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PREMP SYSTEMS OPERATIONS AND MAINTENANCE MANUAL

	Name	Role/Title
Owner	Malcolm Guthrie	Author/ High-Pressure Scientist
Reviewer	Cecilia Lowe Helena Ramsing Nathalie de Ruette	Reviewer/Quality Control Officer Reviewer/NSS Health & Safety Engineer Detector Lab Coordination Officer
Approver	Arno Hiess	Approver/ Director Scientific Activities Division

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1. INTRODUCTION

1.1. Scope

This document covers operational aspects of various high-pressure (HP) sample environment equipment (SEE) and, separately, pressure-generating or pressure-controlling devices (drivers) and including issues of safety and certification (Quality). In normal operation, the driver and device will be connected (typically by a hydraulic line or other piping) forming a complete sample environment system (SES). Such connection and disconnection of equipment and driver will be a frequent and normal activity and this is considered in safe operating procedures described here.

At the present point in time, the number of devices is small. As we approach user operations, device number will increase and this document will grow accordingly. In order to perform both comprehensive risk assessment and to comply with legal requirements individual devices (drivers and SEE) need their own specific consideration. In addition, when these devices are combined to form a *system* an additional risk assessment or consideration of quality issues will, in general, be required.

Accordingly, in this document, each device is described individually and separate sections are also provided for complete systems composed of these devices.

1.2. Summary of individual devices

Here is a table summarizing the current list of devices (correct as of the current version of this document). Where relevant, devices have been categorized as main or sub systems, where it makes physical sense to do so (i.e. the devices are semi-permanently attached to each other)

Table 1 High pressure driver systems

Device Name	Device Class	System type	PREMP ID	Associated SEE
Portable Pressure Driver (PPD)	Driver	Main	PRD-001	PRE-PE-001,PRE-DAC-001
Maximator hydraulic System (MHS)	Driver	Sub system of PPD	PRD-001-001	PRE-PE-001
PACE 5000 gas pressure controller (PACE)	Driver	Sub system of PPD	PRD-001-002	PRE-DAC-001,PRE-DAC-002,PRE-DAC-003

Table 2 Sample Environment Equipment

Device Name	Device Type	Device Class	System type	PREMP IDs	Number of devices
V3 Paris-Edinburgh press (V3)	PE	SEE	Main	PRE-V3-001	1 (on loan, property LLB)
VX6 Paris-Edinburgh press (VX6)	PE	SEE	Main	PRE-VX6-001	1 (on loan, property Université Sorbonne, Paris)
Membrane -driven ESS DAC	DAC	SEE	Main	PRE-DAC1-001	1
Membrane -driven UoE DAC	DAC	SEE	Main	PRE-DAC2-001,PRE-DAC2-002,PRE-DAC2-003…etc	5

1.3. Applicable legal documentation

Due to the special hazards associated with pressure equipment, several pieces of European and Swedish legislation apply to the equipment and activities described in this document. The specific legislation that has been considered in drafting this document includes:

- The European Pressure Directive (PED) 2014/68/EU (formerly 97/23/EC).
- The Swedish directive “Användning och kontroll av trycksatta anordningar” AFS 2017:3 on “Use and Inspection of Pressurised Devices” and
- The Swedish directive “Provning med över- eller undertryck" AFS 2006:8 on “Testing with overpressure or underpressure”.

1.4. Structure of document

In the following, specific procedures relating to operation of high-pressure sample environment at the ESS are given. Following this, each device or system is described in a separate sub-section. Each section includes, where appropriate:

- Summary of purpose of equipment
- Description of the components
- Piping and Instrumentation diagram (P&ID)
- Declaration on the legal status (classification) of the device (where appropriate)
- A risk assessment for both maintenance and operation of equipment
- A manual describing both maintenance and operation of equipment (also copied in appendices)

- Where appropriate a maintenance schedule for the device (also copied in appendices)
- Where appropriate, a record-keeping scheme for device operations
- details of any required training for operators

1.5. Note on risk-assessment methodology

The risk assessments have been documented in excel format files that are based on Section 2 of the ESS RAMS document available on:
https://confluence.esss.lu.se/pages/viewpage.action?pageId=265308713

The meaning of the likelihood conditions is not stated in that document, therefore, in this document, the following assignments have been consistently used:

Likelihood (L)	**Average frequency of event**
1	Less than once in 10 years
2	Once in 10 years
3	Once a year
4	Monthly
5	Daily or certain to happen

Estimation of frequency has been determined by consultation with existing facilities, using similar procedures/equipment for periods of several decades

The meaning of “severity” is as defined in ESS RAMS:

Severity (S)	**Consequence of event**
1	Minor injury no lost time or delays/disruption
2	First aid injury, less than 3 days absence, minor disruption
3	Minor injury, more than 3 days absence or minor delays
4	Major injury, long term absence or major delays
5	Fatality or total loss

And the risk matrix employed is also taken from the ESS RAMS:

	Severity (S)				
Likelihood (L)	1	2	3	4	5
	2	4	6	8	10
	3	6	9	12	15
	4	8	12	16	20
	5	10	15	20	25

green = low risk (L); orange = medium risk (M); red = high risk (H)

1.6. Note on units

For convenience, both Bar and Pascal (Pa) units are used in this document, the former most commonly for low pressures and the latter most commonly for very high pressure (e.g. gigapascal, GPa, range). Where relevant, lower pressures, quoted in bar, are described as either Barg (gauge pressure) or Bara (absolute pressure) depending on what is appropriate.

By definition, 1 bar = 10^5Pa = 0.1 MPa

Similarly, loads are described in either metric tonnes or kN. While the correct conversion between these units (on the surface of the Earth) is 9.806 kN = 1 tonne, we have here generally rounded up taking 10kN as an approximation for 1 tonne.

2. COMMON PROCEDURES FOR OPERATION OF HIGH-PRESSURE EQUIPMENT

The are several activities common to the commissioning, maintenance and use of high-pressure equipment that are governed by applicable law in both the EU and Sweden. In this section of the document, we describe our approach to these activities with explicit reference to relevant legal directives.

2.1. Pressure testing

Pressure testing of equipment is a necessary safety operation to ensure that a pressure device works as intended up to a defined safe working pressure. Pressure testing is required after any repair, modification of replacement of any part of pressurised equipment and may also be prescribed as a component of routine checking of equipment.

The legal requirements when conducting pressure testing in Sweden are governed by directive AFS2006:8. The directive mandates several common provisions for all pressure testing as detailed on its pages 3-5, ESS policy in respect to these requirements is detailed here with reference to the relevant sections in the directive.

2.1.1. Expertise

AFS2006:8 mandates that testing must be carried out by a person ("the Pressure Tester") who has relevant expertise and understands the risks associated with the test. At the ESS, at a minimum, such a person must:

- have successfully participated in training course "Use and Inspection of Pressurised Devices" and hold the related accreditation.
- have successfully participated in training course "Pressure with gas regulation AFS 2006:8" and hold the related accreditation.
- have a good general knowledge of high-pressure systems and practical experience of using these.
- have read and understood the manual for the equipment to be tested.
- have read and understood the associated risk assessment for the work.

The person conducting the pressure test shall put their name on the Pressure Testing Check-list (PTC see §2.1.6) for each test and sign and date

2.1.2. Risk Assessment

Risk assessment (RA) is mandatory for pressure tests. Such RA's have been conducted for all PREMP equipment subject to pressure testing, correspondingly, the PTC includes checkboxes for explicit procedures that the RA mandates for a specific test. If a different system is to be tested, then a separate RA must be conducted and reviewed and this must be documented in the PTC.

Printed copies of all RA's shall be available at the location of the Test Site (currently, testing is restricted to the UPTF, see §2.1.3). These shall have been read by the Tester who shall indicate this by signing the PTC.

2.1.3. Risk Area

Directive AFS2006:8 requires that a risk area is defined for conducting pressure testing. At present, this test area is a dedicated room situated in the west hall of the Utgård laboratory ("Mimir's Well"), which is called the Utgård Pressure Test Facility (UPTF). The UPTF is of a solid construction and, as part of the RA, it shall have been confirmed that, even in a worst case event, the UPTF shall contain all envisaged potential hazards.

In addition, the UPTF has a single entrance point and no windows. As part of standard procedure, systems to be pressure tested shall be positioned inside the UPTF. Prior to testing, it shall be confirmed that no personnel are present in the UPTF room. The door will then be locked with the key being held by the Pressure Tester and clear signage posted to indicate that pressure testing is ongoing.

The design of the UPTF is such that it is suitable for burst testing (see §2.2) for a subset of systems. For each system on which a burst test is conducted, a specific RA shall be conducted and a documented assessment produced to confirm that *in no circumstance* can the burst test inside the UPTF result in injury to equipment or personnel outside of the UPTF. As a general rule, burst tests will entail additional shielding such that the UPTF itself acts *only* as a secondary failsafe protection (see §2.2).

2.1.4. General preparations for testing

Pressure Assessment of system to be tested

With the exception of burst testing (§2.2) devices being tested must have been assessed to demonstrate that the device can withstand the maximum test pressure.

Each system described in this document has a defined maximum operating pressure (MOP). When multiple systems are connected, this MOP shall be defined to be equal to the lowest operating pressure of any of the sub-systems (or of the connecting pipeline is this is lower still). Correspondingly, for systems described in this document, if the test pressure *does not exceed* this MOP, then the content of this document itself contains the appropriate assessment that the equipment can withstand the control pressure.

In the circumstances where a device not covered in this document is to be tested, then several scenarios can be envisaged:

Scenario 1: If the device is commercially obtained and possesses a CE-mark and the pressure test does not exceed normal operating conditions specified by the manufacturer, then no further assessment of pressure performance is needed (other requirements stated in this section, such as conducting an RA, must still be observed)

Scenario 2: If the device is made in-house or by a non-industrial partner (e.g. a university research department), then an assessment must be made of the legal classification of the device, according to the PED. The resulting requirements of the PED shall be followed, which shall include an assessment of the MOP of the device. Determination of MOP *may* include a pressure testing *in excess of MOP* (as per Scenario 3 below), in this case, the test should be treated as a Burst Test (see §2.2).

Scenario 3: Proof Testing. As part of the technical assessment to determine MOP for non CE-marked equipment, or for as standard practice following maintenance or modification of CE-marked equipment, the PED requires testing of devices in excess of MOP by at least a factor of 1.43 (see 2014/68/EU Annex I §7.2). When conducting such a Pressure Test that exceeds the MOP, the test shall be treated as a Burst Test (see §2.2).

Set up of system to be tested

All systems to be tested shall be connected according to instructions in their respective operating manuals.

The system to be tested shall be placed in the UPTF and its stability shall be insured. In particular, any wheeled systems shall have wheel locks engaged, or chucks inserted. The centre of mass of any test system should be such that it cannot topple over. If this is not the case, it should be securely mounted to a non-moving surface. If any non-rigid pressure hosing is to be used, it's length should be as short as possible and it should be securely fixed at both ends.

An initial Leak Test (see §2.3) shall be conducted prior to initiating the Pressure Test.

2.1.5. Test Equipment and inspection

Test Equipment here refers to the devices used to generate, control and measure the pressure applied during the test (this does *not* include the item being tested). Only ESS equipment described in this document may be used for pressure testing, and that equipment shall be subject to the appropriate inspection and maintenance schedules documented here. According to AFS2006:8, a calibrated pressure gauge with an accuracy class of >1 (max 1% deviation at meters maximum range) must be present. As standard, all driving systems at the ESS have transducers with a maximum deviation <0.1%, thus exceeding the standard. Details for these transducers are given in the relevant section of this document and calibration certificates are available (currently these are stored here). As detailed in the relevant driver sub-sections, these transducers are re-calibrated on a regular schedule and the re-calibration date (typically every 12 months) is marked on them. The Pressure Tester shall check the calibration date, prior to a Pressure Test to ensure that it is current.

Prior to a Pressure Test, the entire system to be tested and any separate driving system shall be visually inspected for obvious signs of damage, corrosion, or leakage (for example visible oil on any fittings). If any such problem is found, it shall be addressed prior to initiating pressure testing. This inspection, and any observations or actions shall be noted on the PTC.

It shall be ensured that any air is purged from a hydraulic system and a Leak Test shall be conducted on the system prior to initiating the Pressure Test.

2.1.6. Pressure Testing Checklist for testing in Utgård Pressure Test Facility (UPTF)

Name of tester:

Signature/date:

Indicate system(s) being tested:

Maximator Hydraulic System □	V3 Paris-Edinburgh Cell □	VX6 Paris-Edinburgh Cell □
UoE DAC □	ESS DAC □	□ Other *

* If “Other” is checked: has a Risk Assessment been conducted and posted in the testing area? YES/NO

Tester has read and understood the relevant RA’s □

Procedures prior to Pressure Testing:

- All pressure equipment has been safely installed in the UPTF □
- A visual inspection of all test equipment and test system has been conducted□

  Detail any special observations or actions here:

- Pressure transducer calibration is current □
- An initial leak test has been conducted □
- The UPTF room been searched to confirm absence of personnel □
- The UPTF door has been locked and “Pressure Testing” signage is present □

Does pressure testing include a Burst Test? YES/NO

(If yes, complete the additional checks below)

- The primary shielding design document (PSDD) been reviewed□
- The Primary Shielding has been visually inspected□
- The Primary Shielding is correctly installed according to the PSDD□

2.2. Burst testing

Burst Testing differs from Pressure Testing in that the goal of the activity is to induce failure of a component, for example, by greatly exceeding the safe operating pressure. There are several reasons where such tests are important including:

- In order to determine a safe operating pressure of a component, it may be necessary to experimentally determine the failure pressure in order to apply an appropriate safety factor.
- In order to assess the level of risk associated with failure of a given component (e.g. by measurement with high-speed video or sound measuring equipment).
- During development of prototypes where performance criteria are related to optimising failure pressure.

As a general principle, it shall be ensured that there is no possibility for personnel to be injured as a result of a Burst Test. This entails all of the essential requirements for Pressure Testing (described above) shall be followed and, in addition, further requirements relating to Primary Shielding (§2.2.1) shall followed. In addition, Burst Tests shall only be carried out in the UPTF (or future facilities that are specially designed for this purpose e.g. the proposed PTF in the E03 Sample Environment Laboratory)

2.2.1. Primary Shielding

A central principle of Burst Testing is that primary shielding shall be present during the test. This shielding shall be designed to contain any physical hazard arising from the Burst Test that has been identified during RA. This shielding shall be designed to be sufficient to contain any hazard so that the additional protection of the UPTF itself acts only as a failsafe. This design shall lead to a Primary Shielding Design Document (PSDD) that contains:

2.2.1.1. ***Details of real-World experience*** *where available these shall be the primary guide to safe design. Due to the complexity of ballistic interaction with materials, real-World experience is considered more valuable than calculation, although these are also required.*

2.2.1.2. ***Calculations*** *that shall be conducted that demonstrate that the shielding is sufficient to contain any hazard such as ejected shrapnel. These calculations shall be documented and recorded.*

2.2.1.3. ***Details of any materials testing*** *where a material is relied upon to act as Primary Shielding, it may be necessary to test the strength of this material. This testing shall be documented.*

2.2.1.4. ***Certificates*** *relating to material used that are relevant.*

The PSDD shall be reviewed in additional to the standard Pressure Testing RA.

The Pressure Tester shall ensure that the PSDD exists and shall be familiar with its contents and shall confirm this in the PTC.

The Pressure Tester shall also inspect the Primary Shielding for any defects. If any defects are found, the Burst Test is to be postponed until these are corrected.

The Pressure Tester shall ensure that the Primary Shielding is correctly installed according to design documented in the PSDD.

All of the above are to be recorded on the PTC (§2.1.6) prior to initiating the Burst Test.

2.3. Leak testing

Leak testing is an activity that is frequently carried out to ensure that a freshly made connection (such as between Driver and SEE) is leak tight. It differs from Pressure Testing in that it applies to operational equipment that has a current Pressure Test (according to the schedule in the relevant section of this document) and has not been modified or damaged since of equipment has been conducted since that test. In addition, the pressure used in a leak test shall never exceed the MOP.

The procedure to first conduct an initial leak test is the following:

- Conduct a visual inspection of the fittings to be tested and then to introduce a test pressure of less than or equal to 10% of the MOP.
- Allow the system temperature to stabilise
- Observe the pressure in the system over time to determine if it is dropping

If a leak is observed, then action should be taken to correct the leak and then further tests can be conducted to determine if the action is successful.

If the leak test is successful, it is allowed to then further increase the pressure (without exceeding the MOP) in order to conduct additional leak tests under conditions of higher operational pressures.

3. DRIVER: PORTABLE PRESSURE DRIVER

The Portable Pressure Driver (PPD) is a mobile device holding two independent pressure generating sub-systems: the Maximator Hydraulic System (MHS) and the PACE. It is a driving system, envisaged to be transported to a beamline or laboratory and deliver and control pressure, via either hydraulic or gas media to different sample-environment equipment. It is also designed to transport a single piece of floor-mounted (up to Level 2) SEE (as defined in ESS-38078).

