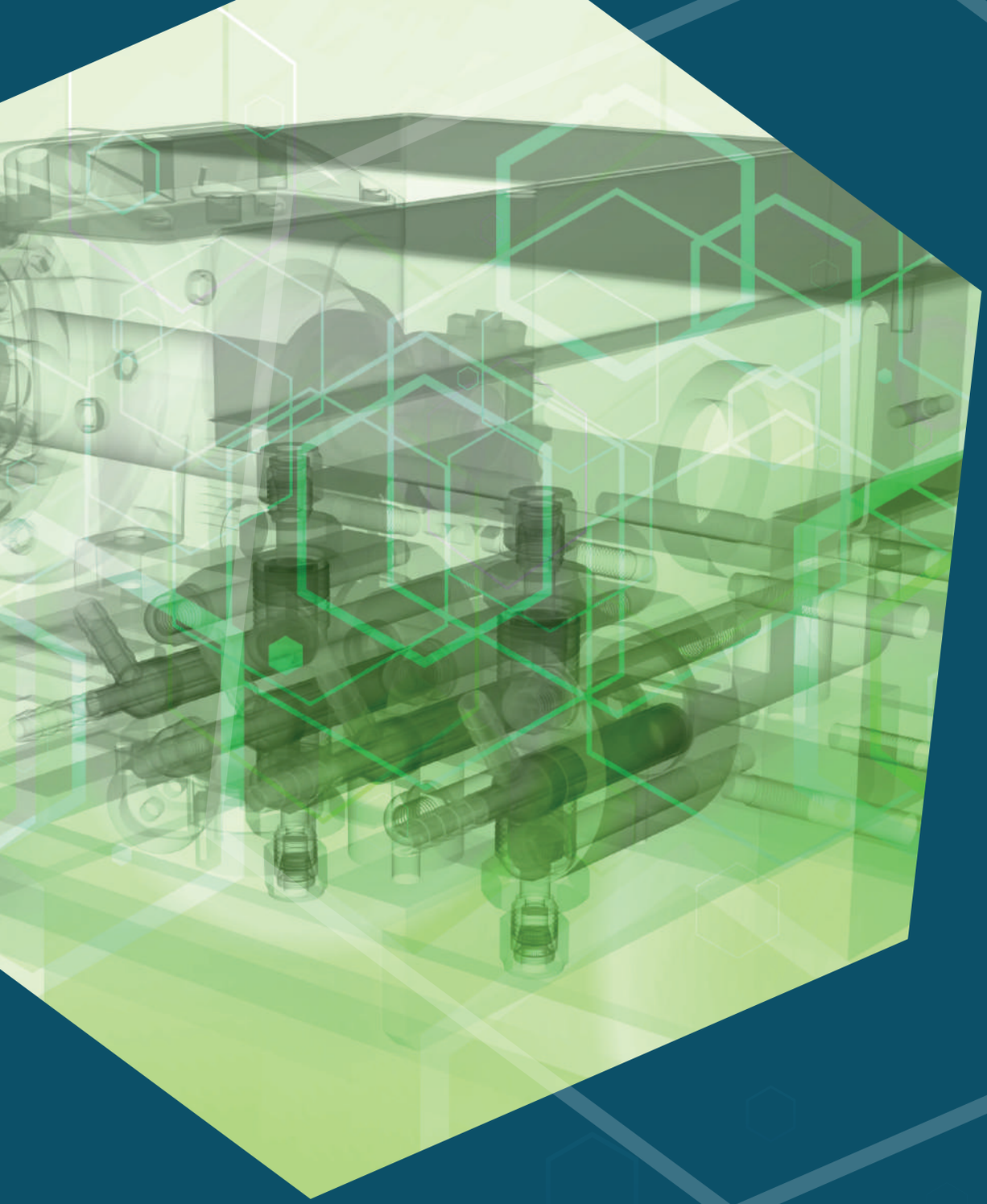

Core | **Pumps**

Operating Guidelines for CI and CU Series Pumps v1.1

Author: Dr J Tunstall

SciMed™
Core Separations

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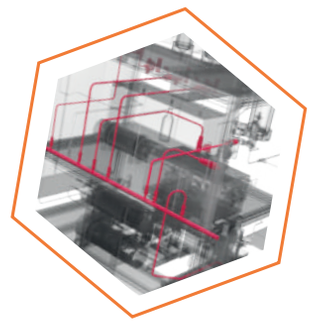

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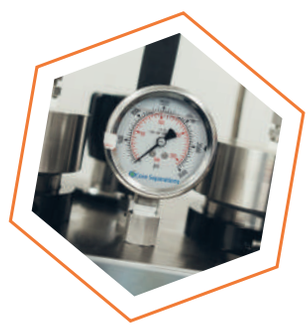


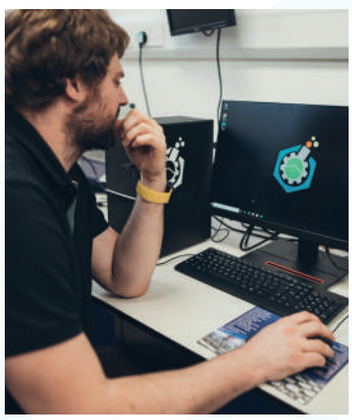
01. Supercritical Fluids

At Core Separations we specialise in Supercritical Fluid Extraction (SFE), which is commonly used to extract compounds from solid botanical material. Supercritical CO_2 is prodominately used for these extractions, due to its achievable presure and low temperature (critical temperature and pressure of 31 °C and 74 bar.). The CO_2 can be further modified using a co-solvent (usually methanol or ethanol) to help change the polarity of the mobile phase.

Supercritical Fluid Extraction can separate and fractionate 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 the ones provided with a Core Separations SFE system, you can manipulate the pressure and temperature of the supercritical CO_2, in order to selectively extract and collect the desired material.

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, and nut-oil extracts).
- Extraction processes which isolate food and botanical compounds, that require a clean and safe extraction process which does not leave any residual materials in the collected product.



