Document Type	Description
Document Number	ESS-1545382
Date	Sep 25, 2019
Revision	1 (1)
State	Review
Confidentiality Level	Internal
Page	1 (27)

HIGH-PRESSURE SAMPLE ENVIRONMENT SYSTEMS – GAS, LIQUID, CLAMP AND PE TYPE - PRELIMINARY DESIGN DOCUMENTS

	Name	**Role/Title**
Owner	<<Malcolm Guthrie>>	<<Role/ Title>>
Reviewer	<<Alexander Holmes, Harald Schneider>>	<<Reviewers>>
Approver	<<Arno Hiess>>	<<Approver>>

TABLE OF CONTENT PAGE

LIST OF TABLES

LIST OF FIGURES

1. SCOPE

This document provides conceptual designs for high-pressure sample environment systems supporting science activities on the first 8 instruments within the ESS construction budget. The focus is limited to “conventional” devices which are well established and in routine use at existing neutron facilities. This covers device types including: gas cells, liquid cells, clamp cells and Paris-Edinburgh cells.

Excluded are more experimental and developmental devices and devices based on single-crystal windows or anvils such as diamond-anvil cells.

1.1. Overview of document

The document is split into two sections. The first relates to the category of high-pressure device here called, gas, liquid and clamp pressure vessels. Vessels of this type have been in use in neutron research since the 1960s and typically are limited to maximum pressures in the vicinity of 2.5 GPa. The second section relates to so-called Paris-Edinburgh devices, developed in the 1990s and which can access pressures up to ~ 20 GPa for several neutron techniques.

The approach here is to first survey the needs of the first 8 instruments in the ESS construction budget and then to survey provision of high-pressure sample-environment equipment at other facilities. This is done separately for both device types as described above. With this input a conceptual strategy for supporting high-pressure science on the first 8 instruments at the ESS is proposed, with initial ideas of hardware requirements.

2. GAS, LIQUID AND CLAMP PRESSURE VESSELS

2.1. Introduction

Gas liquid and clamp (GLC) cells are a critical part of conventional high-pressure sample environment at neutron facilities. They cover pressure ranges up to 1.0 GPa (gas) and 2.5 GPa (clamp) and deliver the highest available sample volumes in this pressure range. Although the key elements of the design are well established, a relatively wide variety of dimensions and materials are needed to cater for different neutron measurements. Here, we examine the requirements for the first 8 instruments in the ESS suite following the current (Feb 2018) baseline suite summarized in Table 1

Table 1 Baseline first 8 instruments

Instrument	Class	Max. Beam size height x width	Min. Beam size H x w (mm)	Demand for GLC cells (3=high, 1=low,0=none)

		(mm)		
MAGIC	DIFF (SXL)	5.0 x 5.0	0.1 x 0.1	2
DREAM	DIFF (PWD)	10.0 x 10.0	1.0 x 1.0	3
CSPEC	SPECT	38.6 x 23.7	10.0 x 10.0	3
BIFROST	SPECT	20.0 x 20.0	1.0 x 1.0	3
BEER	ENGINEERING	40.0 x 40.0	0.2 x 0.2	1
ODIN	IMAGING	50x50	10.0 x 10.0	1
LOKI	SANS	12.0 x12.0	2.5 x 2.5	1
ESTIA	REFLECT	NA	NA	0

[Numbers in YELLOW are estimates where information was not available or otherwise not provided]

2.2. Survey of Existing Facilities

We have conducted a survey of existing facilities (See §7). From this, we identify the key technologies employed and adapt these to the baseline suite of 8 (Table 1) with a focus on supporting early science success at the ESS.

A survey of existing facilities (ILL and ISIS) has been conducted by looking at published information on their cells and capabilities. Details of the results are in §7, here we summarise our key observations:

Materials: Three main materials are used in existing designs: high strength Al alloy (7075), null-scattering TiZr, and heat-treated CuBe. Each material has its own unique properties from both mechanical and neutronics perspective (discussed further below in §2.3). Note: single-crystal sapphire is also used as a material for windows in specialised SANS cells, these are not covered in this document, being treated separately as part of PREMP projects supporting gem-anvil type cells.