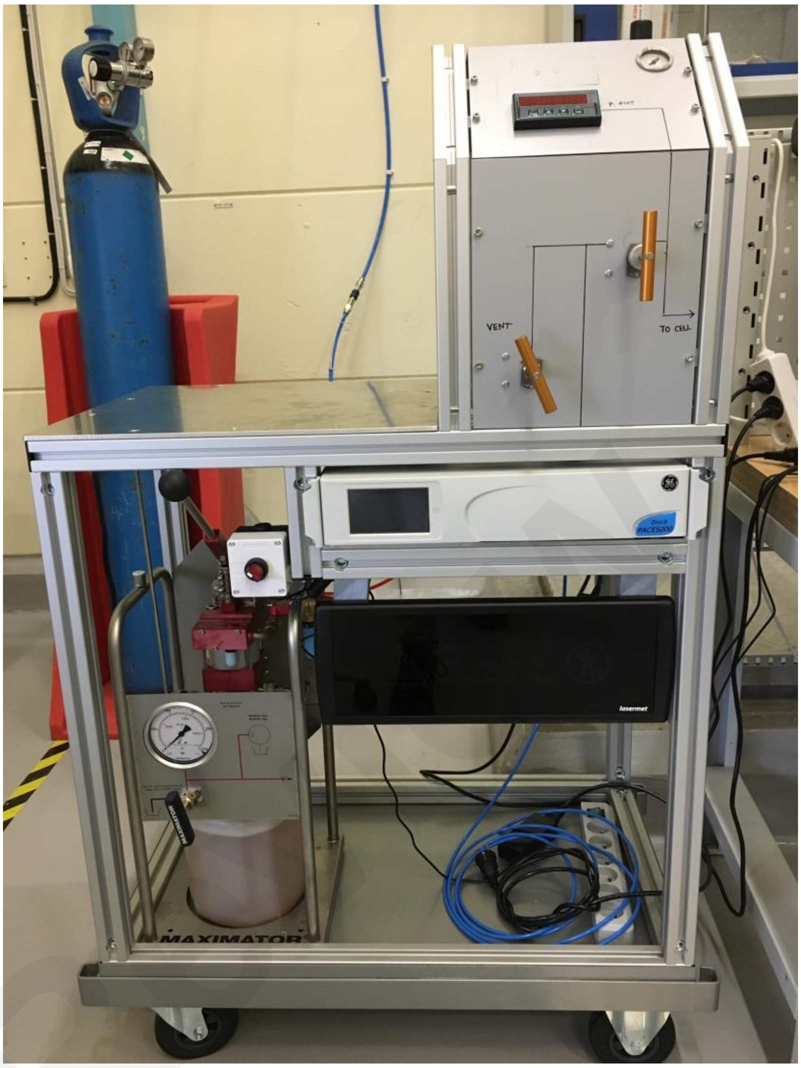


Figure 1 **Photograph of the Portable Pressure Driver in its current form. This is a prototype device, so it will evolve over time.**

3.1. Description of components

- A wheeled frame holding sub-systems and containing additional oil drip tray and a mounting table compatible with Level 2 SEE interface standard.
- The Maximator Hydraulic System (MHS), providing a manual, 2000 bar, air-driven hydraulic supply (see §4).
- The GE PACE 5000 (PACE), providing automatic control of an input gas pressure up to 200 bar (see §5)

3.2. Piping and Instrumentation diagram (P&ID)

See diagrams for sub-systems (§4 and §5)

3.3. Declaration on the legal status (classification) of the device (where appropriate)

See declarations for sub-systems (§4 and §5)

3.4. A risk assessment for both maintenance and operation of equipment

SYSTEM: PORTABLE PRESSURE DRIVER												
				Initial Risk Rating				Residual Risk Rating				
Number	Activity or Event	Hazard	Who might be harmed and how?	Likelihood	Severity	Risk H,M,L	Actions to mitigate Risk - controls	Likelihood	Severity	Risk H,M,L	required training	additional reference
1	SEE falls off PPD or causes it to topple	physical injury	operator, heavy SEE fall causing an injury. Or, entire cart topple due to top heavy SEE.	2	3	M	1) Operator must bolt SEE onto MHS 2) Weight limit displayed on MHS must be respected	1	2	L	Instruction in use of PPD	PPD ops manual
2	Lose control of PPD on slope	physical injury, damage to equipment	passersby collision with PPD	2	3	M	1) Define safe routes avoiding slopes 2) Train operators to be aware of hazard	1	3	L	Instruction in use of PPD	PPD ops manual
3	Tripping on utility connections	tripping/fall	passersby or operator	4	1	M	1) follow manual to safely make utility connections	3	1	L	Instruction in use of PPD	PPD ops manual
4	Short circuit resulting in electrification of metal frame	electrical hazard	operator	2	5	M	1) Ensure trolley is earthed whenever energised. 2) Electrical installation completed by qualified person	1	5	L		

Also see attached risk assessments for sub systems (§4 and §5)

3.5. A manual describing both maintenance and operation of equipment

This manual is also copied into attached appendices and a printed copy is available on the device itself.

3.5.1. Transportation of PPD

Prior to transporting the PPD between locations, the following must be observed:

- Any SEE connected to the PPD should either be fully downloaded or loaded, but isolated from the pressure-driving systems by means of an isolation valve. This applies to equipment connected to both the PACE and MHS sub systems.
- MHS should then be vented to zero pressure by opening either of vent valves V2 ior V3 and confirming complete pressure loss on analogue G1. At least one of Valves V2 or V3 should remain open during transport (see §4.4 for detailed location of valves on MHS).
- The PACE shall also be vented by entering a set point of 1 bara (0 barg). Once at atmospheric pressure, the PACE should be set to "Measure" mode (see PACE manual)
- Any SEE mounted on the PPD shall comply with marked weight limits.

- If SEE is mounted on the PPD, this shall be bolted down to ensure it cannot disengage and fall off.
- All utility connections (electrical power, compressed air or gas, hydraulic lines and ethernet) must be safely disengaged.
- There shall be no loose tools placed on top surfaces of the PPD.

3.5.2. Set-up of the PPD

Once the PPD reaches the location where it is to be used:

- It shall be positioned safely, in a convenient location relative to the necessary utility connections.
- Both brakes shall be fully engaged.
- Utility connections shall be made and, where necessary hydraulic or gas connections Leak Tested (see instructions for sub-systems and §2.3). PPD shall be positioned to minimise tripping hazard from utility connections, which shall not lie across walkways.
- Where necessary, the SEE will then be transferred from the PPD to a secondary location (e.g. installed at the sample position of an instrument)
- The hydraulic or pneumatic connection between the SEE and the PPD should be re-established following the relevant procedure for the specific sub-system.
- Where connections are re-made these shall be Leak Tested as appropriate for the relevant sub-system.

3.5.3. Maintenance and modification of the PPD

When performing maintenance on the PPD

- All pressurizing sub-systems must be downloaded to zero pressure and made safe.
- Unless necessary for maintenance operations, utilities shall be disconnected.
- Any replacement of parts must use CE-marked components of appropriate pressure rating for the relevant sub system.
- Any modification that results in a change of the total system volume or MOP shall be assessed for possible impact on classification.

3.6. Where appropriate a maintenance schedule for the device

The PPD does not have a maintenance schedule, however, subsystems do.

3.7. Record-keeping requirements

Records are not kept for the PPD, although sub-systems may need records.

3.8. Required training for operators

No specific training is required for the PPD. However, specific subsystems do have requirements, as described in the relevant sections of this document.

4. DRIVER: MAXIMATOR HYDRAULIC SYSTEM (MHS)

4.1. Summary of purpose of equipment

The MHS is an air-driven hydraulic pump capable of generating and monitoring hydraulic pressures up to 2000 bar (although see §4.2.1 below on temporary definition of MOP) and allowing controlled delivery of these to drive SEE either on beamlines or in laboratories at the ESS. At the present time, the only item of SEE that will be driven by the MHS are Paris-Edinburgh type devices. Other hydraulic systems may be used in the future.

4.2. Specification

4.2.1. Operating Pressure

The design operating pressure of the MHS is 2000 bar. However, during development activities, have chosen to define a temporary lower MOP of 1000 bar. We have included safety features to prevent an operator exceeding this value (see §4.3.2)

4.2.2. Operating Temperature

The MHS is designed to operate under normal ambient temperature in the laboratory. Some frictional heating of oil can be expected but this shall not exceed 40°C during normal operation.

4.3. Description of the components

Tables summarise the components comprising the MHS:

Table 3 Components of MHS pipeline

Component	Number or total length (cm)	Manufacturer	Part number	Manufacturer MOP (bar)	Total Vol (cc)	Date of procurement
HP tubing	213	SITEC	730.234	2900	9.636	02/2018
HP elbows	3	SITEC	720.1532	4000	0.143	02/2018
HP T-fittings	1	SITEC	720.1533	4000	0.048	02/2018
3-way valve	1	SITEC	710.534	4000	0.212	02/2018
Needle valve	1	SITEC	710.5312	4000	0.212	02/2018

Table 4 Components of MHS

Component	Number or total length (cm)	Manufacturer	Part number	Manufacturer MOP (or relief pressure) (barg)	Total Vol (cc)	Date of procurement
Maximator pump	1	MAXIMATOR	3110.0214 /TYP: M 189-HL-4H	2200	9.6	04/2018
Pressure transducer	1	ESI	HP1003	2000	<0.1	
Air pressure relief	1	Caleffi	311460	6	N/A	06/2019
Oil relief valve	1	SITEC	729.4003-2	600	<0.1	08/2019
Oil Safety relief valve	1	SITEC	720.5531-1	1100	<0.1	08/2019
Rupture disc	1	SITEC	720.5032	1250	<0.1	08/2019

Table 5 MHS Sub system Specification

Hydraulic fluid	Total system volume	Max operating pressure (barg)	Minimum/Max. operating temperature	PS.V (bar.L)
HPL 467 mineral oil in accordance with DIN 51524	0.011	1000	-20/+60	22

4.3.1. Pressure transducers

The MHS has an ESI HP1003 transducer with an output of 4-20mA and calibrated linearity of ±0.1% (Best fit straight line). Calibration certificates are stored here. The recalibration schedule is 12 months and the due date is for the next calibration is indicated on the transducer body.

4.3.2. Safety components

The MHS has three independent pressure-limiting systems to ensure the MOP is not exceeded. Relevant manufacturers certificates are stored here.

The first safety device is a pressure relief valve that limits the input air pressure of the system. As shown in Figure 3, the output hydraulic pressure is a fixed multiple of the inlet gas pressure - given by the ratio of areas on a double-sided piston. This is illustrated in the test data given in Figure 2, which shows a good linear dependence between input and output (small glitches are observed when pilot valves trigger to reset the piston position at the end of a stroke). The relation shows that output pressure is related to input pressure by a factor of 202.

A Caleffi 311460 safety relief valve, set to 6 bar, has been installed. Measurement with the calibrated transducer of the PACE show that the Caleffi opens at 5.6 barg. This limits the output pressure to 1131.2± 32 barg = 1.1*MOP.

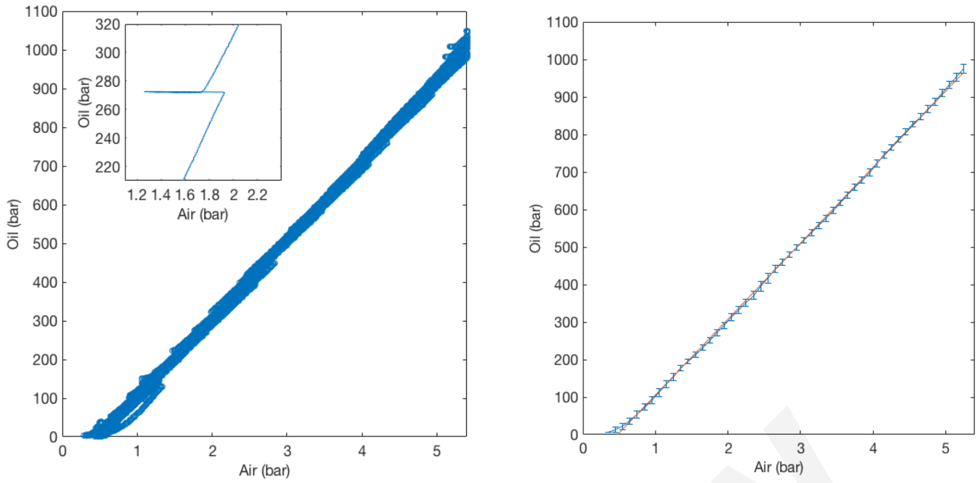


Figure 2 (Left) Blue circles show test data from 10 successive runs showing output oil pressure as a function of input air pressure for the MHS. Inset shows a close up a behaviour at the end of piston stroke: oil pressure is held by non-return valve while pump head recharges. Oil pressure begins to increase again when sufficient pressure is developed in the pump head. This occurs at a lower air pressure than at the beginning of the stroke due to frictional effects. These “dips” have been excluded from the plotted test data (Right) A histogram of all 10 data sets with mean absolute deviation given as error bars. These take account of the “offsetting” effect of the pump cycle. The red line passing through the data points is a linear regression with a slope of 202.5.

The second pressure limiting device acts on the high-pressure hydraulic line and this is a calibrated safety relief valve (SITEC 720.5531-1). The safety valve is set in the factory to 1100 bar as per manufacturer instruction that it should be set at 1.1*MOP. This acts as a failsafe for the pressure relief valve on the air-side of the system.

The third pressure limiting system is a rupture disc, (SITEC 720.5032). As per manufacturer instruction, this is set at 1.25xMOP = 1250 barg.

In addition to the three safety systems described above, an additional ‘low-pressure’ relief valve (LPRV, SITEC 729.4003-2) set to 600 bar has been installed on the hydraulic line. The intention of this valve is to allow the possibility to limit the operating pressure to ‘low risk’ operations of the MHS when a PE cell with ZTA anvils is connected (see §6.6.3), which is an envisaged need during user operations. This component has a bypass feature to allow properly-trained operators to exceed this limiting pressure.

4.4. Piping and Instrumentation diagram (P&ID)

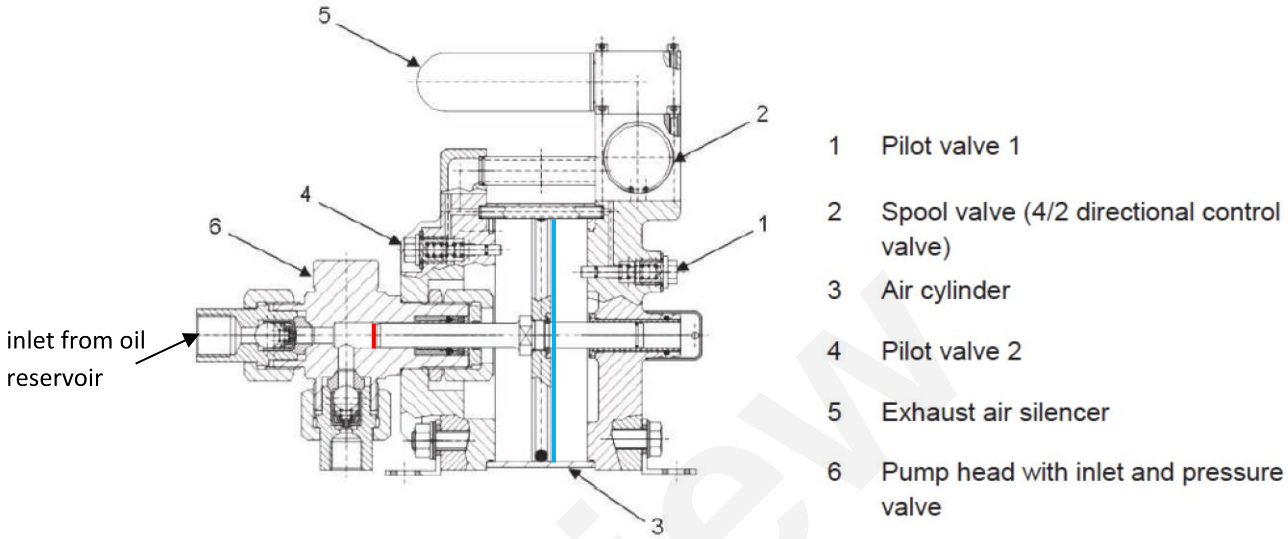


Figure 3 Overview of Maximator pumping head. The force generated across the area indicated in blue on one end of the piston (within the air cylinder) is communicated to the much smaller area indicated in red at the other end of the piston in the pump head, where the hydraulic oil is pressurised. Thus, hydraulic pressure is related to air pressure by a geometrically-fixed multiple, measured to be 202.

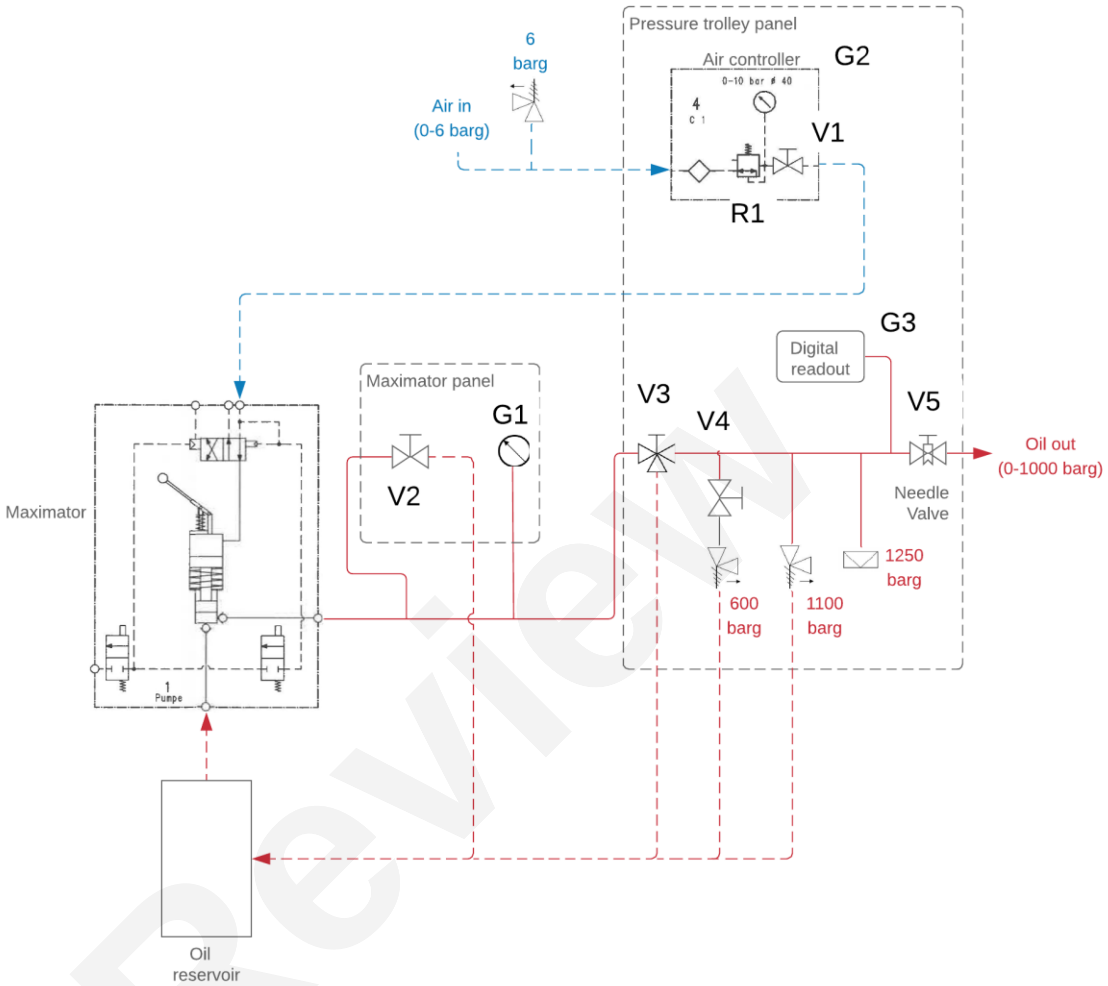


Figure 4 Maximator hydraulic system P&ID dashed blue lines indicate low-pressure air lines. Dashed red lines indicate low-pressure hydraulic lines. Solid red lines indicate high-pressure oil lines

4.5. Declaration on the legal status (classification) of the device (where appropriate)

4.5.1. Classification of the MHS according to AFS2017:3

Working fluid: The hydraulic oil used by the MHS is a class 2a fluid.