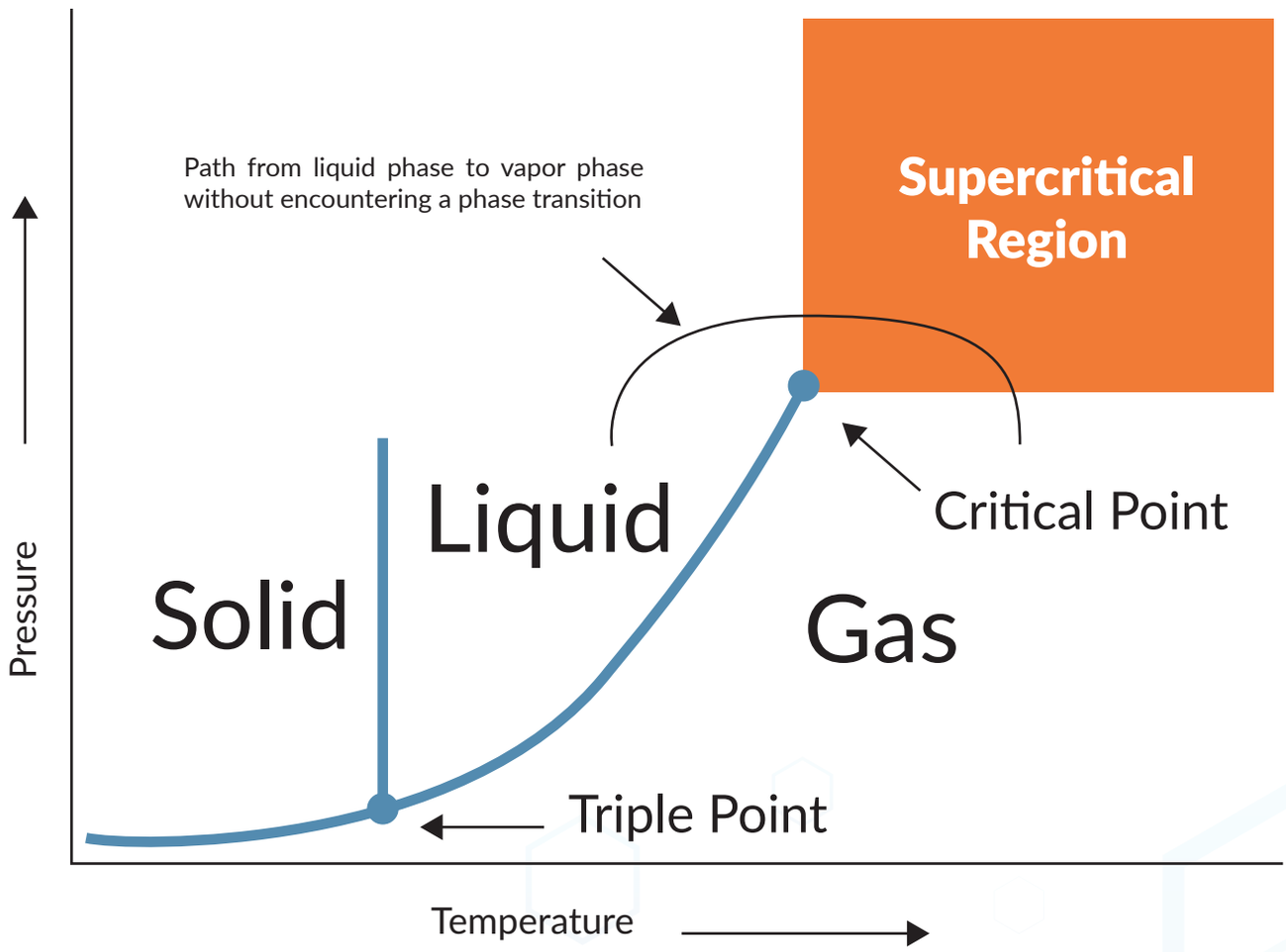


Figure 1–1: Supercritical CO_2 is tunable without changing phases

During extraction and collection, by raising the pressure of the supercritical CO_2 flow in the system, you will 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. Introduction

The Core Separations high pressure liquid CO_2 pumps, are a dual piston positive displacement pump. It is designed for use in any application, where a liquid is to be compressed within a small volume to develop pressure. All wetted parts are manufactured using 316 stainless steel and/or 17-4PH stainless steel, with PTFE graphite seals as standard.

	Pressure Range 0-10,000 psi	Pressure Range 0-14,500 psi

The High-Pressure pump 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.

2.1. EC Mark and Certification - Declaration of Conformity

The Core Separations high pressure piston pumps described in this manual, are manufactured in compliance with Directive 2006/42/CE and European Community Directives that are pertinent and applicable at the time they are sold.

These instructions were drafted in accordance with Annex VI of the above Directive and complies with Annex I section 1.7.4, as well as the UNI 10893 standard, and ISO/IEC 37 guidelines.

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

2.2. 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 freight paid for inspection, repair, or replacement by the manufacturer.