Maximum Operating Pressure: For gas and liquid cells, both facilities surveyed tend to support 3-4 standard pressures e.g. for ILL gas cells, these are 0.1, 0.3, 0.5 and 0.7 GPa. The highest reported pressure for gas cell operation in neutron user programmes is 0.8 GPa (ISIS CuBe cell with TiZr layer). We note ongoing projects within the Sine2020 framework to enable operation of gas cells up to 1 GPa. This is observed with interest by the ESS, but, as this is an experimental technology is out of scope of this document.

For clamp cells, typical pressures are 1 – 1.5 GPa. Both ISIS and ILL also offer McWahn cells up to 2.5 GPa.

As discussed further in §2.4, maximum operation pressure is dictated by local regulatory restrictions and certification requirements, for example safety factors. At the present time, this is not well defined for ESS operations.

Sample volumes: For gas and liquid cells, a range of volumes are provided, catering to different experimental needs. Typically, ILL and ISIS both appear to standardize sample volumes, although the outer diameter of the vessels increase with pressure. ILL also has several non-standard sizes. Cells with a volume of 1.5 to 2.0 cc seem to be most prevalent, although there are some (e.g. the ILL 0.5 GPa Al gas cell), which can be as large as 10 cc. Clamp cells operate at higher pressure and, correspondingly have lower volumes of 0.4-0.6 cc, with the exception of McWhan cells, which have only 0.06cc.

2.3. Cell construction and Materials

The combined demands of neutronic and mechanical properties strictly limit the possible materials. As seen in §2.2, typically only 3 basic materials are used:

- **High strength aluminium alloys**: although these are the weakest of the 3 materials we consider, they offer significant neutronics benefits in the form of relatively high transmission (σ_{abs} = 0.231 barn), low coherent (σ_{coh} = 1.495 barn) and very low incoherent (σ_{abs} = 0.008 barn) scattering cross-sections. These properties primarily benefit spectroscopic measurements using single-crystals, where the scattering background from the container can be avoided to a certain extent.
- **$Ti_{0.676}Zr_{0.324}$ null-scattering alloy:** The key benefit is an almost negligible coherent scattering length: no Bragg peaks are generated. This is highly advantageous for powder diffraction, although there are the disadvantages of high incoherent scattering and high absorption. Its mechanical properties are also significantly better than Al.
- **CuBe:** The neutronic properties of CuBe are not favourable (it activates, scatters very strongly and also absorbs), however, it also has some important advantages: it is mechanically the strongest of the three materials, has a high thermal conductivity (advantageous for low-temperature studies), it is not susceptible to hydrogen embrittlement, making it important for use in hydrogen-proof vessels and, last but not least, is non-magnetic.

Table 2 Mechanical properties pressure-cell materials

Material	Yield strength (MPa)	Density (gcm^3)	Thermal conductivity (W/mK)
7075 Al alloy	503	2.81	130

TiZr alloy	690-840	5.23	Low?
CuBe (HT)	1200	8.26	84-130

Table 3 neutronic properties pressure-cell materials

Material	Coherent scattering length (fm)	Coherent cross-section (barn)	Incoherent cross-section (barn)	Absorption cross-section (barn) at 2200ms^{-1}	Activation
7075 Al alloy	3.676	1.772	0.0222	0.333	low
TiZr alloy	0.000	3.094	1.9408	4.174	low
CuBe (HT)	7.709	7.475	0.5433	3.779	med

2.4. Safety considerations and certification requirements

2.4.1. Normal operating scenarios

At the time of writing, the ESS has not yet fully established its requirements on certifications for specialised high-pressure sample environments. The relevant EU documentation is the Pressure Equipment Directive (PED-2014/68/EU), with additional local requirements from Swedish Law (in particular AFS 2017:3 and AFS 2006:8) . However, it is unclear whether the PED is sufficiently broad to cover all envisaged high-pressure SES at ESS and we note there are directly conflicting requirements between the PED and neutronic behaviour of the various materials used.