Pipeline: The standard piping used in the MHS is SITEC brand ¼” HP tubing, rated for safe operation below 4000 bar (SITEC part no. 730.234). In addition, connections are made with a combination of elbow fittings (SITEC part no. 720.1532); T-fittings (SITEC part no. 720.1533) and valves (3-way SITEC part no. 710.534 and needle (SITEC part no. 710.5312).

As the operating temperature of the liquid is < 65°C, the pipeline is unclassified (§11 AFS2017:3)

Pressure vessel: In addition to piping, the MHS contains the maximator pump itself. The maximator is CE-marked and is compliant with the machinery directive 2006/42/EC. The stroke volume of this component is 0.0006L (0.6cc). Thus, at maximum operating pressure (PS) of 2000 bar, the product PS.V = is 1.2. In order to be classified, PS.V must be > 5000. (§10 AFS 2017:3)

Conclusion: according to AFS 2017:3 the MHS is *unclassified*

4.5.2. Classification according to 2014/68/EU (PED)

The MHS procured as a fully-compliant (CE-marked) component from a European manufacturer.

4.6. A risk assessment for both maintenance and operation of equipment

System: Maximator Hydraulic System												
				Initial Risk Rating				Residual Risk Rating				
Number	Activity or Event	Hazard	Who might be harmed and how?	Likelihood	Severity	Risk H,M,L	Actions to mitigate Risk - controls	Likelihood	Severity	Risk H,M,L	required training	additional reference
1	slow oil leak leads to oil on floor	slipping hazard	passersby or operator could slip	4	3	M	1)Design of PPD includes drip tray to catch spills 2) Routine maintenance includes leak test 3) Anti-vibration fittings in MHS prevent loosening of connection in use	1	3	L		MHS ops manual
2	Leak from low P input (high pressure air (<= 6 bar)	aeroembolism	operator could suffer aeroembolism	2	5	M	1) Build guard around air connection to prevent air contacting skin 2) Follow procedure to leak test connection at low pressure before increasing pressure	1	1	L	"Use and Inspection of Pressurised devices (AFS2017:3)"	MHS ops manual
3	Incorrectly made output connection (oil pressure)	slipping injury	passersby or operator could slip	4	3	M	1) After making connection, always perform a quick leak check at low pressure 2) Monitor pressure throughout use to detect if a leak occurs	2	1	L	Instruction in use of MHS	MHS ops manual
4	failure of internal high-pressure hydraulic components	physical Injury from projected oil	operator could be injured by high-pressure oil	1	5	M	Build secondary shielding around MHS	1	1	L		
5	unexpected pressure generation during set up	pressurised oil	operator could be injured by high-pressure oil	3	4	M	1) when setting up MHS, vent valve (V2 or V3) **must** be open 2) when setting up MHS, input air valve V1 **must** be closed	2	4		Instruction in use of MHS	MHS ops manual Maximator manual
6	Exceding maximum input pressure	physical Injury from projected oil	operator could be injured by high-pressure oil	1	5	M	1) Build secondary shielding around MHS 2) ensure supply pressure is limited to 6 bar	1	1	L	Instruction in use of MHS	MHS ops manual
7	Impact from heavy pressure tubing	physical injury from impact	operator could be hit by heavy tubing when a difference in floor level exists between MHS and connected SEE	2	2	L	1) training so operator is aware of risk	2	2	L	Instruction in use of MHS	MHS ops manual

4.7. A manual describing both maintenance and operation of equipment

4.7.1. Pressure testing the MHS

The MHS is currently operated with an MOP of 1000 barg and has two independent safety systems set at 1.1xMOP. The MHS shall be Pressure Tested (§2.1) every 12 months to a pressure of 1.1xMOP = 1100 barg. In addition, if any modification is made or repair conducted, the MHS shall be retested. The date of the latest pressure test shall be recorded in the MHS maintenance manual, attached to the MHS.

4.7.2. Leak testing the MHS

SEE equipment is frequently connected and disconnected from the MHS. Each time such a connection is made, the combined MHS-SEE system shall be Leak Tested (§2.3)

4.7.3. Connecting and disconnecting SEE to/from the MHS

SEE will need to be frequently be connected and disconnected from the MHS during installation on an instrument. It will normally be necessary to maintain a sealing load on the SEE during this operation (e.g. in the case of a PE cell with standard toroidal gaskets 7 tonnes is required). Additionally, it is sometimes necessary that the SEE will have a larger load of up to 20 tonnes (specific scenarios and limits are described in separate sections on specific SEE).

Prior to any operation, the following initial checks should be performed:

- The operator shall check the calibration date of the MHS transducer G1 to ensure that this is current.
- A visual check should be made of all hydraulic connections to identify any obvious signs of damage or oil leakage.
- If the MHS is connected to an air supply, the main air isolation valve V1 shall be closed to ensure no pressure can be developed in the system.

The following procedure should be used to connect SEE to the MHS.

- The MHS shall be connected to the SEE using a loose, finger-tight connection. At this point, a small amount of oil should be bled through to minimise trapped air in the system. This should be done by gentle use of the manual hand pump of the MHS. Once oil is seen to exit through the bleed hole of the HP connection, the connection can be made tight, to the specified 30 Nm.
- If the SEE is unloaded (at zero pressure with isolation valve open), the connection is now complete and ready to use. If the cell is loaded, the additional steps below must also be followed to balance the system:
- The internal SEE pressure P_{SEE} must be visible to the user and the internal pressure on the MHS increased to exactly match this to within < 1 bar (correct calibration of the gauges as per initial checks is necessary to ensure there is not an offset between different gauges).
- At this point, the isolation valve on the SEE should be opened slowly, while continually monitoring P_{SEE} to ensure it remains stable.

The following procedure must be followed to disconnect pressurised SEE from the MHS.

- After any pressure change, the SEE must be left to stabilize for at least 5 minutes prior to disconnection. If the SEE is at non-ambient temperature, it shall be allowed to return to ambient temperature prior to the 5-minute stabilisation time.
- It is required that the SEE has an isolation valve enabling it to be closed off from the MHS.

- It is required that a pressure transducer, or other pressure-measuring device is connected on the SEE-side of the isolation valve, thus this will display P_{SEE}, the internal pressure on the equipment (versus the MHS pressure P_{MHS}).
- The first step in disconnection is to close the isolation valve separating the SEE from the MHS. At this point, the SEE transducer must be powered up so that P_{SEE} can be observed.
- The MHS should now be downloaded by first closing the needle valve V5 to isolate the connection to the SEE and then venting the remaining system by opening either of the vent valves V2 or V3 and confirming loss of pressure on Gauges 1 or 2. At this stage, needle valve V5 should be gradually opened allowing the pressure in the SEE connection to begin dropping. At this point P_{SEE} should be inspected. If P_{SEE} is stable, this confirms the isolation valve is functioning correctly. At this stage, is the user is free to fully open V5 to complete the download of the MHS.
- At this stage, the SEE is isolated from the MHS, which is at zero pressure. The output needle valve V5 should now be reclosed, to minimise any oil loss. The SEE may now be disconnected taking care to ensure no torque is placed on the isolation valve. Once disconnected, any open hoses/piping etc. shall be sealed to ensure no loss of oil and prevent slipping hazard.

4.7.4. Operating SEE using the MHS:

- The operating principle of the MHS is to set and control the internal output pressure by setting the input air pressure, which is a fixed factor (measured to be 202) below the output. This pressure is then communicated to connected SEE via the needle valve V5. The rate at which P_{SEE} increases is determined by a combination of the pressure differential between P_{SEE} and P_{MHS}, by the opening setting of the needle valve and by the relative volumes of the MHS and the SEE.
- For certain SEE, changes in pressure must be conducted at a controlled low rate (see specific operating instructions for each SEE). As the relative volumes of the SEE and MHS systems are typically fixed, this rate can be minimised by ensuring a low differential between P_{SEE} and P_{MHS} in this case, needle valve V5 can allow extremely slow changes in pressure.
- To establish the desired P_{MHS} the input air pressure can be adjusted using regulator R1 and monitored by inspection of the analogue manometer in the air controller. Subsequently, the flow rate (and therefore the rate of increase of P_{SEE}) can be adjusted using output needle valve V5.)
- After changing pressure, P_{SEE} shall be monitored to ensure that it is stable. Typically, pressure increase will induce warming of the fluid causing a subsequent drop in pressure as the fluid cools. It may be necessary to compensate for this in cases where a stable pressure is critical. However, if P_{SEE} continues to drop after ~ 5 mins following pressure change, this could indicate a leak and should be investigated.

- It should be noted that normal changes in ambient temperature can have a significant effect on total system pressure: which drops upon cooling and increases upon warming. This effect becomes less pronounced as the volume of the connected SEE increases.

4.8. Where appropriate a maintenance schedule for the device

Normally piping components should not need maintenance, beyond occasional tightening of HP fittings and anti-vibration fittings are used to minimise this. Maintenance of the maximator pump shall be followed as per the maximator manual.

Pressure transducers shall be recalibrated on a 12 months interval.

Maintenance activities on the MHS shall be recorded in a maintenance manual kept in a folder attached to the device.

4.9. Record-keeping requirements

A log-book shall be maintained detailing operations of the MHS including date/time of use, maximum pressure, and any observations on performance.

In addition, a maintenance schedule shall be maintained detailing maintenance activities on the device.

4.10. Required training for operators

Users of the MHS must complete all required ESS training for the following activities:

- Use of compressed air/gas
- Use of pressurised hydraulic fluids

Additionally, they must receive specific instruction from a trained operator of the MHS

5. DRIVER: GE PACE 5000 PRESSURE CONTROLLER

5.1. Summary of purpose of equipment

The GE PACE 5000 pressure controller (PACE) takes a gas input pressure P and provides control and monitoring of the output pressure. It operates using either compressed air or dry N_2 or He gas inputs. This provides a way to control pressure on membrane-driven devices such as DACs (see §7).

5.2. Description of the components

The PACE 5000 is a self-contained commercial unit consisting of a touchscreen display and internal components that control pressure and external components that allow connection of a gas supply to the input and SEE to the output. Electronic connections are also provided to allow remote control of the device.

According to the manufacturer's documentation it is classified within the PED as manufactured according to sound engineering practice (PED, Article 4.3).

5.3. Piping and Instrumentation diagram (P&ID)

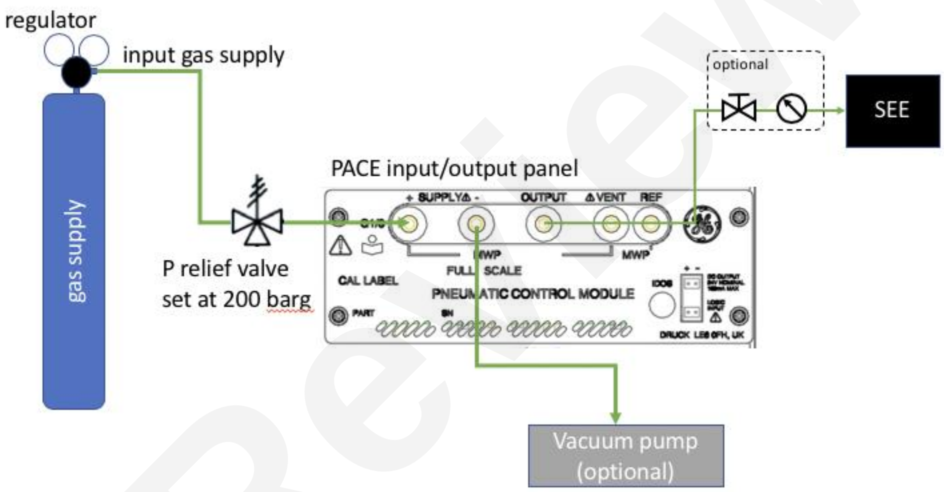


Figure 5 typical connection diagram when using PACE 5000. Note, P relief valve is required by manufacturer, but hasn't yet been implemented. Instead, we are using a regulator with a maximum output pressure of 50 barg, thus ensuring MOP of the PACE cannot be exceeded.

5.4. Declaration on the legal status (classification) of the device (where appropriate)

According to manufacturer, devices is classified as manufactured according to sound engineering practice.

5.5. A risk assessment for both maintenance and operation of equipment

SYSTEM: GE PACE PRESSURE CONTROLLER

Number	Activity or Event	Hazard	Who might be harmed and how?	Initial Risk Rating: Likelihood	Initial Risk Rating: Severity	Initial Risk Rating: Risk H,M,L	Actions to mitigate Risk - controls	Residual Risk Rating: Likelihood	Residual Risk Rating: Severity	Residual Risk Rating: Risk H,M,L	required training	additional reference
1	gas leak during connection, operations or disconnection to PACE	High pressure gas	operator could be harmed by physical injury (whipping pressure line)	2	4	M	1) Training for use of compressed gas 2) Training in use of PACE	1	4	L	Instruction on use of PACE	PACE ops manual
2	gas leak during connection, operations or disconnection to PACE	High pressure gas	Operator could be harmed by aeroembolism	2	5	M	1) Training for use of compressed gas 2) Training in use of PACE 3) wear appropriate gloves, when making connections	1	5	M	"Use and inspection of pressurised (AFS 2017:3)" Instruction on use of PACE	PACE ops manual
3	Incorrect set-point creating dangerous pressure conditions	explosion	Operator and passersby could be physically injured	3	4	M	1) software limits preventing unsafe pressure request 2) pressure relief attached to connected SEE	1	4	L	"Use and inspection of pressurised (AFS 2017:3)" Instruction on use of PACE	PACE ops manual

5.6. A manual describing both maintenance and operation of equipment

5.6.1. Connecting and disconnecting SEE from PACE

SEE will need to be frequently connected and disconnected from the PACE during installation on an instrument. Often the SEE will be pre-loaded in a SE-workshop and, thus, additional steps are needed to make and the connection (see below).

Prior to any operation, the following initial checks should be performed:

- It shall be confirmed that calibration of all transducers and pressure readouts in the system should are current (calibration certificates for the PACE are available here).
- A check should be made of all gas connections to identify any obvious signs of damage or leakage. This should be done setting the PACE to control at a small pressure of 1-2 barg and using a leak-detecting fluid (e.g. Snoop® or equivalent) to identify leaks. As an additional safety step during this operation, the regulator controlling input pressure should be set to 2 barg, thus preventing any accidental exceeding of the initial leak test pressure.
- It shall be ensured that appropriate gas supply is connected. This is especially important when cryogenic operation is envisaged and users should be aware of possible phase changes in the gas supply. In the cryogenic case the most appropriate supply gas is He, which should be used to minimise risk of supply gas condensing in the pressure lines.
- The source pressure from the input gas cylinder pressure shall not exceed 200 barg.

The following procedure shall be used to connect SEE to the PACE.

- The PACE should be physically connected to the SEE.
- If the SEE is unloaded (at zero pressure with isolation valve open), the connection is now complete and ready to use. However, where cryogenic operation is envisaged, it is additionally necessary to fully evacuate the SEE (and connecting capillaries) to avoid the presence of any moisture, which can freeze and induce a

blockage. If the cell is loaded, the additional steps below must also be followed to balance the system:

- The internal SEE pressure P_{SEE} must be visible to the user and the internal pressure on the PACE increased to exactly match this to within < 1 bar (correct calibration of the gauges as per initial checks is necessary to ensure there is not an offset between different gauges).
- At this point, the isolation valve on the SEE should be opened slowly, while continually monitoring P_{SEE} to ensure that it remains stable.

The following procedure must be followed to disconnect SEE from the PACE.

- After any pressure change, the SEE must be left to stabilize. This can be confirmed by monitoring the *effort* value of the PACE, when this drops to 0, the pressure is stable. This typically takes <1 min, however, can take longer if the SEE is at non-ambient temperature. e.g. in the case where the SEE is at cryogenic temperatures, the gas input at room temperature will cool, resulting in a pressure drop, which the PACE will compensate for. If *effort* remains non-zero after ~1 min, and the SEE is at ambient temperature, this could indicate a leak and steps (beginning with a Leak Test) should be taken to identify and correct this.
- It is recommended that the SEE has an isolation valve enabling it to be closed off from the PACE. In this case, there shall also be a pressure transducer between the SEE and the isolation valve.
- If the SEE is to be fully downloaded, then any isolation valve between it and the PACE unit shall be open and the control pressure reduced to 0 barg at an appropriate rate. The rate can be SEE dependent and, in some cases, must be sufficiently slow to avoid damage to the SEE.
- If pressure is to be maintained on the SEE, the first step in disconnection is to close the isolation valve separating the SEE from the PACE. At this point, the SEE transducer must be connected to a readout so that P_{SEE} can be observed.
- The PACE should now be downloaded. This is done by first dropping the control pressure to a value of P_{SEE} – 5 bar. At this point P_{SEE} should be inspected for a period of 30-60 s. If P_{SEE} is stable, this confirms the isolation valve is functioning correctly. At this stage, the user is free to rapidly download the system by setting the control pressure to 0 barg.
- At this stage, the SEE is isolated from the PACE, which is at zero pressure. The SEE may now be disconnected taking care to ensure no torque is placed on the isolation valve.

5.6.2. Operating SEE using the PACE:

- The input pressure to the PACE limits the maximum pressure that may be delivered to the SEE. This should be adjusted, via the regulator, to an appropriate value for the SEE and the planned experiment (e.g. if the maximum pressure required is 40barg, there is no need to have an input pressure exceeding this).

- The output pressure to the SEE is set by adjusting the set point of the PACE. At this point, if the PACE is in *control* mode, the output pressure will immediately begin to increase/decrease to match the requested value. Each particular SEE may have limits on maximum pressure and/or limits on the rate of change of pressure. These limits shall be imposed as soft limits on the PACE operation (by following instructions in the PACE manual).
- Certain SEE may have further operational requirements described in their operating manual, which must be consulted prior to use.

5.7. Where appropriate a maintenance schedule for the device

The maintenance schedule as described in the PACE manual shall be followed. In particular, note that the inbuilt pressure transducer shall be recalibrated at least once yearly.

5.8. Record-keeping requirements

For calibration purposes, the gauge should be marked with a sticker indicating last date of calibration.