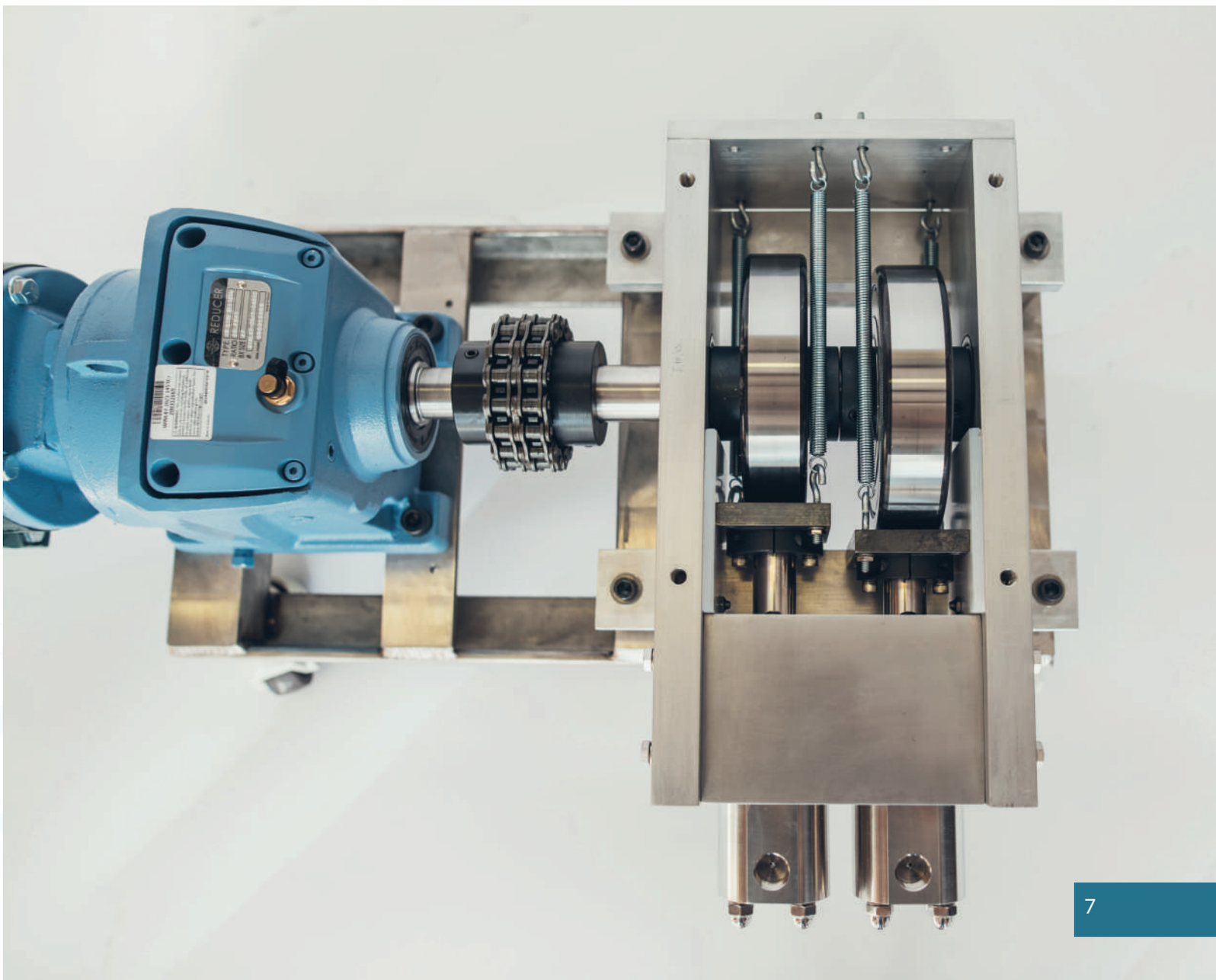
4. This limited warranty is the only form of valid guarantee and replaces any other form of explicit or implicit warranty, including any guarantee of fitness for sale or any particular purpose.

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 by 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.

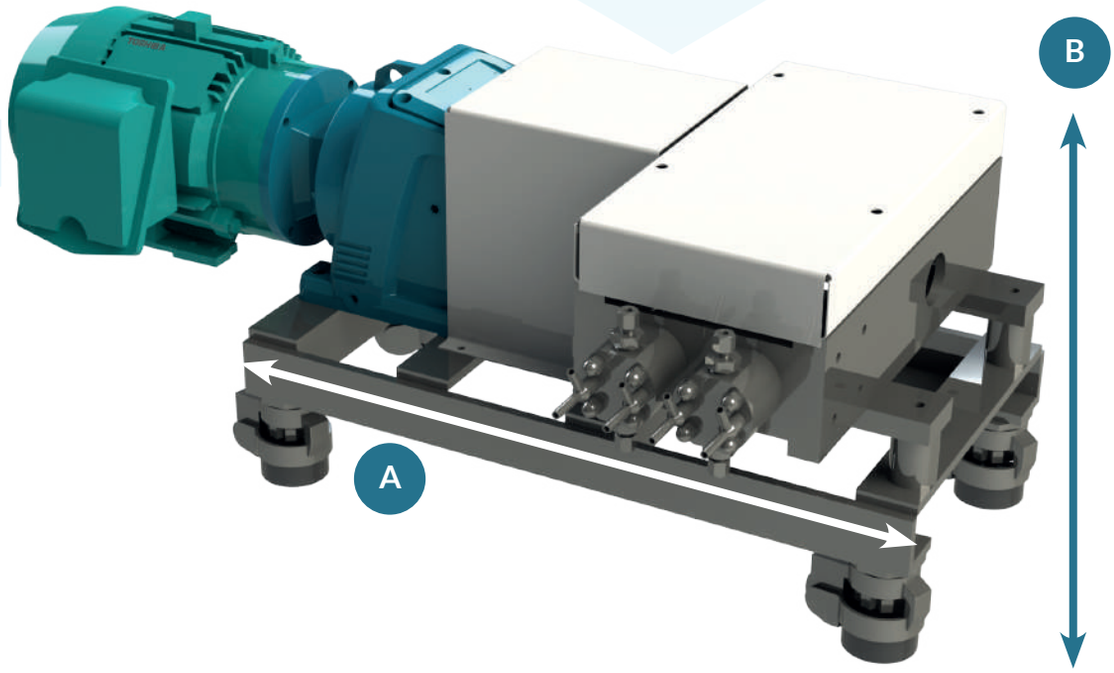
There are no additional guarantees other than the guarantee described above.