5.9. Required training for operators

Users of the MHS must complete all required ESS training for the following activities:

- Use of compressed air/gas

Additionally, they must receive specific instruction from a trained operator of the PACE

6. SEE: PARIS-EDINBURGH (PE) PRESS

6.1. Summary of purpose of equipment

The PE press is a commercially-manufactured, compact, 50-200 tonne capacity press, designed for *in situ* pressurisation of samples during neutron measurements. By applying force on samples contained in an opposed-anvil arrangement, sample volumes of 20-100mm^3 can be taken to pressures in the vicinity of 20 GPa. These samples can then be studied using neutron diffraction, spectroscopy, imaging or QENS measurements. The PE press is considered a critical item of sample environment for early operations at the ESS.

6.2. Description of the components

6.2.1. The PE Press

The PE press comes in two primary variant designs: the V-class and VX-class. The V-class has a 4-fold geometry, while VX-class has a 2-fold geometry. Both classes of press come in a range of capacities. The class and maximum-operating pressure for each press is stamped on the press body.

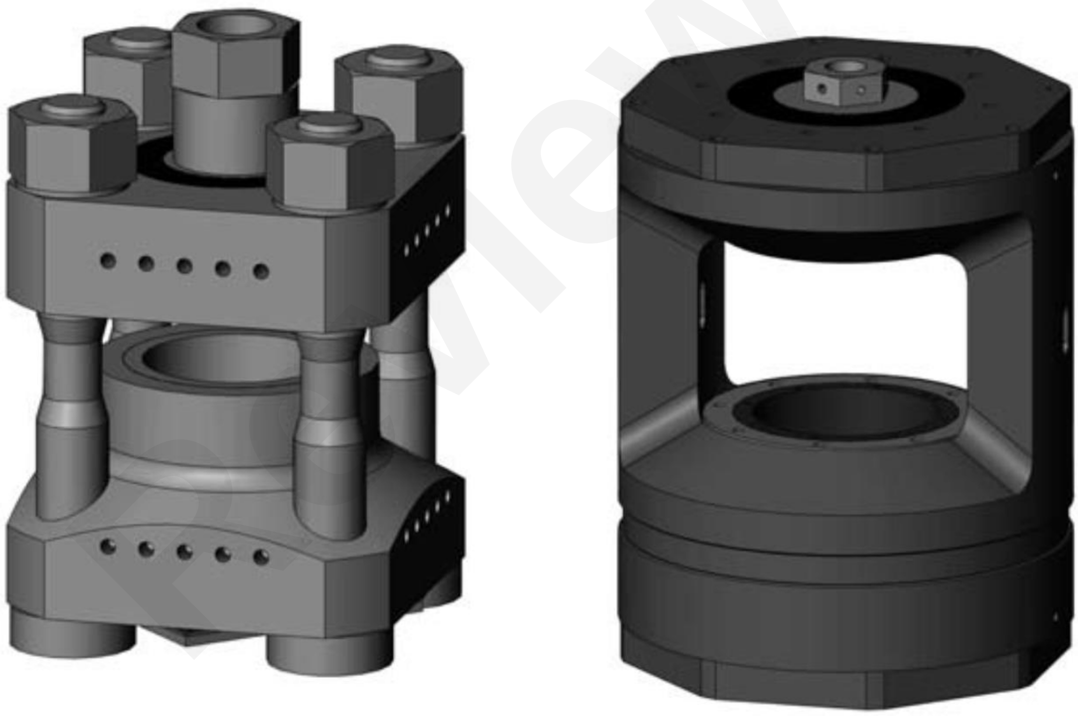


Figure 6 two designs of PE-press, left shows V-class, right shows VX-class

The press includes a piston situated within a cylinder, the piston being driven forward in the cylinder by means of hydraulic fluid (typically oil, but fluid pentane and He gas are also used depending on temperature) with pressure maintained by a seal appropriate for the fluid used (e.g. for hydraulic oil a simple o-ring is used). The force generated by the hydraulic oil is determined by the product of the area of the rear of the piston and the oil pressure. Thus, there is a simple linear dependence between oil pressure and force generated.

The ESS currently operates the PE press models described in Table 6.

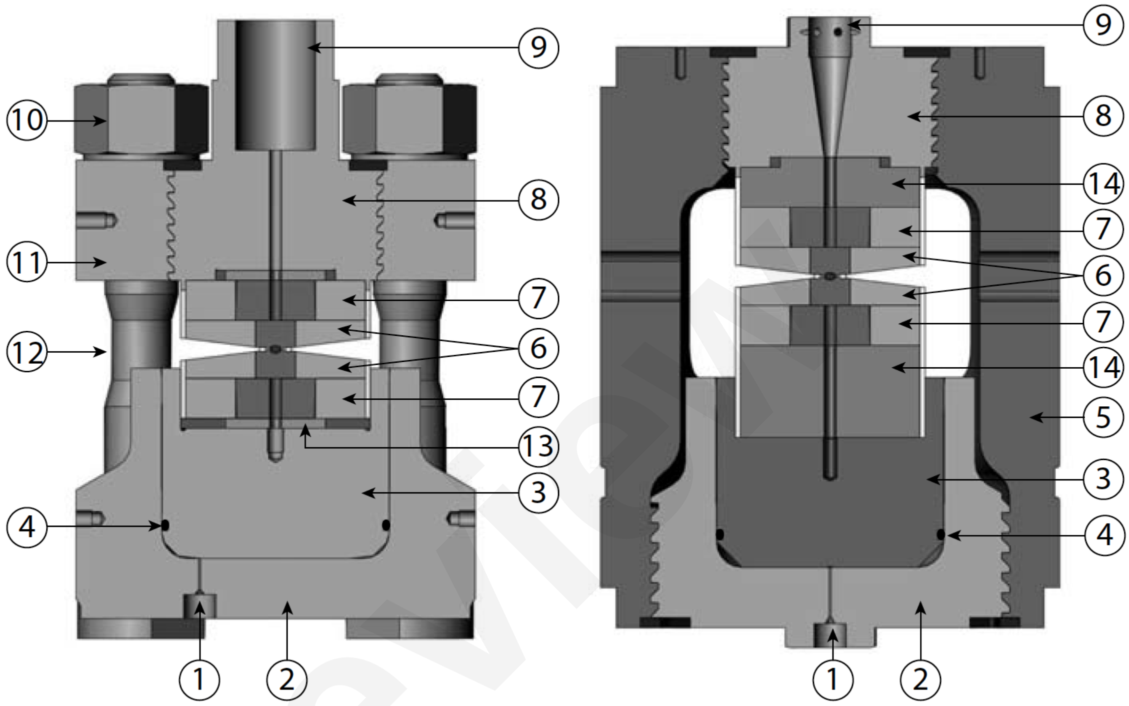


Figure 7 Cross section of PE presses V-class (left) and VX-class (right). (1) Hydraulic fluid inlet; (2) cylinder; (3) piston; (4) O-ring seal; (5) load frame; (6) anvils; (7) backing plates (seats); (8) breech; (9) front collimator; (10) nut; (11) top platen; (12) tie rod; (13) backing disc; (14) steel spacer)

Table 6 ESS PE-press inventory

Press Model	Max. Operating Pressure (bar)	MOP at ESS (bar)	Max. tonnage at ESS (tonnes)	No. Devices	PREMP ID	
V3	2000	2000	200	1	PRE-V3-001	Owned by LLB, on loan for a period of 2 years from 25/08/2019 until 24/08/2021.

Table 6 ESS PE-press inventory

Press Model	Max. Operating Pressure (bar)	MOP at ESS (bar)	Max. tonnage at ESS (tonnes)	No. Devices	PREMP ID	
VX6	2000	2000	130	1	PRE-VX6-001	Owned by Sorbonne University, on loan for a period of 2 years from 11/06/2019 until 10/06/2021

6.2.2. Anvil assembly

Many variants on anvil assembly are possible. However, at present, the ESS uses only standard (or "Los Alamos") profile single-toroid anvils. These anvils can be fabricated from a range of materials including tungsten carbide, sintered diamond and zirconium-toughened alumina (ZTA). At present only ZTA anvils are used at the ESS.

The anvils act to intensify the hydraulic pressure acting on the piston by concentrating the resulting force onto a smaller area. The pressure-load curve is approximately linear and, with standard-profile anvils, the force in tonnes can be divided by 10 to estimate sample pressure in GPa. Thus, *50 tonnes is approximately ~5 GPa*, although the exact value can vary depending by as much as 1-2 GPa depending on the details of the loading. For different anvil profiles, this relation will change.

For ZTA anvils, the maximum operating pressure is ~ 8 GPa and anvil failure becomes increasingly like as pressure is increased beyond this value.

6.2.3. The gasket

The opposing anvils apply pressure to a sample which is contained by a gasket, which is usually made out of metal. The metal used can also limit the maximum sample pressure. TiZr is commonly used for neutron-diffraction applications as it is null-scattering (n.b. this material is flammable). For offline testing, steel gaskets will be used. For both steel and TiZr gaskets, their maximum operating pressure exceeds that of the anvils.

6.3. Piping and Instrumentation diagram (P&ID)

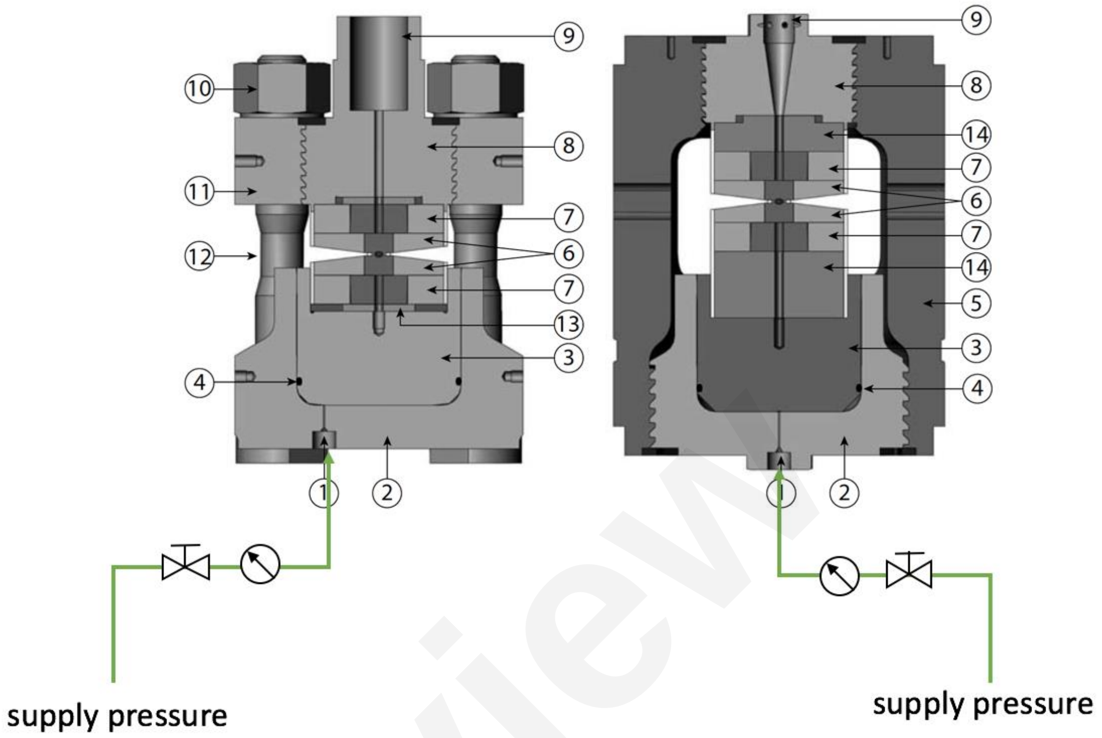


Figure 8 Diagram shows the hydraulic connections to the PE press. Green lines indicated hydraulic supply. Maximum Operating Pressure of hydraulic supply is dictated by capacity of press. This connection set up allows the cell to be disconnected from the driver, which is necessary to install it on an instrument or in the situation where multiple cells are prepared in advance. The transducer between shut off and cell allows the operator to measure the pressure within the cylinder of the cell.

6.4. Declaration on the legal status (classification) of the device

Pipeline: The standard piping used in the MHS is SITEC brand ¼" HP tubing, rated for safe operation below 4000 bar (SITEC part no. 730.234). In addition, a 3-way valve SITEC part no. 710.534) is present. As the operating temperature of the liquid is < 65°C, the pipeline is unclassified (§11 AFS2017:3)

The PE-press: The press is a vessel containing hydraulic oil, which is a Group 2 liquid with a vapour pressure of less than 0.5 bar g. Correspondingly Article 4 (a) (ii), second indent of the PED applies, indicating the relevant table is Table 4, Annex II copied below.

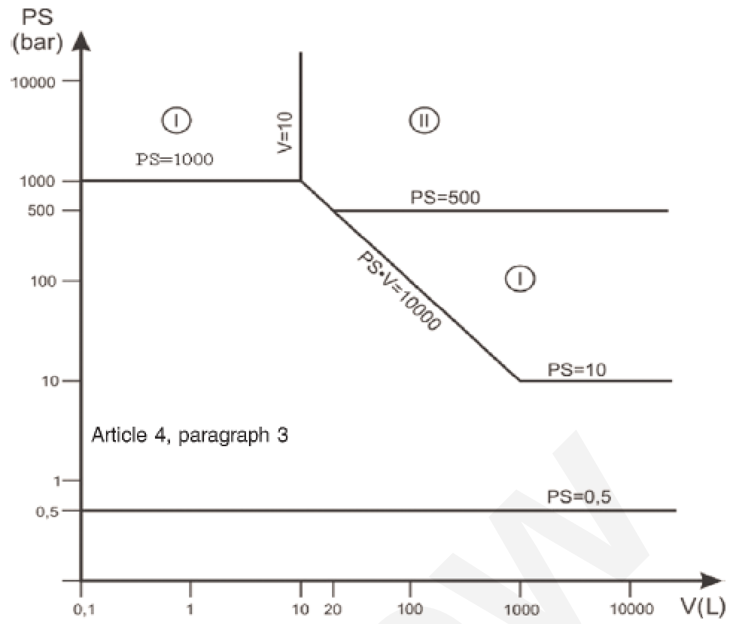


Table 4

Vessels referred to in Article 4(1)(a)(ii), second indent

Figure 9 Table 4, Annex II of the European Directive 2017/68/EU

The maximum working pressure of both PE presses is 2000 barg, however, the volume of 0.016 L is less than the minimum covered by the table. Correspondingly, Guideline A-05 Point 2 and, as working pressure is >1000 bar g, Point 3 applies. These state:

Point 2: “If a vessel has a volume less than or equal to 0,1L, and a value of PS above the limits defined in Article 4 paragraph 1, then the vessels must satisfy the essential safety requirements of Annex I.”

Point 3: “In the absence of specific information in the Tables of Annex II for the conformity assessment of vessels described in point 2 above, the manufacturer may choose any module, or single combination of modules, set out in Section 1 of Annex II.

According to Point 2, the Essential Safety Requirements of Annex I must be met. For Point 3, we propose the following: The closest category to our situation is (I) as per Table 4 above. Category (I) references Module A “Internal Production Control”, so this should be the module we choose.

6.5. A risk assessment for both maintenance and operation of equipment

Number	Activity or Event	Hazard	Who might be harmed and how?	Initial Risk Rating: Likelihood	Initial Risk Rating: Severity	Initial Risk Rating: Risk H,M,L	Actions to mitigate Risk - controls	Residual Risk Rating: Likelihood	Residual Risk Rating: Severity	Residual Risk Rating: Risk H,M,L	required training	additional reference
1	Heavy component falls of desk	foot/hand injury	Personnel setting up device could have foot or hand crushed by falling object	2	3	M	PPE: safety shoes	2	1	L		
2	Hands trapped between platten and cylinder	hand injury	If cell is supported by cylinder and anvil assembly is not present platten and tie-rods can slide down, trapping hands	3	3	M	1) Always use properly designed mounts 2) Ensure that all mounts used are designed to prevent this eventuality 3) Training to make operator aware of hazard	1	3	L	Instruction in PE operation	PE operating manual
3	Injury while lifting cell	back injury	Cell is very heavy (ca 70kg), lifting by hand is unsafe	3	3	M	1) always use mechanical lifting device 2) training to make operator aware of hazard	1	3	L	Instruction in PE operation crane training	PE operating manual
4	Blowout	injury from shrapnel (*nb. risk to be better quantified by planned tests in Utgård)	Blowout leads to high velocity shrapnel being ejected	3	4	H	1) always use appropriate shielding when a blowout risk is present. 2) follow appropriate procedures to minimise risk of blowout	3	1	L	Instruction in PE operation	PE operating manual
5	Blowout	hearing damage (*nb. risk to be better quantified by planned tests in Utgård)	Blowout results in loud bang	3	3	M	1) hearing protection 2) warn those nearby of risk	3	1	L	Instruction in PE operation	PE operating manual

6.6. A manual describing both maintenance and operation of equipment

Note 1: Some of the procedures and recommendations in this section may be modified in light of planned tests in Utgård (Q3-Q4/2019), where Burst Tests (artificially-induced blowouts of the gasket) will be conducted and the consequences accurately quantified.

Note 2: The present manual describes operation of the oil-driven V3 and VX6 PE presses at ambient conditions (~20° C) using ZTA standard single-profile anvils. The manual shall be extended to include alternative set-ups as and when these are developed.

Note 3: The manual has been written to cover usage of both PE cell types used at the ESS. However, there are some important differences between both devices and, where these affect usage, this has been noted explicitly in the manual. The main consideration is that the conversion from hydraulic pressure to force in tonnes is different for both presses (due to the different diameters of the pistons):

- ***For the V4: 10 bar = 1 met. tonne***
- ***For the VX6: 15 bar = 1 met. tonne***

6.6.1. Preparing the PE press for loading

- Prior to any work, he PE press should be mounted securely in an appropriate support that provides space for hydraulic connection and allows for the cell axis to be vertical, with the piston on the lower side.
- When the breech is removed from the platen, it should be placed vertically on the desk, anvil side up (so it doesn't roll off).
- All hydraulic connections to drivers should have been made tight and a visual inspection should be conducted of connections within the cell piping system.
- It *must* be ensured that the piston is driven fully back. This is done by using the provided blank between the breech and piston and tightening the breech nut to drive back the piston. Any oil within the cylinder will be pushed out through the hydraulic connection, so the lines should be fully opened towards a 1 bara reservoir.
- The cell should then be Leak Tested. It is recommended to do this by installing the testing blank and applying a pressure of at least 500 bar g (after a successful initial

Leak Test §2.3). This should then be monitored for at least 1 hour to observe any potential loss of pressure that would indicate a leak.