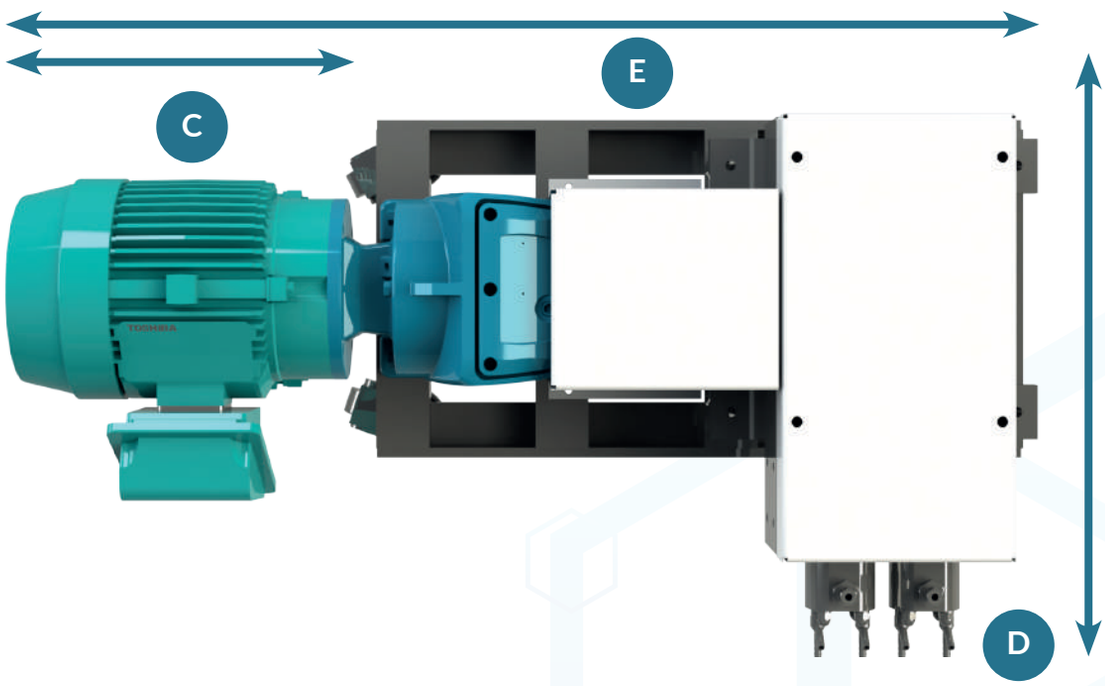


2.3. Technical characteristics

The main dimensions and specifications for the CI + CU SERIES Pumps are as follows:

A (inch)	B (inch)	C (inch)	D (inch)	E (inch)	Weight
24.4	14.3	14.8	18.8	39.2	150lb / 107 kg





2.4. 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 pump described in this manual was not designed for operation in potentially explosive environments.

2.5. Power Requirements

Power supply (3 phase). 415V 3~N 50Hz

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

Component	Amperage (FL) 240/415 V	KW
P500 Pump	6 / 3	1.5
P1K	8 / 4	2.2
P251K	6 / 3	1.5

2.6. Vibration

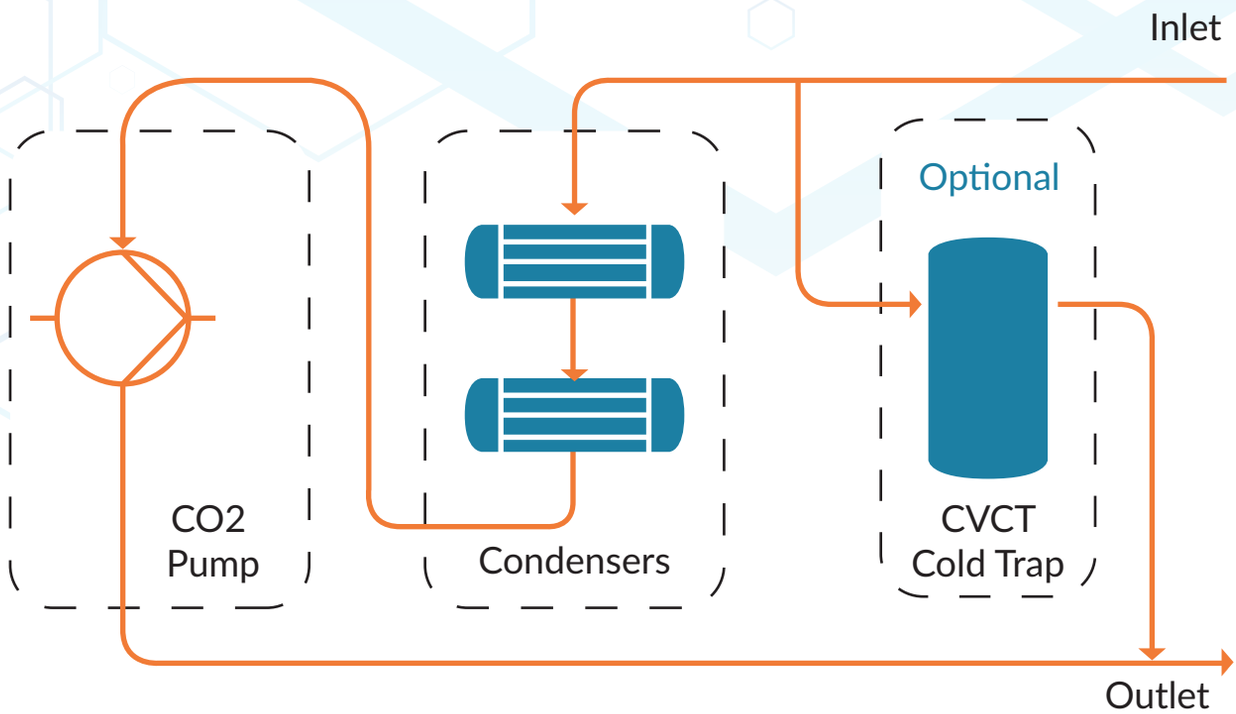
Core Separations pumps do not produce hazardous vibrations under normal use, provided the installation and assembly instructions in this document were carried out properly.

2.7. 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.

2.8. Pump Cooling Circuit

A common loop found within all super and subcritical CO_2 systems is the cooling circuit. The cooling loop recirculates a water glycol mixture (50/50) around the condenser (heat exchanger) and through the pump heads. This is to ensure that 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 Cooling Cartridges

2.8.1. Chiller

Any pump where the primary operation is to pump liquid CO_2 must be cooled. This ensures the incoming CO_2 remains a liquid and the heat generated from compression is removed. The chiller required depends on the amount of heat generated, which can depend up a number of factors such as flow rate, pressure and the temperautre of the incoming liquid CO_2.

When a chiller is to be provided by by the end user we recommend it matches or exceeds the following specification:

Pump	Power (KW @ 15 C)	Flow Rate	Pump Pressure
P500	1.5	22L/min	2.5 bar
P1K	2.2	22L/Min	2.5 bar
P251K	2.2	22L/Min	2.5 bar

03. Safety

3.1. User Responsibilities

The ultimate responsibility for the safe operation of this pump is with the user. The pressure limits 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 pump 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 accessory should be directed into a safe area. Consideration should be taken when defining the safe area. Please consult a Core Seperations engineer if required.
3. The pump must be immobilised using the self-levelling feet during use.
4. The pump should be installed in a well-ventilated lab.

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.

3.2. Unpacking the Pump

The CI and CU SERIES pumps are supplied on a trolley, allowing the pump to be correctly positioned for the required process. The pipe work for the pump is removed before packing and transport.

Installation of the Pump after transport

3.3. Installation

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

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

1. That the pump has the correct power supply.
2. The electrical parts are correctly and adequately protected.
3. 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 pump must be stopped immediately, and the pressure brought down to zero.

The pump should be located close to:

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

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

1. Installation of pipework directed to a safe discharge area.
2. Appropriate ear protection in case of rupture disk activation. Especially for operators standing near the pump.

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

3.4. Fittings

The fittings used to connect to the ports on the inlet and outlet check valves depend up the model and maxmum pressure. The pump check valve holders have been machined using dimensions provided by the manufacturer. Interchanging fittings with other manufacturers is not recommend. We recommend the use of swagelok (or equivalent) for creating the Inconnecting pipework

Pump	Check Valves	Interconnecting
P500	HIP - AF4	Swagelok (SS-400-3)
P1K	HIP - AF4	Swagelok (SS-400-3)
P251K	1/4 M/P	Core Manifold (1/4 M/P)

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/

Autoclave Engineer (Parker)
http://www.autoclave.com/aefc_pdfs/FT_MedPress.pdf

3.5. 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 pump. Frequent checks should be carried out ensuring the equipment is free from:

1. Cracks
2. Corrosion
3. Bulging in the outer wall (pump heads)

If any of these are observed, stop using the system immediately and contact a Core Separations 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.

3.6. Personal Protective equipment (PPE)

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

Wear Safety Googles

Wear Gloves

3.7. Product Labels

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

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

Under no circumstances should labels be removed from the equipment.

04. Pipework

4.1. Making Pipework

Before installing ensure the high pressure pipe has been correctly made!

Core Separations use a variety of widely available high-pressure fittings (HIP, Autoclave Engineer and Swagelok). It is the user's responsibility to be familiar with the installation and removal of these fittings before attempting to install the pump. 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 differences between the different 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
AF4 (1/4")	Inlet and Outlet Check Valves	Initial Compression – 30 ft/lb Tighten Connection – 50ft/lb
SF250CX (1/4")	Inlet and Outlet	Initial Compression – 10 ft/lb

Do not form create new pipework using the check valve holders to compress the tapered ferrule. Instead use an appropriate HIP fitting.

We advise the use of Swagelok high pressure fittings (and equivalents) to make the interconnecting pipes. The 2-part ferrule fittings DO NOT require a torque wrench. Manufacturer instructions should be followed to prevent in-correct installation of these fittings. If in doubt, please contact a Core Separations engineer for advice.

CI Series Pump - P500 and P1K

CU Series Pump - P251K

05. Servicing

5.1. Servicing the piston seals

When starting any service, ensure the pump is fully depressurised before removing the pipework (See 04. Pipework). Seals can be changed as a preventative measure or when a seal has become damaged/worn. A common effect of a damaged seal is the release of CO_2 between the head and the pump casing. If this is the case, a change of seal can be carried out following the instructions below:

1	First remove the copper cartridges from the pump heads. ***Note: There is no reason to remove the water pipes from the copper cartridges.***
2	The pump head is held on the body by 4 domed nuts. Using a 9/16" spanner or socket to remove the domed nuts.
3	Once the nuts have been removed, slide the pump head towards you. Take care not to damage the pistons when removing the heads. ***Note: You may feel resistance from the seals.***

Step 1 – Remove Copper Cartridges

Step 2 – Remove domed nuts

Step 3 – Slide pump head towards you

Take care not to damage the pistons when removing the heads.

Do not scratch the seal, this will cause seals to leak prematurely.

4	When removing the pump head, you may find the seals remain on the ceramic piston. Remove these by carefully sliding them towards you. If the seals remain in the pump head, you may need to use the O-ring pick to remove them. Be careful not to scratch the head. Clean the head with IPA (iso-propanol) to remove any debris before installing a new PTFE sprung seal. Ensure the spring is facing towards the process. ***Note: Once installed the spring should not be visible to the user.***
5	Once the sprung seal has been installed, the PEEK seal can be placed into the recess.
6	Now the seals have been replaced the head can re-installed. The threaded rods act as a guide to ensure the head remains straight. The domed nuts are then re-installed and the process is repeated for both heads. ***Note: Torque setting required for the tightening of the nuts is 20nM or 15ft/lb***

Step 4 – Remove old seals

Step 5 – Replace with new seals

Step 6 – Return pump head

The pump should be removed from service before any maintainence is attempted. Ensure the pump has been completely depressurised.