- The press can then be assembled by installing, in order from outside to centre: backing discs, seats and anvils on both piston and breech sides of the press. Appropriate centring rings should be used and *set-screws used to hold anvil in place on the breech should be tightened sufficiently so that the anvils don't fall out*. (Important note: the VX6 uses ceramic backing discs and the VX4 uses steel discs. Also, although the anvils are interchangeable between cells, the aluminium centring rings and seats differ in dimensions. Observe marking on the rings to ensure the correct ones are used).

6.6.2. Loading the sample into the PE press

- The gasket is then put in place and the sample loaded. In some cases, it may be easier to remove the piston anvil and position the gasket and sample before lifting into place in the cell. For encapsulating gaskets, the lower part should be installed prior to inserting the sample and the upper part of the gasket should be installed after the sample is in place.
- The breech is then inserted and screwed down until contact is made with the sample. The breech should be made tight using an appropriately sized spanner.
- The safety shield shall now be installed.
- If the sample contains a liquid, a sealing load of ~7 tonnes will form a positive seal preventing leakage. (Important note: for the V4 this corresponds to 70 bar hydraulic pressure, while for the VX6, this corresponds to 105 bar).
- For certain samples, for example volatile or reactive materials, the above procedure may need to be done quickly. In this case, at least two people (A &B) shall be present and the following procedure followed:
  - A: inserts sample into cell and places top of encapsulating gasket, if it is being used.
  - B: tightens breech down onto cell and applies shield
  - A: applies sealing load to the cell.

6.6.3. Using the PE press with ZTA anvils

After preparation, the sample can now be pressurised by increasing the hydraulic load. It is important to do this at a controlled pace. The pace depends on sample type, tonnage and anvil type. Currently, this manual covers operations with liquid or solid samples[1], in ZTA single-toroid anvils. Loads and rates are specified in tonnes, this will correspond to a different hydraulic load depending on cell type.

[1] The loading of gaseous samples at the ESS will be addressed in the future.

At all times when the cell is being actively pressurised, some form of physical shielding must be in place. Physical shielding should be installed at all times when the load is above 10 tonnes *(see § 6.6.7)*

Low-pressure operation: up to 10 tonnes, pressure should normally be increased at a rate of ~0.3 tonnes/s (18 tonnes/min). Shielding must be in place during pressure change. If the pressure has been held stable for at least 10 minutes, it is permissible to remove the shielding (this is occasionally necessary, for example, to adjust collimation), however, PPE including safety glasses and Kevlar gloves should be worn.

Medium-pressure operation: 10 to 60 tonnes. Physical shielding should be permanently in place. Pressure shall be increased at a moderate rate of ~1 tonne/min and, in addition, for every 5 tonnes, pressure shall be kept static for a period of at least 5 minutes. Likelihood of a blowout (see §6.6.6) is low.

High-pressure operation: 60-80 tonnes. Pressure should be increased at a slow rate of ~0.5 tonnes/min and, in addition, for every 0.5 tonnes, pressure should be kept static for a period of at least 5 minutes. In this pressure range, blowout has a medium probability.

Conversion Table

Load or load rate	**Hydraulic pressure V4**	**Hydraulic pressure VX6**
10 tonnes	100 bar	150
60 tonnes	600 bar	900
80 tonnes	800 bar	1200
0.3 tonnes/s	3 bar/s	4.5 bar/s
1 tonne/min	10 bar/min	15 bar/min
0.5 tonnes/min	5 bar/min	7.5 bar/min

6.6.4. Cryogenic loadings

In circumstances where samples decompose at room temperatures, it is sometimes necessary to load these at cryogenic temperature (usually under liquid N_2) and then rapidly pressurise in order to achieve stable pressure conditions before the sample degrades. In this case, faster rates up to ~1 tonne/s can be employed. Although blowout risk is low, some form of shielding must always be employed. After sealing, the sample and anvil assembly shall be allowed to warm to room temperature before further increasing pressure. Throughout warming, shielding must stay in place.

6.6.5. Downloading the PE press with ZTA anvils

The risk of a blowout is highest during download and most often occurs in the final stages of decompression

At all times when the cell is being downloaded, some form of physical shielding must be in place.

In order to minimise the risk of a blowout occurring, the entire download should be conducted at a very slow rate ~ 0.02 tonnes/s

6.6.6. Blowouts

With the PE-press, a "blowout" refers to an event where either the gasket or anvil fails suddenly under the internal pressure of the sample. In this case, often, small fragments of the metal gasket can be violently ejected from the anvil assembly resulting in a risk of physical injury. In addition, the anvils then hit together with significant force, resulting in a loud banging sound and possible ejection of anvil fragments.

Blowouts are, by nature, probabilistic phenomena, but some 30 years of experience using the devices have quantified the level of risk in different scenarios. Moreover, the key risk factors are well understood and include: the physical form of the sample (e.g. solid, stable liquid, unstable liquid[2] or gas), the load on the anvils and the material of the gasket and anvil material.

These risks are quantified here for the various factors in Table 7. At present, only ZTA anvils with either TiZr or steel gaskets are considered as these are the only ones currently used at ESS. Table 7 will be continually updated as new anvil and gasket materials are employed

Table 7 Risk factors when operating the PE press

Anvil/gasket material	Sample state (@ambient P & T)	Load (tonnes)	Likelihood* (1-4)	Severity** (1-5)	Risk
ZTA/(TiZr or steel)	solid	0-10	1	4	Low
		10-60	2	4	Medium
		> 60	4	4	High

[2] Primarily any liquid whose vapour pressure at ambient temperature is less than 0.5 bar g (i.e. the liquid will itself pressurize the sample chamber)

	Stable liquid	0-10	1	**4**	**Low**
		10-60	2	**4**	**Medium**
		> 60	4	**4**	**High**
	Unstable liquid	0-60	2	**4**	**Medium**
		> 60	4	**4**	**High**

In all cases where the risk of a blowout is medium or higher, physical shielding must be in place. Where the risk is low, it is possible to remove the shielding if the hydraulic pressure has been constant for at least 10 minutes and appropriate PPE is used.

6.6.7. Shielding

Due to the intrinsic risk of blowouts when using the PE press, some form of shielding is considered an essential part of their operation.

In normal cases, this shielding will comprise physical barrier of sufficient strength to stop any shrapnel that may be released in the event of a blowout, thus negating the risk of physical injury. All possible lines-of-sight from sample out of the cell must be covered by the shield, as this will correspond with paths for the incident and scattered neutron beams, then a low attenuating material, such as Al, will likely be used during neutron operation (an alternative material for offline testing is polycarbonate, which has the advantage of being transparent). In addition to specially-made “blast shields”, ancillary sample environments e.g. vacuum vessels for cryostats may provide sufficient protection. In any case, calculations and measurements must be provided to demonstrate that the shielding has adequate strength (this is particularly important where the wall of an evacuated vessel acts as shielding as perforation of the wall will result in an implosion).

In extraordinary cases, it may be necessary to directly access the sample while there is a medium risk of a blowout. In this case, the shielding will take the form of PPE such as Kevlar gloves and face shield and should only be attempted by trained personnel. This is never permitted when the risk of blowout is high.

6.7. Where appropriate a maintenance schedule for the device

The seals on the cell shall be replaced every 12 months or 50 cycles or if any leak is observed.

The cell shall be inspected for corrosion and this should be removed with a fine abrasive.

6.8. Record-keeping requirements

It is extremely helpful to keep a record of usage of the press. Each time it is used, anvil material/profile, gasket material and maximum pressure should be recorded. If a blowout occurs, this should be adequately documented and the anvils/gasket retained for future inspection.

In addition, a maintenance record shall be kept.

6.9. Required training for operators

Users of the PE presses must have received specific instruction from a trained operator of the PE press.

7. SEE: DIAMOND ANVIL CELLS

7.1. Summary of purpose of equipment

Diamond anvil cells (DACs) are a form of opposed-anvil device, based on a mechanism that applies a force (generated either mechanically, hydraulically or otherwise) that drives together two flat-tipped (or bevel-tipped) diamonds. Experimental samples are contained between the two tips, called culets, and a surrounding gasket, usually made from various metals. The DAC's used for neutron activitiers can generate extreme pressures, exceeding 1,000,000 bar, however, sample sizes are microscopic (typically not exceeding 0.00005 cc).

DACs are envisaged to have a role in multiple early science scenarios at the ESS. Primarily, these will be used for diffraction experiments, but some spectroscopy and SANS are also possible.

The current generation of neutron DACs are a relatively new technology. Developments began around 2011 as a collaboration between The Geophysical Laboratory, Washington, DC, and the SNS facility in Oak Ridge, TN and are ongoing.

7.2. Description of the components

At the time of writing, the neutron DAC exists in several distinct variants. The ESS collaborates with the SNS and benefits heavily from their continued development of the devices. The ESS currently owns two DAC variants: UoE-type (University of Edinburgh) and ESS-type. Both are based on the original GL-SNS design and operate with the same principles:

- A piston-cylinder arrangement is used to guide two opposing diamonds together under force
- diamonds are typically 4-6mm in diameter and have a double-conical geometry supported by so-called Boehler-Almax seats which have a mating conical bore that the diamonds are held in.
- a metal gasket formed from a sheet of metal, held between the diamonds, with a small hole in which the sample, pressure media and any pressure calibrants can be placed.
- A bolt-driven mechanism is present allowing the application of small forces (< 20kN) to drive the diamonds together. These also allow the possibility of locking in this force enabling the cell to be transported between facilities under load.
- An additional 'gas-driven, double-membrane' driving unit is a closed system that can convert applied gas pressure to mechanical force driving the anvils together.
- A series of apertures in both the piston and cylinder parts provides access for incident and diffracted neutrons.

7.2.1. The sample chamber, gasket and anvils

The sample in a DAC is contained on two sides by the flat tips of the diamonds (called culets), which are concentric and parallel, and a gasket. Standard gaskets are normally made of a small section of thin metal sheet with a thickness of ~400 µm. It is first "pre-indented" by placing it between the diamond anvils and applying force sufficient to induce plastic deformation. The final thickness of the material between the anvil tips chosen according to variables such as the maximum pressure desired and culet diameter.

Subsequently, the gasket is removed and a bore hole is drilled in the centre of the indent with a diameter that also depends on maximum required pressure and culet diameter. The gasket is then re-positioned on one of the anvils to form the sample chamber, which is then filled with sample (and pressure medium etc). Lastly, the second diamond is lowered onto the other side of the gasket and force applied to further deform the gasket to make a seal.

The precise pressure generated within the sample depends on applied force, culet diameter, anvil geometry and gasket material. However, a representative guide is given in Table 8 for several realistic culet diameters.

It is useful to note that the sample volume is microscopic and only less than 0.1% of the gasket volume in all cases.

Table 8 Representative pressures and volumes for samples in DAC cells

Culet rad. (cm)	Culet area (cm^2)	sample rad. (cm)	sample area (cm^2)	sample thick. (cm)	sample vol (cm^3)	sample vol (L)	Approx. P at 6 tonnes (GPa)
0.0800	0.0201	0.0320	0.0032	0.0160	5.15E-05	5.15E-08	29.8
0.1000	0.0314	0.0400	0.0050	0.0200	1.01E-04	1.01E-07	19.1
0.15	0.0707	0.0600	0.0113	0.0300	3.39E-04	3.39E-07	8.5

7.2.2. Gas-driven double-membrane Drivers

Both DAC designs incorporate gas-driven, double-membrane (DM) drivers in their design. This device is formed from two circular, thin and flexible sheets of steel, welded around their edges so that pressure gas pressure can be held between them. A braised $\frac{1}{16}$" pressure tube allows gas pressure to be introduced into the gap between the membranes. Both DAC types at ESS use membranes that have have a central hole (also

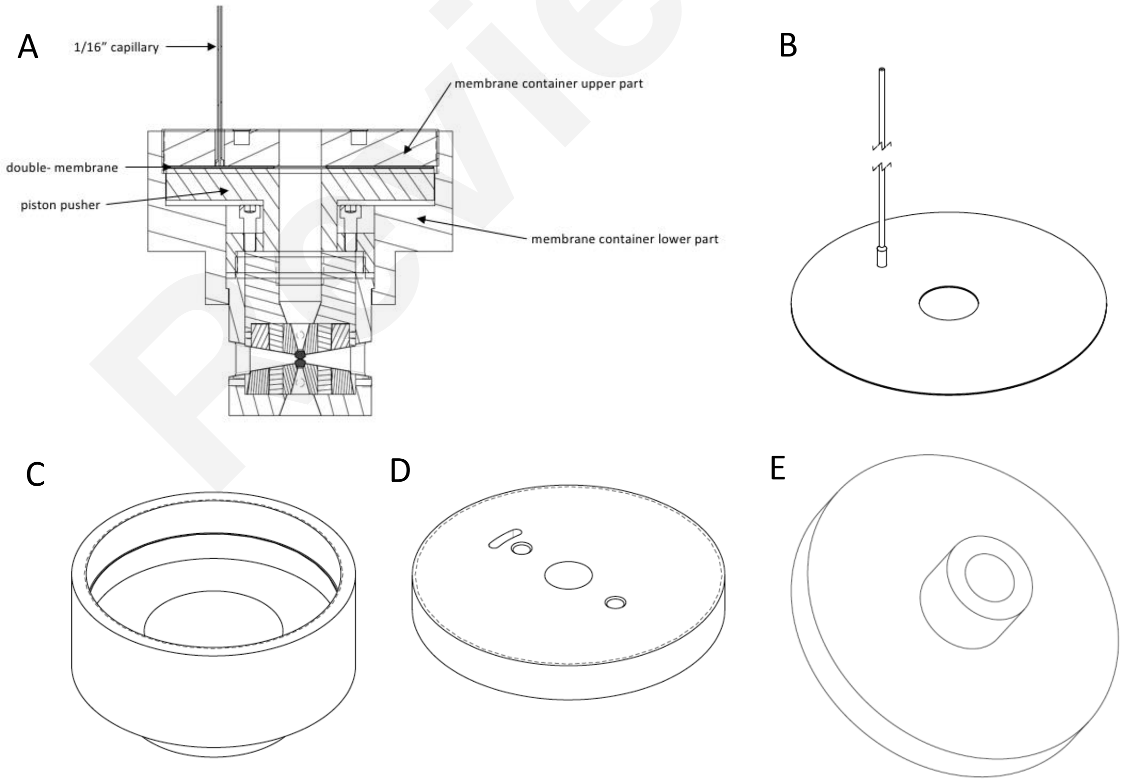


Figure 10 (A) Double-membrane driver as assembled with a UoE DAC (see §7.2.3). (B) the double-membrane with attached 1/16" stainless steel pressure tubing. (C) The lower part of the membrane enclosure (D) the upper part of the membrane enclosure (E) The membrane pusher, which transmits the force generated by the membrane to the back of piston and, ultimately, pushing the diamonds together.

welded), which provides both optical and neutron access through the membrane (Figure 10B).

The DM is fully contained within an assembly of three main parts (see Figure 10) an enclosure (which has upper and lower parts, which are screwed together) and pusher. Thus, when gas pressure is introduced, a force is generated on both the enclosure and the pusher. The enclosure is fixed relative to the cylinder of the cell, while both pusher and cell piston are free to move relative to both enclosure and cylinder. Thus, the force generated by the gas acts to push the piston into the cylinder, thus pressurising the diamonds and sample. Meanwhile, this force is balanced in the opposite direction by tension in the lower membrane enclosure walls. A secondary function of the enclosure is to provide safety should the DM rupture; as such, the DM should ***never*** be operated without the enclosure in place and correctly assembled.

An important feature of the DM operation is that, by design, it has extremely limited stroke, of order 1-2 mm. It is thus critical to ensure that the assembly is fully tightened before introducing gas pressure as this would waste stroke and potentially rupture the DM. If correctly assembled, the only travel required is that required to

1) deform the gasket of the cell, which cannot exceed the 0.1 mm thickness of the gasket indent.
2) Accommodate the combined extension of the cylinder body and membrane enclosure body, estimated to be ~1 mm.

The short stroke of the DM also limits its maximum volume as shown in Table 9.

When gas pressure is introduced into the DM, the resulting force depends on the area of the DM that is in contact with the enclosure and pusher. The ESS-type membrane is smaller than the UoE membrane and therefore applies a smaller force for a given pressure (see Table 9).

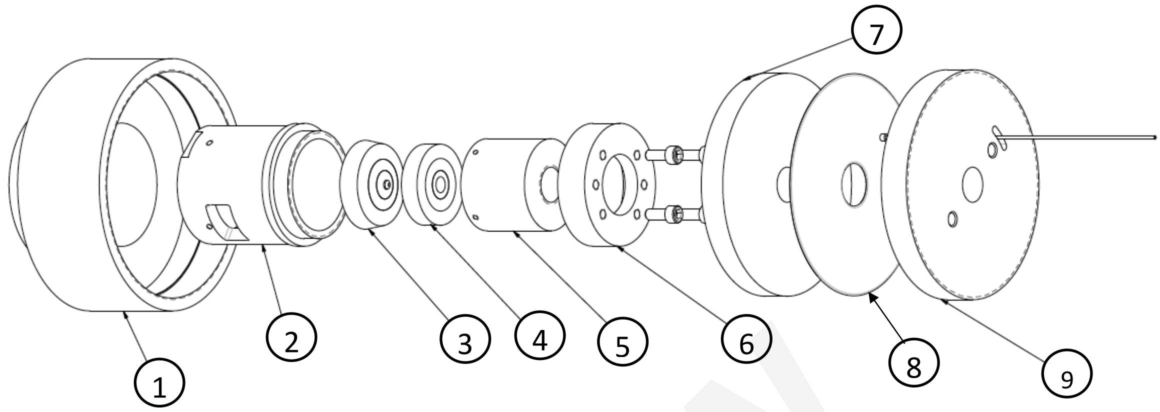
Table 9 Pressures and volumes of both DM types in use at ESS

DAC type	DM outer rad.	DM inner rad.	DM area	max. height	DM vol	DM vol	MOP	Approx. Force at MOP*
	(cm)	(cm)	(cm^2)	(cm)	(cm^3)	(L)	(bar)	(kN)
UoE	5.080	0.953	78.223	0.200	15.645	0.016	150	117.3
ESS	4	0.9	47.721	0.200	9.544	0.010	150	71.6

(* note: actual force will be less than this due to friction in the system. Ongoing tests with load gauges will quantify this better in the future).

7.2.3. The UoE DAC

The UoE DAC was fabricated by the University of Edinburgh but has an identical design to the first-generation GL-SNS design {Boehler, 2013 #103}. The UoE DACs have Inconel bodies to enable compatibility with hydrogen samples. The outer diameter of the cells is compatible with GL-type gas loader available at the University of Edinburgh, enable high-



pressure loading of gaseous media.