5.2 Check Valve Servicing - P500/P1K

The check valves on a pump ensure the flow and pressure are maintained in the correct direction. A common fault is the failure of a check valve, which results in the inability to reach the desired pressure or flow rate. If this occurs the check valves can be serviced following the instructions below:

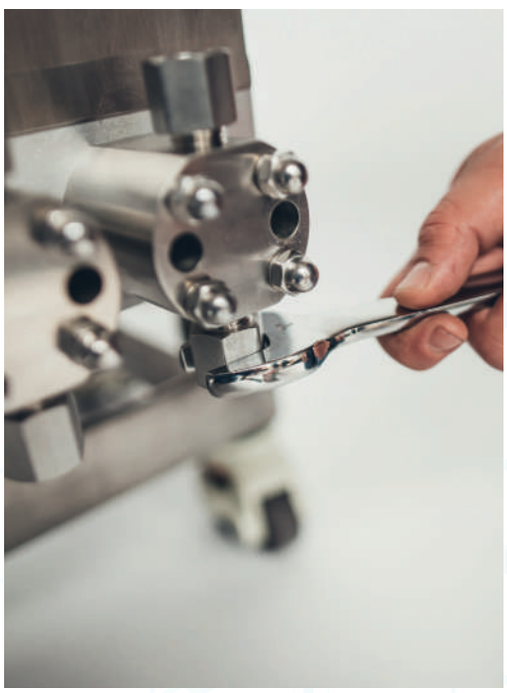
1	The check valve holders are removed by turning the holder anticlockwise using a 1" spanner.
2	Once the holders have been removed the old check valves can either been replaced or cleaned. The arrow on the check valves should point in the following directions Outlet – towards the process Inlet – towards the pump head ***Note: The check valves can be cleaned by sonication in alcohol to remove oils and debris.***
3	Apply Teflon spray or lubricant to the check valve holder threads. Tighten the check valve holder into the head by turning clockwise. ***Note: Screw the holder down hand tight, then tighten with the 1" by 1/4 of a turn.***

Step 1 - Remove check valve holder

Remove check valve from head

Ensure check valve is in correct orienartion

Replace check valve



Tighten check valve holder

Take care that the check valve holders are clean before installing the check valves.

Scratches to the seals, could cause them to leak prematurely.

5.3 Check Valve Servicing - P251K

Unlike the P500 and P1K pumps that use a cartridge type check valve, the P251K incorporates the check valve into the holder. This allows the pump to maintain the flow at pressures exceeding 1000 bar. It also means the check valve can be full serviced, from replacing the check valve ball to the seals used to seal the check valve onto the pump head.

1	The check valve holder is removed by turning the holder anticlockwise using a 1" spanner
2	Once the holders have been removed the old check valves can either been replaced, cleaned or serviced (see 5.2.1 and 5.2.2). The arrow on the check valves should point in the following directions Outlet – towards the process Inlet – towards the pump head ***Note: The check valves can be cleaned by sonication in alcohol to remove oils and debris.***
3	Apply Teflon spray or lubricant to the check valve holder threads. Tighten check valve holder into the head turning clockwise. ***Note: Screw the holder down hand tight, then tighten with the 1" spanner by 1/4 of a turn.***

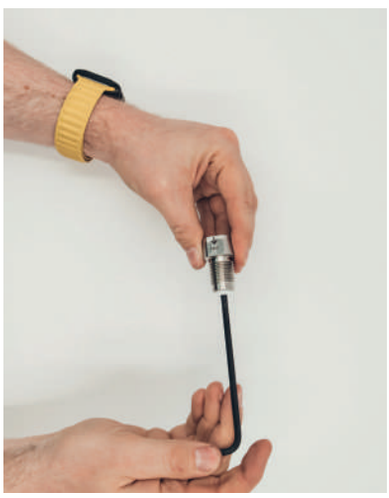
Step 1 - Remove check valve holder

Inlet

5.3.1. Inlet Check Valve

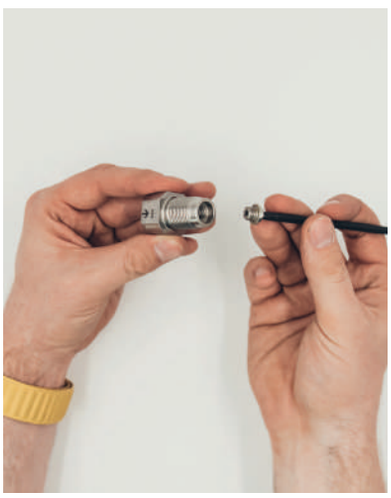
The P251K uses a self contained check valve holder for the inlet and outlet. Unlike the CI series pumps that use a standard cartridge style check valve design, these holders can be fully serivced, from the check valve ball to the spring. Below describes how to dismantle and serivce an inlet check valve holder:

1	Once the check valve holder has been removed from the pump, the seal can be removed by un-screwing the check valve cap using a 3/16” allen key.
2	Once the seal has been removed the check valve **CAP** can be fully removed to reveal the spring.
3	Remove the spring from the check valve
4	Turn the check valve upside down ensuring to capture the check valve ball. The check valve can now be serviced by replacing the check valve ball, spring etc.



Step 1a - Unscrew check valve cap

Step 1b - Remove seal



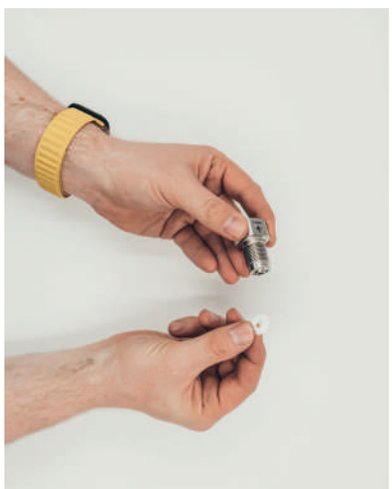
Step 2 - Remove check valve CAP

Step 4 - Remove check valve ball

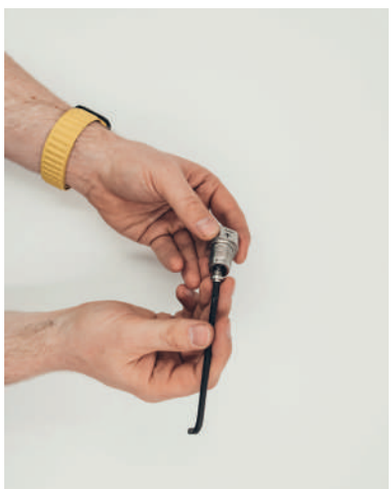
5.3.2. Outlet Check Valve

The outlet check valve holders for the P251K are slightly different to the inlet holders. The hex cap contains the checkball SEAT, with the check valve ball being in the upper portion of the valve. Care should be taken to ensure the ball is not lost when the hex SEAT is removed. Below describes how to dismantle and service an outlet check valve holder:

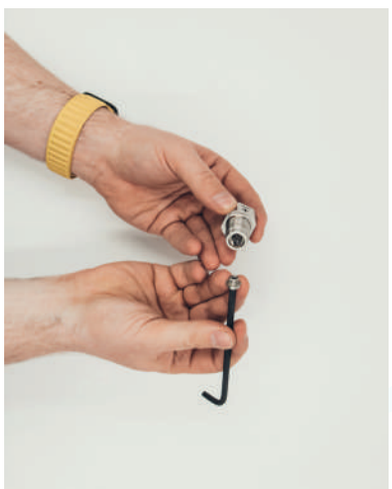
1	Once the outlet check valve holder has been removed from the pump, the seal can be removed by unscrewing the check valve cap using a 3/16" allen key.
2	Once the seal has been removed the check valve **SEAT** can be fully removed to reveal the check valve ball.
3	Remove the check valve ball from the check valve
4	The check valve springs can now be accessed and removed. The check valve can now be serviced by replacing the check valve ball, spring etc.



Step 1 - Remove the seal



Step 2a - Remove check valve SEAT



Step 2b - Reveals check valve ball

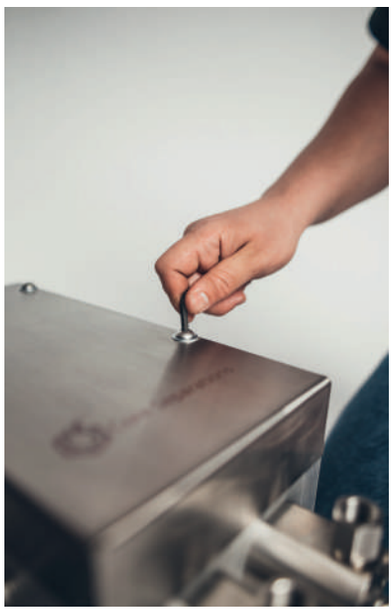
Step 4 - Remove check valve ball

5.4. Bearing Maintainence

The pistons are driven by two bearings located inside the casing. The lubrication for the bearings are “sealed for life” so the inside of the bearing does not require further lubrication. However the outer wheel that strikes the piston plate does require lubrication. Lubrication of the bearings should be carried out every month to ensure problem free operation of the pump.

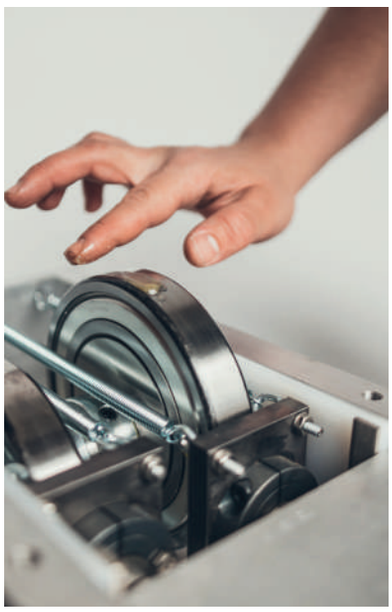
1	Before starting, ensure the pump is fully depressurised and the power has been isolated. Remove the top case from the pump by unscrewing the allen screws with a 7/32” allen key.
2	Lift the casing from the pump to reveal the two bearings.
3	Apply grease to both of the bearings either using a brush or your finger.
4	Move the outer ring of the bearing so to ensure the entire face is be sufficiently greased.
5	Replace the casing on the pump and tighten down the 4 allen screws using 7/32” allen key.

Bearing Maintainence



Step 1 - Un-screw allen screws on the casing

Step 2 - Remove casing to reveal bearings



Step 3 - Apply grease to the surface of the bearings

Step 4 - Move the outer ring of the bearing to apply grease to the entire surface

Step 5 - Replace the outer casing

06. Spare Parts

P500-2 – Pump (500g/min @ 689 bar)

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

P1K– Pump (1kg/min @ 400 bar)

Part Number	Description	Quantity
SP050	Seal, Piston 1kg/min	2
SBKP050	Seal, Backup Piston 1kg/min	2
VCHKP0505	Check Valve, P1K Pump	4
SCHKP050	Check Valve Seal P1K pump	8

P251K– Pump (250g/min @ 1000 bar)

Part Number	Description	Quantity
SP375	Seal, Piston 250g/min	2
SBK375	Seal, Backup Piston 250g/min	2
VCHKInletP251K	Inlet Check Valve, P251K Pump	2
VCHKOutletP251K	Outlet Check Valve, P251K Pump	2
CVSP251K	Check Valve Seal P251K pump	8

VCHKInletP251K– Inlet Check Valve

Part Number	Description	Quantity
CVBP251K	CV Ball	1
SVSIP251K	CV inlet Spring	1
CVInsInP251K	CV Inlet Insert	1
CVSP251KBodyInlet	CV Body Inlet	1

VCHKOutletP251K– Outlet Check Valve

Part Number	Description	Quantity
CVBP251K	CV Ball	1
SVSOP251K	CV Outlet Spring	1
CVInsOutP251K	CV Outlet Insert (seat)	1
CVSP251KBodyOutlet	CV Body Outlet	1

07. Schedule

The pump's efficiency can be safeguarded by following the preventive maintenance schedule below:

Control	Daily	Weekly	Monthly	6 Monthly	Year
PTFE seals (Replaced)				x	
PEEK seals (Replaced)					X
Check Valves (Cleaned)			x		
Replace oil in Gear Box*					X
Grease bearing			X		

***The oil in the gear boxed should be replaced with Mobil SHC 634 Synthetic Lubricant**

The operating cycle, the temperature, the quality of the pumped fluid, the type and quality of the supply, and the condition of the accessories used, are all fundamental factors that influence the life of pump parts.