The press includes a piston situated within a cylinder, the piston being driven forward in the cylinder by means of hydraulic fluid (typically oil, but fluid pentane and He gas are also used) with pressure maintained by a seal appropriate for the fluid used (e.g. for hydraulic oil a simple o-ring is used). The force generated by the hydraulic oil is determined by the product of the area of the rear of the piston and the oil pressure, thus,

The designed load capacity of the UoE DAC is rated to 10 tonnes.

Figure 11 Exploded view of GL-SNS design. Labelled parts are: (1) Membrane enclosure, lower part (2) Cylinder (3) Cylinder seat and anvil (4) Piston seat and anvil (5) Piston (6) cell cap (7) Pusher (8) double-membrane (9) membrane enclosure, upper part.

The ESS DAC is a modified version of the GL-SNS design, that is optimised for low-temperature operation and features larger diffraction apertures. The cell is manufactured from CuBe C17200 material and FE analysis was used to minimise its mass. In addition to being made from, a different material, the ESS DAC is significantly smaller than the UoE variant.

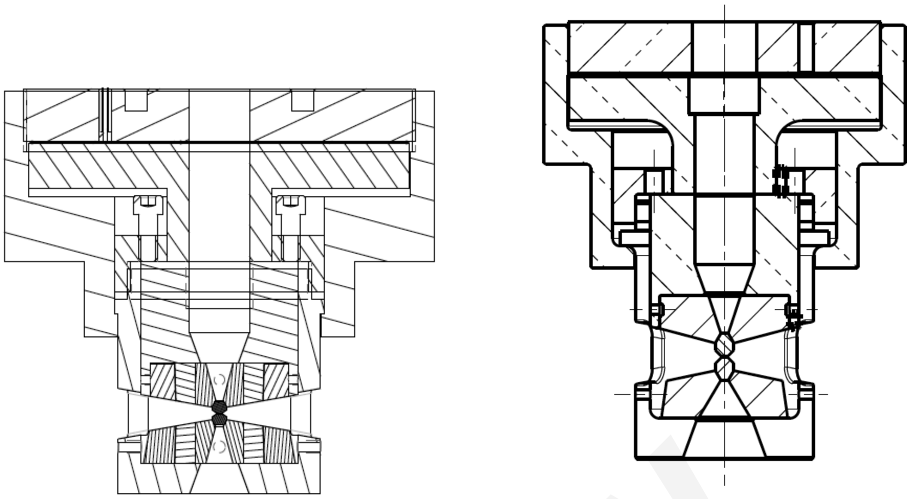


Figure 12 Two designs of DAC used at the ESS showing the UoE DAC on the left and the ESS DAC on the right. Drawings are at a scale of approximately 1:2. Largest diameter of ESS DAC

7.2.5. ESS DAC cell inventory

Error! Reference source not found. summarises the current inventory of DAC-type cells n use at the ESS.

DAC Model	MOP of gas in DM (bar)	Max. tonnage (tonnes)	Number of devices	Device Status
UoE DAC	150	10	5	Owned by ESS.
ESS DAC	150	10	1	Owned by ESS.

7.3. Piping and Instrumentation diagram (P&ID)

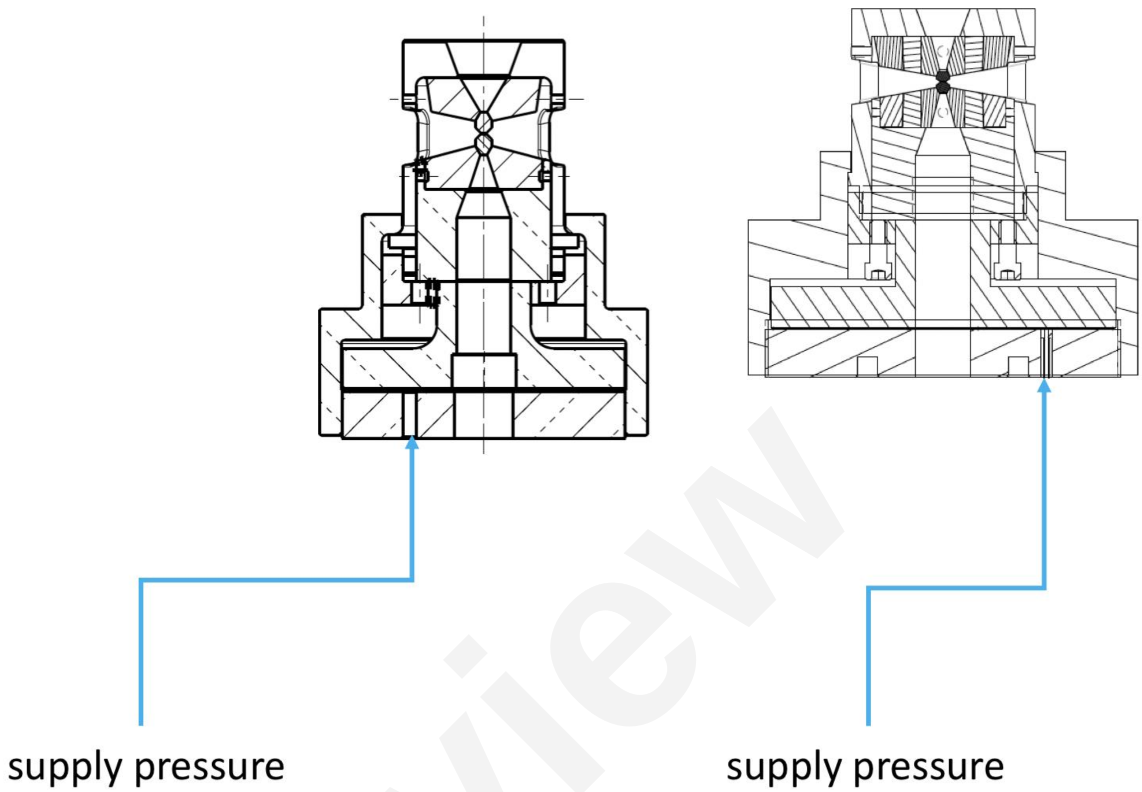


Figure 13 Diagram shows the gas connections to the DAC cells. Blue lines indicate gas supply.

7.4. Declaration on the legal status (classification) of the device

Pipeline: The standard piping used ito connect to the DM is Swagelok 1/16" OD x 0.014" wall thickness stainless tubing (part No. SS-T1-S-014-6ME), which has an allowable working pressure of 8100 psig (558 barg). For convenience, the length of the piping is chosen to be as short as possible 100-150 mm being typical. The nominal diameter DN for 1/16th tubing is not defined, so accordingly (as stated in §11 AFS2017:3), the ID of 0.87 mm is used, rounded up to DN = 1.

The medium will be an inert gas (either He or dry N2, depending on temperature of operation), which are group 2a fluids according to (§3 AFS2017:3). The product of DN and max pressure is thus 150 bar so, according to §11 AFS2017 the pipeline is unclassified.

Double membrane: The DM is an unheated pressure vessel containing a type 2a gas. Classification is, therefore defined in (§10 of AFS2017:3) on the basis of the product of its volume (L) and operating pressure (bar). According to Table 9 this number is 2.4 bar.L for the UoE DAC and 1.5 bar.L for the ESS DAC. As the lowest value of described in the relevant table of §10 of AFS2017:3 is 50 bar.L, it is determined that the DM is unclassified.

The EU pressure directive 2014-68-EU also imposes requirements on pressure equipment according to a classification scheme. According to the 2014-68-EU, Article 4 (a)(i), first indent, indicating the relevant classification is given in Annex II, table of 2014-68-EU,which is copied below:

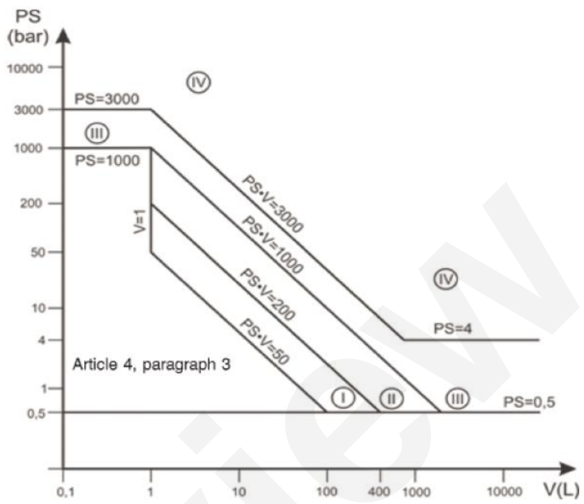


Table 2

Vessels referred to in Article 4(1)(a)(i), second indent

Figure 14 Table 2, Annex II of the European Directive 2017/68/EU

The volumes of the UoE and ESS DM's, 0.016 and 0.010 L respectively, both fall below the minimum specification in the table. At the minimum specified volume of 0.1L, and the operating pressure of 150 bar, the table denotes that Article 4, paragraph 3 (S

The maximum working pressure is 150 barg, however, the volumes of 0.016 L and 0.010 L for the UoE and ESS DM's respectively are both less than the minimum covered by the Table. Correspondingly, Guideline A-05 Point 2 was consulted, which states Article 4.3 (Sound Engineering Practice) applies (as PS <= 1000 bar), so neither membrane is classified according to 2014-68-EU.

7.5. A risk assessment for both maintenance and operation of equipment

SYSTEM: DIAMOND ANVIL CELLS												
				Initial Risk Rating				Residual Risk Rating				
Number	Activity or Event	Hazard	Who might be harmed and how?	Likelihood	Severity	Risk H,M,L	Actions to mitigate Risk - controls	Likelihood	Severity	Risk H,M,L	required training	additional reference
1	Heavy component falls of desk	foot/hand injury	Personnel setting up device could have foot or hand crushed by falling object	2	3	M	Utilise a stand to prevent cell from rolling	2	1	L		
2	Failure of membrane	injury from shrapnel	Personnel adjacent to DAC at point of failure	1	4	L	1) Ensure correct operation of DAC 2) Have designed enclosure to completely mitigate risk 3) Training to make operator aware of hazard	1	1	L	Instruction in DAC operation	DAC operating manual
3	Failure of membrane	hearing injury (* not sure if this is a real hazard. Planned tests in Utgård to quantify risk)	Failure of membrane make create a loud noise	1	3	L	1) Ensure correct operation of DAC 2) Training to make operator aware of hazard	1	3	L	Instruction in DAC operation	DAC operating manual
4	Exposure to BeCu dust particles	lung cancer, berylliosis	personnel exposed to dust particles	2	5	M	1) Ensure no abrasive is used on ESS-DAC body. Use appropriate environmental controls (e.g. fume hood) and PPE (e.g. repiratory mask) if engaging in any activity that might produce dust	1	4	L	BeCu training (if/when it exists). Instruction in DAC operation	DAC operating manual

7.6. A manual describing both maintenance and operation of equipment

Loading and operating DAC cells is a special activity because of the delicate and expensive nature of the equipment. In addition, each design of DAC has its own considerations, so experience of one type of DAC does not ensure competence with other designs. Correspondingly, this manual should not be considered a replacement for one-on-one instruction by an expert user, who is familiar with the operation of the devices.

7.6.1. Preparing the gasket for loading.

- The first step is to consider what material to use for the gasket. Issues such as maximum pressure needed, temperature and possible chemical reaction of the sample are part of this assessment.
- If gas loading is required (note this is currently not available at ESS), then the gasket must be carefully polished and cleaned to ensure a seal. Most gas-loading devices have insufficient force to plastically deform the gasket with large neutron-size culets. Therefore, small scratches and pieces of dirt will result in a leak.
- As a next step, the alignment of the diamonds must be confirmed: both culets must be exactly concentric and parallel to each other. Interference fringes can be observed under a microscope to assess parallelism, with a goal of having only 1—2 fringes across the entire culet surface.
- Prior to inserting the gasket, an alignment mark should be made (a carbide scribe works well for this). For gas loading, it must be ensured that the mark does not overlap with the culet.
- Both gasket and diamond culets should be cleaned at this point. And this should be continued repeatably, throughout the loading.
- The gasket is then placed on one of the diamond culets and aligned to be perfectly flat and concentric with it. Use of some wax is helpful to maintain this alignment. A mark should be made on the seat opposite the score on the gasket. This ensures that the gasket can be repeatably inserted in the same orientation relative to the anvils.
- The gasket is then indented to an appropriate depth by applying force on the cell. The approach at ESS is to use hydraulic press and a load gauge to repeatably apply load. As, for a given force, the resulting indent depth depends both on culet size and gasket material, several runs may be needed do determine the appropriate force for a given set-up.
- The indent thickness is chosen by taking account of the required maximum pressure, culet size and compressibility of the sample. Typical values are 100-190 µm.
- After indenting, the gasket must be removed from the cell and the sample hole drilled. At the ESS, this is done using electro-discharge machining (EDM) with carbide rod bits of appropriate diameter. As a rule of thumb, gasket holes should be ~ 40% of the culet diameter. However, for highly compressible samples, such as pure hydrogen, much larger holes will be needed ~ 60-65% of culet size.

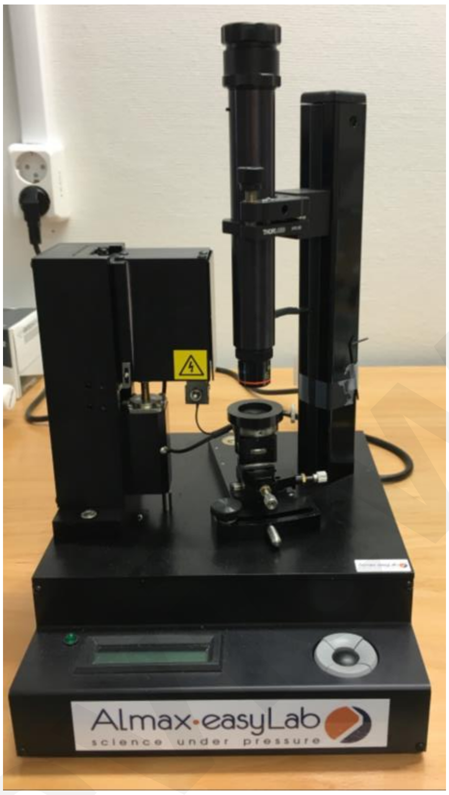


Figure 15 EDM gasket drilling device

Figure 16 DAC gaskets. On left is a new un-indented gasket, which has a diameter of 6mm and thickness of 400 µm. On the right is an indented and drilled gasket. The alignment mark is seen at approximately 1 o'clock. Not that, in addition to the indentation, the gasket is seen to cup downwards.

- After thoroughly cleaning the gasket (e.g. using an ultrasonic bath), the gasket should be re-introduced onto the diamond. Typically, the indent itself will help ensure that the gasket correctly re-centres on the diamond. However, it can still rotate relative to the culet, so the alignment mark should be used to ensure orientational repeatability.
- As a final step, the DAC should be fully reassembled and a small load applied by hand to 'bed down' the gasket. It is helpful at this stage to re-apply wax to hold the gasket in place.

7.6.2. Loading the sample

- After the preparations described in §7.6.1, the DAC is ready for loading the sample. Depending on the nature of the sample (solid, liquid or gas), the exact details of this will vary.
- It is also best practice to include a pressure calibrant in the sample chamber at this point. Typically, a small chip of ruby can be used, as the laser-induced fluorescence of ruby has been calibrated with respect to pressure. Diffraction standards can also be useful for *in situ* pressure determination.
- In addition to the sample and pressure calibrant, pressure media should also be used to ensure hydrostatic pressure generation.
- After sample, calibrant and pressure media are in the sample chamber, the cell should be closed and sealed, normally by applying initial load using the screws on the cell cap. This load should be sufficient to plastically deform the gasket, thus making a seal.
- At this point the cell should be stable indefinitely and can be easily transported between facilities.

7.6.3. Adding the DM driver

Whereas with more conventional x-ray DACs, the cap screws could be used to further increase pressure, however, the large culet sizes used for neutrons mean that the some mechanical, hydraulic or other device must be used to generate sufficient force. At the ESS the gas-driven DM drivers are used. These are assembled by:

- Dropping the DAC into the lower part of the DM enclosure, adding the pusher and DM and then screwing the upper part of the enclosure down trapping the DM between pusher and enclosure. Prior to this, both pusher and upper enclosure should be cleaned to be free of dust, which will otherwise indent into the DM.
- It must be ensured that the DM enclosure is fully tightened and a tool is available to do this. It is especially important to ensure that no torque is placed on the 1/16" tubing while tightening the upper enclosure.

- Note, tightly closing the DM will likely also increase the sample pressure, which might be an important consideration in certain circumstances. If a ruby pressure calibrant is in place, any increase in sample pressure can be determined.
- At this point, the initial locking screws in the cell cap will be disengaged, and the entire force on the gasket is being delivered by the DM enclosure.

7.6.4. Changing pressure with the DM driver

- Once correctly assembled, positive gas pressure in the DM will increase force on the anvils, leading to pressure generation on the sample.
- The 1/16” capillary on the DM should be connected to a gas source (e.g. a gas cylinder via a regulator or the PACE gas driver). If cryogenic operation is envisaged, the tubing should then be evacuated to ensure no moisture is present in the line.
- As described in §7.2.2, the force generated will depend on membrane size. Meanwhile, sample pressure will depend on both the load generated and the details of culet, gasket and sample. Normally sample pressure *vs* load curves will be available, but these should be considered guides only as each loading will be different.
- When increasing pressure, the gasket hole should be continually observed. If any enlargement or deviation from roundness of the gasket hole is seen, or if the hole moves off the centre of the culet, then the loading should be terminated to ensure safety of the diamond anvils.
- In addition, to observing the gasket, the sample pressure *vs* load should be continually monitored. In normal operations, the dependence should be almost perfectly linear. If this curve is observed to plateau or if its slope changes, this can indicate problems such as mechanical failure of cell components, gasket failure, or blockage in the capillary. Further pressure increase should be postponed until an explanation is found.
- When decreasing gas pressure, a significant hysteresis in sample pressure should be expected due to friction in the system. This often means that even after the DM is fully downloaded, significant pressures (in excess of 10 GPa) may be present within the sample. Due to the microscopic sample size, there is no hazard from this pressure. However, this may be an important consideration for the planned experiment.

7.7. Where appropriate a maintenance schedule for the device

The cell should be inspected for corrosion and this should be with a fine abrasive removed. Abrasive *should not* be used for the ESS DAC due as it is made from BeCu.

7.8. Record-keeping requirements

It is extremely helpful to keep a record of usage of the press. Each time it is used, anvil material/profile, gasket material and maximum pressure should be recorded.

7.9. Required training for operators

Users of the PE presses must complete all required ESS training for the following activities.

- High-pressure opposed anvil cells

Additionally, they must receive specific instruction from a trained operator of the ESS and UoE DACs.

8. GLOSSARY

Term	Definition
PE	Paris-Edinburgh
DAC	Diamond anvil cell
PPD	Portable Pressure Driver
BTD	Burst Test Documentation
PTC	Pressure Testing Checklist
RA	Risk Assessment
MOP	Maximum Operating Pressure
(U)PTF	(Utgård) Pressure Testing Facility
PSDD	Primary Shielding Design Document
SEE	Sample Environment Equipment
SES	Sample Environment System
MHS	Maximator Hydraulic System
PACE	A gas pressure controlling system

9. REFERENCES

[1] <<Sample reference to CHESS document: ESS Document (ESS-00XXXXX)>>
[2]

10. DOCUMENT REVISION HISTORY

Revision	Reason for and description of change	Author	Date
1	First issue <<Keep only full number revisions when approving document>>	<<Malcolm Guthrie>>	<<2019-07-xx>>

11. APPENDICES

Appendix A: Risk Assessments

Portable pressure driver Risk Assessment (§)

SYSTEM: PORTABLE PRESSURE DRIVER				Initial Risk Rating				Residual Risk Rating				
Number	Activity or Event	Hazard	Who might be harmed and how?	Likelihood	Severity	Risk H,M,L	Actions to mitigate Risk - controls	Likelihood	Severity	Risk H,M,L	required training	additional reference
1	SEE falls off PPD or causes it to topple	physical injury	operator, heavy SEE fall causing an injury. Or, entire cart topple due to top heavy SEE.	2	3	M	1) Operator must bolt SEE onto MHS 2) Weight limit displayed on MHS must be respected	1	2	L	Instruction in use of PPD	PPD ops manual
2	Lose control of PPD on slope	physical injury, damage to equipment	passersby collision with PPD	2	3	M	1) Define safe routes avoiding slopes 2) Train operators to be aware of hazard	1	3	L	Instruction in use of PPD	PPD ops manual
3	Tripping on utility connections	tripping/fall	passersby or operator	4	1	M	1) follow manual to safely make utility connections	3	1	L	Instruction in use of PPD	PPD ops manual
4	Short circuit resulting in electrification of metal frame	electrical hazard	operator	2	5	M	1) Ensure trolley is earthed whenever energised. 2) Electrical installation completed by qualified person	1	5	L		

Maximator Hydraulic System (§)

System: Maximator Hydraulic System				Initial Risk Rating				Residual Risk Rating				
Number	Activity or Event	Hazard	Who might be harmed and how?	Likelihood	Severity	Risk H,M,L	Actions to mitigate Risk - controls	Likelihood	Severity	Risk H,M,L	required training	additional reference
1	slow oil leak leads to oil on floor	slipping hazard	passersby or operator could slip	4	3	M	1)Design of PPD includes drip tray to catch spills 2) Routine maintenance includes leak test 3) Anti-vibration fittings in MHS prevent loosening of connection in use	1	3	L		MHS ops manual
2	Leak from low P input (high pressure air (<= 6 bar)	aeroembolism	operator could suffer aeroembolism	2	5	M	1) Build guard around air connection to prevent air contacting skin 2) Follow procedure to leak test connection at low pressure before increasing pressure	1	1	L	"Use and Inspection of Pressurised devices (AFS2017:3)"	MHS ops manual
3	Incorrectly made output connection (oil pressure)	slipping injury	passersby or operator could slip	4	3	M	1) After making connection, always perform a quick leak check at low pressure 2) Monitor pressure throughout use to detect if a leak occurs	2	1	L	Instruction in use of MHS	MHS ops manual
4	failure of internal high-pressure hydraulic components	physical Injury from projected oil	operator could be injured by high-pressure oil	1	5	M	Build secondary shielding around MHS	1	1	L		
5	unexpected pressure generation during set up	pressurised oil	operator could be injured by high-pressure oil	3	4	M	1) when setting up MHS, vent valve (V2 or V3) **must** be open 2) when setting up MHS, input air valve V1 **must** be closed	2	4		Instruction in use of MHS	MHS ops manual Maximator manual
6	Exceding maximum input pressure	physical Injury from projected oil	operator could be injured by high-pressure oil	1	5	M	1) Build secondary shielding around MHS 2) ensure supply pressure is limited to 6 bar	1	1	L	Instruction in use of MHS	MHS ops manual
7	Impact from heavy pressure tubing	physical injury from impact	operator could be hit by heavy tubing when a difference in floor level exists between MHS and connected SEE	2	2	L	1) training so operator is aware of risk	2	2	L	Instruction in use of MHS	MHS ops manual

GE PACE PRESSURE CONTROLLER (§)

SYSTEM: GE PACE PRESSURE CONTROLLER				Initial Risk Rating				Residual Risk Rating				
Number	Activity or Event	Hazard	Who might be harmed and how?	Likelihood	Severity	Risk H,M,L	Actions to mitigate Risk - controls	Likelihood	Severity	Risk H,M,L	required training	additional reference
1	gas leak during connection, operations or disconnection to PACE	High pressure gas	operator could be harmed by physical injury (whipping pressure line)	2	4	M	1) Training for use of compressed gas 2) Training in use of PACE	1	4	L	Instruction on use of PACE	PACE ops manual
2	gas leak during connection, operations or disconnection to PACE	High pressure gas	Operator could be harmed by aeroembolism	2	5	M	1) Training for use of compressed gas 2) Training in use of PACE 3) wear appropriate gloves, when making connections	1	5	M	"Use and inspection of pressurised (AFS 2017:3)" Instruction on use of PACE	PACE ops manual
3	Incorrect set-point creating dangerous pressure conditions	explosion	Operator and passersby could be physically injured	3	4	M	1) software limits preventing unsafe pressure request 2) pressure relief attached to connected SEE	1	4	L	"Use and inspection of pressurised (AFS 2017:3)" Instruction on use of PACE	PACE ops manual

PARIS-EDINBURGH PRESSES (§)

				Initial Risk Rating				Residual Risk Rating				
Number	Activity or Event	Hazard	Who might be harmed and how?	Likelihood	Severity	Risk H,M,L	Actions to mitigate Risk - controls	Likelihood	Severity	Risk H,M,L	required training	additional reference
1	Heavy component falls of desk	foot/hand injury	Personnel setting up device could have foot or hand crushed by falling object	2	3	M	PPE: safety shoes	2	1	L		
2	Hands trapped between platten and cylinder	hand injury	If cell is supported by cylinder and anvil assembly is not present platten and tie-rods can slide down, trapping hands	3	3	M	1) Always use properly designed mounts 2) Ensure that all mounts used are designed to prevent this eventuality 3) Training to make operator aware of hazard	1	3	L	Instruction in PE operation	PE operating manual
3	Injury while lifting cell	back injury	Cell is very heavy (ca 70kg), lifting by hand is unsafe	3	3	M	1) always use mechanical lifting device 2) training to make operator aware of hazard	1	3	L	Instruction in PE operation crane training	PE operating manual
4	Blowout	injury from shrapnel (*nb. risk to be better quantified by planned tests in Utgård)	Blowout leads to high velocity shrapnel being ejected	3	4	H	1) always use appropriate shielding when a blowout risk is present. 2) follow appropriate procedures to minimise risk of blowout	3	1	L	Instruction in PE operation	PE operating manual
5	Blowout	hearing damage (*nb. risk to be better quantified by planned tests in Utgård)	Blowout results in loud bang	3	3	M	1) hearing protection 2) warn those nearby of risk	3	1	L	Instruction in PE operation	PE operating manual

DIAMOND ANVIL CELLS (§)

SYSTEM: DIAMOND ANVIL CELLS												
				Initial Risk Rating				Residual Risk Rating				
Number	Activity or Event	Hazard	Who might be harmed and how?	Likelihood	Severity	Risk H,M,L	Actions to mitigate Risk - controls	Likelihood	Severity	Risk H,M,L	required training	additional reference
1	Heavy component falls of desk	foot/hand injury	Personnel setting up device could have foot or hand crushed by falling object	2	3	M	Utilise a stand to prevent cell from rolling	2	1	L		
2	Failure of membrane	injury from shrapnel	Personnel adjacent to DAC at point of failure	1	4	L	1) Ensure correct operation of DAC 2) Have designed enclosure to completely mitigate risk 3) Training to make operator aware of hazard	1	1	L	Instruction in DAC operation	DAC operating manual
3	Failure of membrane	hearing injury (* not sure if this is a real hazard. Planned tests in Utgård to quantify risk)	Failure of membrane make create a loud noise	1	3	L	1) Ensure correct operation of DAC 2) Training to make operator aware of hazard	1	3	L	Instruction in DAC operation	DAC operating manual
4	Exposure to BeCu dust particles	lung cancer, berylliosis	personnel exposed to dust particles	2	5	M	1) Ensure no abrasive is used on ESS-DAC body. Use appropriate environmental controls (e.g. fume hood) and PPE (e.g. repiratory mask) if engaging in any activity that might produce dust	1	4	L	BeCu training (if/when it exists). Instruction in DAC operation	DAC operating manual

APPENDIX B: PREMP OPERATION AND MAINTENANCE MANUAL

The following is a simple extract of the operational and maintenance manuals in the main text, intended to be separated out for easy printing. To aid legibility of the printout, the section numbering differs from the main document. However, cross-references are given to link back to the relevant main document sections.

1. THE PORTABLE PRESSURE DRIVER

1.1. Transportation of PPD

Prior to transporting the PPD between locations, the following must be observed:

- Any SEE connected to the PPD should either be fully downloaded or loaded, but isolated from the pressure-driving systems by means of an isolation valve. This applies to equipment connected to both the PACE and MHS sub systems.
- MHS should then be vented to zero pressure by opening either of vent valves V2 ior V3 and confirming complete pressure loss on analogue G1. At least one of Valves V2 or V3 should remain open during transport (see §4.4 for detailed location of valves on MHS).
- The PACE shall also be vented by entering a set point of 1 bara (0 barg). Once at atmospheric pressure, the PACE should be set to "Measure" mode (see PACE manual)
- Any SEE mounted on the PPD shall comply with marked weight limits.
- If SEE is mounted on the PPD, this shall be bolted down to ensure it cannot disengage and fall off.
- All utility connections (electrical power, compressed air or gas, hydraulic lines and ethernet) must be safely disengaged.
- There shall be no loose tools placed on top surfaces of the PPD.

1.2. Set-up of the PPD

Once the PPD reaches the location where it is to be used:

- It shall be positioned safely, in a convenient location relative to the necessary utility connections.
- Both brakes shall be fully engaged.
- Utility connections shall be made and, where necessary hydraulic or gas connections Leak Tested (see instructions for sub-systems and §2.3). PPD shall be positioned to minimise tripping hazard from utility connections, which shall not lie across walkways.
- Where necessary, the SEE will then be transferred from the PPD to a secondary location (e.g. installed at the sample position of an instrument)
- The hydraulic or pneumatic connection between the SEE and the PPD should be re-established following the relevant procedure for the specific sub-system.

- Where connections are re-made these shall be Leak Tested as appropriate for the relevant sub-system.

1.3. Maintenance and modification of the PPD

When performing maintenance on the PPD

- All pressurizing sub-systems must be downloaded to zero pressure and made safe.
- Unless necessary for maintenance operations, utilities shall be disconnected.
- Any replacement of parts must use CE-marked components of appropriate pressure rating for the relevant sub system.
- Any modification that results in a change of the total system volume or MOP shall be assessed for possible impact on classification.

2. MAXIMATOR HYDRAULIC SYSTEM

2.1. Pressure testing the MHS

The MHS is currently operated with an MOP of 1000 barg and has two independent safety systems set at 1.1xMOP. The MHS shall be Pressure Tested (§2.1) every 12 months to a pressure of 1.1xMOP = 1100 barg. In addition, if any modification is made or repair conducted, the MHS shall be retested. The date of the latest pressure test shall be recorded in the MHS maintenance manual, attached to the MHS.

2.2. Leak testing the MHS

SEE equipment is frequently connected and disconnected from the MHS. Each time such a connection is made, the combined MHS-SEE system shall be Leak Tested (§2.3)

2.3. Connecting and disconnecting SEE to/from the MHS

SEE will need to be frequently be connected and disconnected from the MHS during installation on an instrument. It will normally be necessary to maintain a sealing load on the SEE during this operation (e.g. in the case of a PE cell with standard toroidal gaskets 7 tonnes is required). Additionally, it is sometimes necessary that the SEE will have a larger load of up to 20 tonnes (specific scenarios and limits are described in separate sections on specific SEE).

Prior to any operation, the following initial checks should be performed:

- The operator shall check the calibration date of the MHS transducer G1 to ensure that this is current.
- A visual check should be made of all hydraulic connections to identify any obvious signs of damage or oil leakage.
- If the MHS is connected to an air supply, the main air isolation valve V1 shall be closed to ensure no pressure can be developed in the system.

The following procedure should be used to connect SEE to the MHS.

- The MHS shall be connected to the SEE using a loose, finger-tight connection. At this point, a small amount of oil should be bled through to minimise trapped air in the system. This should be done by gentle use of the manual hand pump of the MHS. Once oil is seen to exit through the bleed hole of the HP connection, the connection can be made tight, to the specified 30 Nm.
- If the SEE is unloaded (at zero pressure with isolation valve open), the connection is now complete and ready to use. If the cell is loaded, the additional steps below must also be followed to balance the system:
- The internal SEE pressure P_{SEE} must be visible to the user and the internal pressure on the MHS increased to exactly match this to within < 1 bar (correct calibration of the gauges as per initial checks is necessary to ensure there is not an offset between different gauges).
- At this point, the isolation valve on the SEE should be opened slowly, while continually monitoring P_{SEE} to ensure it remains stable.

The following procedure must be followed to disconnect pressurised SEE from the MHS.

- After any pressure change, the SEE must be left to stabilize for at least 5 minutes prior to disconnection. If the SEE is at non-ambient temperature, it shall be allowed to return to ambient temperature prior to the 5-minute stabilisation time.
- It is required that the SEE has an isolation valve enabling it to be closed off from the MHS.
- It is required that a pressure transducer, or other pressure-measuring device is connected on the SEE-side of the isolation valve, thus this will display P_{SEE}, the internal pressure on the equipment (versus the MHS pressure P_{MHS}).
- The first step in disconnection is to close the isolation valve separating the SEE from the MHS. At this point, the SEE transducer must be powered up so that P_{SEE} can be observed.
- The MHS should now be downloaded by first closing the needle valve V5 to isolate the connection to the SEE and then venting the remaining system by opening either of the vent valves V2 or V3 and confirming loss of pressure on Gauges 1 or 2. At this stage, needle valve V5 should be gradually opened allowing the pressure in the SEE connection to begin dropping. At this point P_{SEE} should be inspected. If P_{SEE} is stable, this confirms the isolation valve is functioning correctly. At this stage, is the user is free to fully open V5 to complete the download of the MHS.

- At this stage, the SEE is isolated from the MHS, which is at zero pressure. The output needle valve V5 should now be reclosed, to minimise any oil loss. The SEE may now be disconnected taking care to ensure no torque is placed on the isolation valve. Once disconnected, any open hoses/piping etc. shall be sealed to ensure no loss of oil and prevent slipping hazard.

2.4. Operating SEE using the MHS:

- The operating principle of the MHS is to set and control the internal output pressure by setting the input air pressure, which is a fixed factor (measured to be 202) below the output. This pressure is then communicated to connected SEE via the needle valve V5. The rate at which P_{SEE} increases is determined by a combination of the pressure differential between P_{SEE} and P_{MHS}, by the opening setting of the needle valve and by the relative volumes of the MHS and the SEE.
- For certain SEE, changes in pressure must be conducted at a controlled low rate (see specific operating instructions for each SEE). As the relative volumes of the SEE and MHS systems are typically fixed, this rate can be minimised by ensuring a low differential between P_{SEE} and P_{MHS} in this case, needle valve V5 can allow extremely slow changes in pressure.
- To establish the desired P_{MHS} the input air pressure can be adjusted using regulator R1 and monitored by inspection of the analogue manometer in the air controller. Subsequently, the flow rate (and therefore the rate of increase of P_{SEE}) can be adjusted using output needle valve V5.)
- After changing pressure, P_{SEE} shall be monitored to ensure that it is stable. Typically, pressure increase will induce warming of the fluid causing a subsequent drop in pressure as the fluid cools. It may be necessary to compensate for this in cases where a stable pressure is critical. However, if P_{SEE} continues to drop after ~ 5 mins following pressure change, this could indicate a leak and should be investigated.
- It should be noted that normal changes in ambient temperature can have a significant effect on total system pressure: which drops upon cooling and increases upon warming. This effect becomes less pronounced as the volume of the connected SEE increases.

3. GE PACE PRESSURE CONTROLLER

3.1. Connecting and disconnecting SEE from PACE

SEE will need to be frequently connected and disconnected from the PACE during installation on an instrument. Often the SEE will be pre-loaded in a SE-workshop and, thus, additional steps are needed to make and the connection (see below).

Prior to any operation, the following initial checks should be performed:

- It shall be confirmed that calibration of all transducers and pressure readouts in the system should are current (calibration certificates for the PACE are available here).
- A check should be made of all gas connections to identify any obvious signs of damage or leakage. This should be done setting the PACE to control at a small pressure of 1-2 barg and using a leak-detecting fluid (e.g. Snoop® or equivalent) to identify leaks. As an additional safety step during this operation, the regulator controlling input pressure should be set to 2 barg, thus preventing any accidental exceeding of the initial leak test pressure.
- It shall be ensured that appropriate gas supply is connected. This is especially important when cryogenic operation is envisaged and users should be aware of possible phase changes in the gas supply. In the cryogenic case the most appropriate supply gas is He, which should be used to minimise risk of supply gas condensing in the pressure lines.
- The source pressure from the input gas cylinder pressure shall not exceed 200 barg.

The following procedure shall be used to connect SEE to the PACE.

- The PACE should be physically connected to the SEE.
- If the SEE is unloaded (at zero pressure with isolation valve open), the connection is now complete and ready to use. However, where cryogenic operation is envisaged, it is additionally necessary to fully evacuate the SEE (and connecting capillaries) to avoid the presence of any moisture, which can freeze and induce a blockage. If the cell is loaded, the additional steps below must also be followed to balance the system:
- The internal SEE pressure P_{SEE} must be visible to the user and the internal pressure on the PACE increased to exactly match this to within < 1 bar (correct calibration of the gauges as per initial checks is necessary to ensure there is not an offset between different gauges).
- At this point, the isolation valve on the SEE should be opened slowly, while continually monitoring P_{SEE} to ensure that it remains stable.