If the pump's performance deteriorates, check whether our "Troubleshooting" guide describes the source of the problem. If the pump is running without any problems, check it after 1000-hours of operation and then every 500-hours of operation.

08. Trouble-shooting

Problem	Cause	Solution
Pump does not start	Lack of power to the pump	**Ensure there is power to the controller**
	Error on VFD	Check for error messages on VFD, if error persists, contact Core Separations

Problem	Cause	Solution
The Pump runs but does not build pressure	The pump is not connected to the CO_2 supply, or control valves at the supply are closed	Check there is CO_2 in the cylinder and at pressures between 50 and 55 bar
	Check valve failure	Check the check valves are opening and closing correctly
	Worn piston seals	Check the status of the seal. Replace the seals
	Low speed of rotation	Check the motor and the drive
	High incoming temperature of CO_2	Check the incoming temperature of the CO_2 Ideal conditions are between - 5 °C and + 4 °C

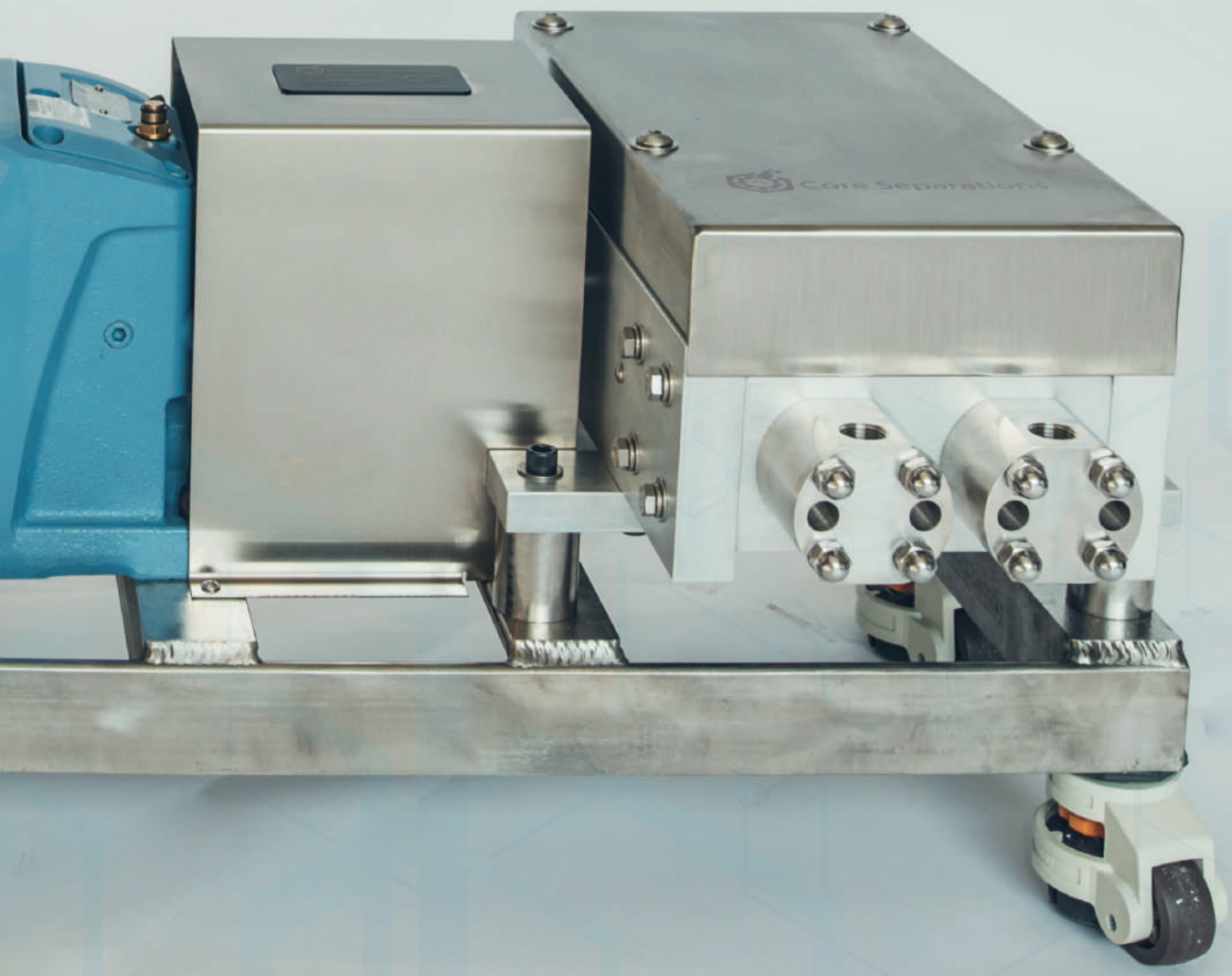
Problem	Cause	Solution
The pump comes up to pressure but pulsates	Dirty check valves	Clean the check valves
	Worn Piston seals	Replace seals

Problem	Cause	Solution
The pump is very noisy	Worn or dry bearing	Grease or replace bearing
	Pump-motor coupling problems	Check status of the keys and coupling

Problem	Cause	Solution
Short piston seal life	Damaged Ceramic Piston	Replace Piston
Leaking from, the Weep hole in the head	Worn Piston Seals	Replace seals

Problem	Cause	Solution
Short bearing life	Pump-Motor coupling problems	Check the status of the keys and coupling
	Excessive Pressure	Change seals

E-Stop ON
Power ON
Power ON / OFF
DANGER



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