The following procedure must be followed to disconnect SEE from the PACE.

- After any pressure change, the SEE must be left to stabilize. This can be confirmed by monitoring the *effort* value of the PACE, when this drops to 0, the pressure is stable. This typically takes <1 min, however, can take longer if the SEE is at non-ambient temperature. e.g. in the case where the SEE is at cryogenic temperatures, the gas input at room temperature will cool, resulting in a pressure drop, which the PACE will compensate for. If *effort* remains non-zero after ~1 min, and the SEE is at ambient temperature, this could indicate a leak and steps (beginning with a Leak Test) should be taken to identify and correct this.
- It is recommended that the SEE has an isolation valve enabling it to be closed off from the PACE. In this case, there shall also be a pressure transducer between the SEE and the isolation valve.
- If the SEE is to be fully downloaded, then any isolation valve between it and the PACE unit shall be open and the control pressure reduced to 0 barg at an appropriate rate. The rate can be SEE dependent and, in some cases, must be sufficiently slow to avoid damage to the SEE.
- If pressure is to be maintained on the SEE, the first step in disconnection is to close the isolation valve separating the SEE from the PACE. At this point, the SEE transducer must be connected to a readout so that P_{SEE} can be observed.
- The PACE should now be downloaded. This is done by first dropping the control pressure to a value of P_{SEE} – 5 bar. At this point P_{SEE} should be inspected for a period of 30-60 s. If P_{SEE} is stable, this confirms the isolation valve is functioning correctly. At this stage, the user is free to rapidly download the system by setting the control pressure to 0 barg.
- At this stage, the SEE is isolated from the PACE, which is at zero pressure. The SEE may now be disconnected taking care to ensure no torque is placed on the isolation valve.

3.2. Operating SEE using the PACE:

- The input pressure to the PACE limits the maximum pressure that may be delivered to the SEE. This should be adjusted, via the regulator, to an appropriate value for the SEE and the planned experiment (e.g. if the maximum pressure required is 40barg, there is no need to have an input pressure exceeding this).
- The output pressure to the SEE is set by adjusting the set point of the PACE. At this point, if the PACE is in *control* mode, the output pressure will immediately begin to increase/decrease to match the requested value. Each particular SEE may have limits on maximum pressure and/or limits on the rate of change of pressure. These limits shall be imposed as soft limits on the PACE operation (by following instructions in the PACE manual).
- Certain SEE may have further operational requirements described in their operating manual, which must be consulted prior to use.

4. PARIS-EDINBURGH PRESS

Note 1: Some of the procedures and recommendations in this section may be modified in light of planned tests in Utgård (Q3-Q4/2019), where Burst Tests (artificially-induced blowouts of the gasket) will be conducted and the consequences accurately quantified.

Note 2: The present manual describes operation of the oil-driven V3 and VX6 PE presses at ambient conditions (~20° C) using ZTA standard single-profile anvils. The manual shall be extended to include alternative set-ups as and when these are developed.

Note 3: The manual has been written to cover usage of both PE cell types used at the ESS. However, there are some important differences between both devices and, where these affect usage, this has been noted explicitly in the manual. The main consideration is that the conversion from hydraulic pressure to force in tonnes is different for both presses (due to the different diameters of the pistons):

- ***For the V4: 10 bar = 1 met. tonne***
- ***For the VX6: 15 bar = 1 met. tonne***

4.1. Preparing the PE press for loading

- Prior to any work, he PE press should be mounted securely in an appropriate support that provides space for hydraulic connection and allows for the cell axis to be vertical, with the piston on the lower side.
- When the breech is removed from the platen, it should be placed vertically on the desk, anvil side up (so it doesn't roll off).
- All hydraulic connections to drivers should have been made tight and a visual inspection should be conducted of connections within the cell piping system.
- It *must* be ensured that the piston is driven fully back. This is done by using the provided blank between the breech and piston and tightening the breech nut to drive back the piston. Any oil within the cylinder will be pushed out through the hydraulic connection, so the lines should be fully opened towards a 1 bara reservoir.
- The cell should then be Leak Tested. It is recommended to do this by installing the testing blank and applying a pressure of at least 500 bar g (after a successful initial Leak Test §2.3). This should then be monitored for at least 1 hour to observe any potential loss of pressure that would indicate a leak.
- The press can then be assembled by installing, in order from outside to centre: backing discs, seats and anvils on both piston and breech sides of the press. Appropriate centring rings should be used and *set-screws used to hold anvil in place on the breech should be tightened sufficiently so that the anvils don't fall out*. (Important note: the VX6 uses ceramic backing discs and the VX4 uses steel discs. Also, although the anvils are interchangeable between cells, the aluminium centring rings and seats differ in dimensions. Observe marking on the rings to ensure the correct ones are used).

4.2. Loading the sample into the PE press

- The gasket is then put in place and the sample loaded. In some cases, it may be easier to remove the piston anvil and position the gasket and sample before lifting into place in the cell. For encapsulating gaskets, the lower part should be installed prior to inserting the sample and the upper part of the gasket should be installed after the sample is in place.
- The breech is then inserted and screwed down until contact is made with the sample. The breech should be made tight using an appropriately sized spanner.
- The safety shield shall now be installed.
- If the sample contains a liquid, a sealing load of ~7 tonnes will form a positive seal preventing leakage. (Important note: for the V4 this corresponds to 70 bar hydraulic pressure, while for the VX6, this corresponds to 105 bar).
- For certain samples, for example volatile or reactive materials, the above procedure may need to be done quickly. In this case, at least two people (A &B) shall be present and the following procedure followed:
    - A: inserts sample into cell and places top of encapsulating gasket, if it is being used.
    - B: tightens breech down onto cell and applies shield
    - A: applies sealing load to the cell.

4.3. Using the PE press with ZTA anvils

After preparation, the sample can now be pressurised by increasing the hydraulic load. It is important to do this at a controlled pace. The pace depends on sample type, tonnage and anvil type. Currently, this manual covers operations with liquid or solid samples[3], in ZTA single-toroid anvils. Loads and rates are specified in tonnes, this will correspond to a different hydraulic load depending on cell type.

At all times when the cell is being actively pressurised, some form of physical shielding must be in place. Physical shielding should be installed at all times when the load is above 10 tonnes *(see § 6.6.7)*

Low-pressure operation: up to 10 tonnes, pressure should normally be increased at a rate of ~0.3 tonnes/s (18 tonnes/min). Shielding must be in place during pressure change. If the pressure has been held stable for at least 10 minutes, it is permissible to remove the shielding (this is occasionally necessary, for example, to adjust collimation), however, PPE including safety glasses and Kevlar gloves should be worn.

Medium-pressure operation: 10 to 60 tonnes. Physical shielding should be permanently in place. Pressure shall be increased at a moderate rate of ~1 tonne/min and, in addition,

[3] The loading of gaseous samples at the ESS will be addressed in the future.

for every 5 tonnes, pressure shall be kept static for a period of at least 5 minutes. Likelihood of a blowout (see §6.6.6) is low.

High-pressure operation: 60-80 tonnes. Pressure should be increased at a slow rate of ~0.5 tonnes/min and, in addition, for every 0.5 tonnes, pressure should be kept static for a period of at least 5 minutes. In this pressure range, blowout has a medium probability.

Conversion Table

Load or load rate	**Hydraulic pressure V4**	**Hydraulic pressure VX6**
10 tonnes	100 bar	150
60 tonnes	600 bar	900
80 tonnes	800 bar	1200
0.3 tonnes/s	3 bar/s	4.5 bar/s
1 tonne/min	10 bar/min	15 bar/min
0.5 tonnes/min	5 bar/min	7.5 bar/min

4.4. Cryogenic loadings

In circumstances where samples decompose at room temperatures, it is sometimes necessary to load these at cryogenic temperature (usually under liquid N_2) and then rapidly pressurise in order to achieve stable pressure conditions before the sample degrades. In this case, faster rates up to ~1 tonne/s can be employed. Although blowout risk is low, some form of shielding must always be employed. After sealing, the sample and anvil assembly shall be allowed to warm to room temperature before further increasing pressure. Throughout warming, shielding must stay in place.

4.5. Downloading the PE press with ZTA anvils

The risk of a blowout is highest during download and most often occurs in the final stages of decompression

At all times when the cell is being downloaded, some form of physical shielding must be in place*.*

In order to minimise the risk of a blowout occurring, the entire download should be conducted at a very slow rate ~ 0.02 tonnes/s

4.6. Blowouts

With the PE-press, a "blowout" refers to an event where either the gasket or anvil fails suddenly under the internal pressure of the sample. In this case, often, small fragments of the metal gasket can be violently ejected from the anvil assembly resulting in a risk of physical injury. In addition, the anvils then hit together with significant force, resulting in a loud banging sound and possible ejection of anvil fragments.

Blowouts are, by nature, probabilistic phenomena, but some 30 years of experience using the devices have quantified the level of risk in different scenarios. Moreover, the key risk factors are well understood and include: the physical form of the sample (e.g. solid, stable liquid, unstable liquid[4] or gas), the load on the anvils and the material of the gasket and anvil material.

These risks are quantified here for the various factors in Table 7. At present, only ZTA anvils with either TiZr or steel gaskets are considered as these are the only ones currently used at ESS. Table 7 will be continually updated as new anvil and gasket materials are employed

Table 10 Risk factors when operating the PE press

Anvil/gasket material	Sample state (@ambient P & T)	Load (tonnes)	Likelihood* (1-4)	Severity** (1-5)	Risk
ZTA/(TiZr or steel)	solid	0-10	1	4	Low
		10-60	2	4	Medium
		> 60	4	4	High
	Stable liquid	0-10	1	4	Low
		10-60	2	4	Medium
		> 60	4	4	High
	Unstable liquid	0-60	2	4	Medium
		> 60	4	4	High

[4] Primarily any liquid whose vapour pressure at ambient temperature is less than 0.5 bar g (i.e. the liquid will itself pressurize the sample chamber)

In all cases where the risk of a blowout is medium or higher, physical shielding must be in place. Where the risk is low, it is possible to remove the shielding if the hydraulic pressure has been constant for at least 10 minutes and appropriate PPE is used.

4.7. Shielding

Due to the intrinsic risk of blowouts when using the PE press, some form of shielding is considered an essential part of their operation.

In normal cases, this shielding will comprise physical barrier of sufficient strength to stop any shrapnel that may be released in the event of a blowout, thus negating the risk of physical injury. All possible lines-of-sight from sample out of the cell must be covered by the shield, as this will correspond with paths for the incident and scattered neutron beams, then a low attenuating material, such as Al, will likely be used during neutron operation (an alternative material for offline testing is polycarbonate, which has the advantage of being transparent). In addition to specially-made "blast shields", ancillary sample environments e.g. vacuum vessels for cryostats may provide sufficient protection. In any case, calculations and measurements must be provided to demonstrate that the shielding has adequate strength (this is particularly important where the wall of an evacuated vessel acts as shielding as perforation of the wall will result in an implosion).

In extraordinary cases, it may be necessary to directly access the sample while there is a medium risk of a blowout. In this case, the shielding will take the form of PPE such as Kevlar gloves and face shield and should only be attempted by trained personnel. This is never permitted when the risk of blowout is high.

5. DIAMOND ANVIL CELLS

Loading and operating DAC cells is a special activity because of the delicate and expensive nature of the equipment. In addition, each design of DAC has its own considerations, so experience of one type of DAC does not ensure competence with other designs. Correspondingly, this manual should not be considered a replacement for one-on-one instruction by an expert user, who is familiar with the operation of the devices.

5.1. Preparing the gasket for loading.

- The first step is to consider what material to use for the gasket. Issues such as maximum pressure needed, temperature and possible chemical reaction of the sample are part of this assessment.
- If gas loading is required (note this is currently not available at ESS), then the gasket must be carefully polished and cleaned to ensure a seal. Most gas-loading devices have insufficient force to plastically deform the gasket with large neutron-size culets. Therefore, small scratches and pieces of dirt will result in a leak.

- As a next step, the alignment of the diamonds must be confirmed: both culets must be exactly concentric and parallel to each other. Interference fringes can be observed under a microscope to assess parallelism, with a goal of having only 1—2 fringes across the entire culet surface.
- Prior to inserting the gasket, an alignment mark should be made (a carbide scribe works well for this). For gas loading, it must be ensured that the mark does not overlap with the culet.
- Both gasket and diamond culets should be cleaned at this point. And this should be continued repeatably, throughout the loading.
- The gasket is then placed on one of the diamond culets and aligned to be perfectly flat and concentric with it. Use of some wax is helpful to maintain this alignment. A mark should be made on the seat opposite the score on the gasket. This ensures that the gasket can be repeatably inserted in the same orientation relative to the anvils.
- The gasket is then indented to an appropriate depth by applying force on the cell. The approach at ESS is to use hydraulic press and a load gauge to repeatably apply load. As, for a given force, the resulting indent depth depends both on culet size and gasket material, several runs may be needed do determine the appropriate force for a given set-up.
- The indent thickness is chosen by taking account of the required maximum pressure, culet size and compressibility of the sample. Typical values are 100-190 µm.
- After indenting, the gasket must be removed from the cell and the sample hole drilled. At the ESS, this is done using electro-discharge machining (EDM) with carbide rod bits of appropriate diameter. As a rule of thumb, gasket holes should be ~ 40% of the culet diameter. However, for highly compressible samples, such as pure hydrogen, much larger holes will be needed ~ 60-65% of culet size.

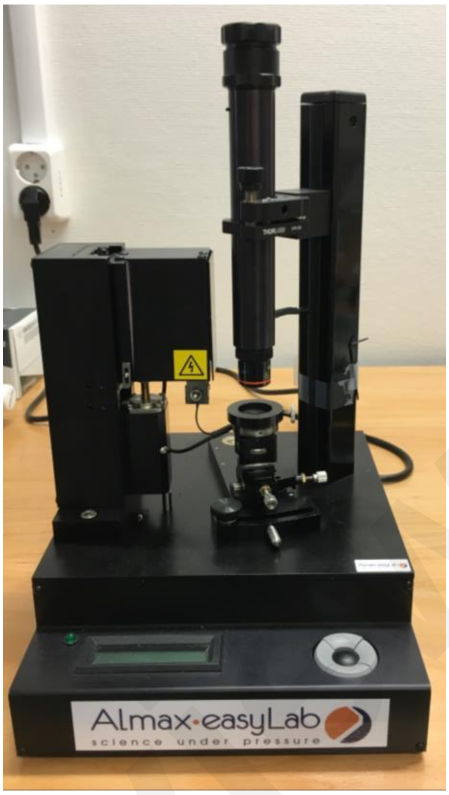


Figure 17 EDM gasket drilling device

Figure 18 DAC gaskets. On left is a new un-indented gasket, which has a diameter of 6mm and thickness of 400 µm. On the right is an indented and drilled gasket. The alignment mark is seen at approximately 1 o'clock. Not that, in addition to the indentation, the gasket is seen to cup downwards.

- After thoroughly cleaning the gasket (e.g. using an ultrasonic bath), the gasket should be re-introduced onto the diamond. Typically, the indent itself will help ensure that the gasket correctly re-centres on the diamond. However, it can still

rotate relative to the culet, so the alignment mark should be used to ensure orientational repeatability.
- As a final step, the DAC should be fully reassembled and a small load applied by hand to 'bed down' the gasket. It is helpful at this stage to re-apply wax to hold the gasket in place.

5.2. Loading the sample

- After the preparations described in §7.6.1, the DAC is ready for loading the sample. Depending on the nature of the sample (solid, liquid or gas), the exact details of this will vary.
- It is also best practice to include a pressure calibrant in the sample chamber at this point. Typically, a small chip of ruby can be used, as the laser-induced fluorescence of ruby has been calibrated with respect to pressure. Diffraction standards can also be useful for *in situ* pressure determination.
- In addition to the sample and pressure calibrant, pressure media should also be used to ensure hydrostatic pressure generation.
- After sample, calibrant and pressure media are in the sample chamber, the cell should be closed and sealed, normally by applying initial load using the screws on the cell cap. This load should be sufficient to plastically deform the gasket, thus making a seal.
- At this point the cell should be stable indefinitely and can be easily transported between facilities.

5.3. Adding the DM driver

Whereas with more conventional x-ray DACs, the cap screws could be used to further increase pressure, however, the large culet sizes used for neutrons mean that the some mechanical, hydraulic or other device must be used to generate sufficient force. At the ESS the gas-driven DM drivers are used. These are assembled by:

- Dropping the DAC into the lower part of the DM enclosure, adding the pusher and DM and then screwing the upper part of the enclosure down trapping the DM between pusher and enclosure. Prior to this, both pusher and upper enclosure should be cleaned to be free of dust, which will otherwise indent into the DM.
- It must be ensured that the DM enclosure is fully tightened and a tool is available to do this. It is especially important to ensure that no torque is placed on the 1/16" tubing while tightening the upper enclosure.
- Note, tightly closing the DM will likely also increase the sample pressure, which might be an important consideration in certain circumstances. If a ruby pressure calibrant is in place, any increase in sample pressure can be determined.

- At this point, the initial locking screws in the cell cap will be disengaged, and the entire force on the gasket is being delivered by the DM enclosure.

5.4. Changing pressure with the DM driver

- Once correctly assembled, positive gas pressure in the DM will increase force on the anvils, leading to pressure generation on the sample.
- The 1/16" capillary on the DM should be connected to a gas source (e.g. a gas cylinder via a regulator or the PACE gas driver). If cryogenic operation is envisaged, the tubing should then be evacuated to ensure no moisture is present in the line.
- As described in §7.2.2, the force generated will depend on membrane size. Meanwhile, sample pressure will depend on both the load generated and the details of culet, gasket and sample. Normally sample pressure *vs* load curves will be available, but these should be considered guides only as each loading will be different.
- When increasing pressure, the gasket hole should be continually observed. If any enlargement or deviation from roundness of the gasket hole is seen, or if the hole moves off the centre of the culet, then the loading should be terminated to ensure safety of the diamond anvils.
- In addition, to observing the gasket, the sample pressure *vs* load should be continually monitored. In normal operations, the dependence should be almost perfectly linear. If this curve is observed to plateau or if its slope changes, this can indicate problems such as mechanical failure of cell components, gasket failure, or blockage in the capillary. Further pressure increase should be postponed until an explanation is found.
- When decreasing gas pressure, a significant hysteresis in sample pressure should be expected due to friction in the system. This often means that even after the DM is fully downloaded, significant pressures (in excess of 10 GPa) may be present within the sample. Due to the microscopic sample size, there is no hazard from this pressure. However, this may be an important consideration for the planned experiment.