After internal discussions and in dialogue with other facilities we have defined a *preliminary* set of documentation, certification and testing requirements that we expect to apply to at the ESS. This list is considered provisional until ratified.

- All equipment must have a full operation manual (in English).
- Full set of mechanical drawings for all components.
- Documents detailing calculations that identify mechanical weak-points within equipment and help define its operational life.
- A detailed risk assessment including specific mitigations of any hazards to an acceptable level.

- Documents detailing calculations confirming that the *burst* pressure (BP) of any vessel is at least a factor of **2.0** above the maximum operating pressure (MOP)[1].
- Material conformity, especially for the mechanical properties, is assured by the delivered inspection certificate 3.1 after norm EN 10204.
- The pressure test will be relied on to veryify that no inner defects which otherwise would not be detectable.
- Reports detailing testing of equipment to a test pressure (TP) **TBC**. The PED requires a test factor of 1.44 MOP, however, that this is physically impossible in some cases, with test equipment unavailable to reach such a high pressure. We also note that for equipment that is operating very close to the intrinsic limits of the materials (as may be required in scientific research), such testing regimes may actually induce a *higher* risk of failure in particular in brittle materials (which are sometimes unavoidable in order to obtain neutronic compatibility) through over-plastification.

A provisional safety factor of 1.4, which is compliant with the PED as stated above, has been used as references to define the proposed ESS suite listed below in §2.5. During detailed design phase of future work, these safety factors will be defined and this may lead to slight modification of designs and specifications for the ESS suite.

2.4.2. Special operating scenarios

It is envisaged that in some operational scenarios it will be desirable to operate cells in excess of the 2.0 safety factor with respect to BP. Consultation with operating sources, indeed suggests many scenarios where vessels actually reach YP during an experiment.

We believe it is important to facilitate such scenarios as, by nature, cutting-edge science will explore the limit of technical possibilities. We, therefore, will develop special operating procedures that allow higher pressure operations, ***up to but not exceeding the test pressure***. Procedures will negate all risk to personnel, for example by employing additional shielding or by only allowing pressurization within the experimental cave when no personnel are present.

2.5. Proposed ESS gas, liquid and clamp cell suite

As detailed in the introduction, the strategy for defining the suite of cells covered by this work package has been to focus on providing essential capabilities to the first 8 instruments. The following considerations are made:

- For powder-diffraction, including nano-crystalline and amorphous diffraction measurements, null-scattering TiZr cells are deemed to be essential. The key instruments that will benefit from these are DREAM and BEER.

---

[1] Note ILL uses an alternative limit relating to yield pressure (YP) so that MOP = YP/1.2. This usually will correspond to a more stringent constraint (i.e. BP/YP > 1.7, which for piston cylinders is true for all K>1.8)

- The high-transmission of aluminium cells are most beneficial for flux-limited techniques such as INS. Where single-crystal samples are used, powder lines from the Al can be avoided. CSPEC will benefit directly from these cells as will ODIN, where the high transmission will facilitate imaging measurements
- CuBe is advantageous for low temperature applications.
- SANS measurements on LOKI and SKADI will probably require separate cells using low-background sapphire or diamond windows, these are not considered here.

2.5.1. Standardised sample sizes

Sample diameter It is considered beneficial to define a series of standard sample sizes for ESS pressure cells. This may allow for the possible interchange of internal components (such as seals) and may also aid in definitions of incident and diffracted beam collimation. As pressure is related to area, we propose to follow a series where area decreases in increasing factors of two (and radii decrease by factors of $\frac{1}{\sqrt{2}}$) starting from a reference diameter.

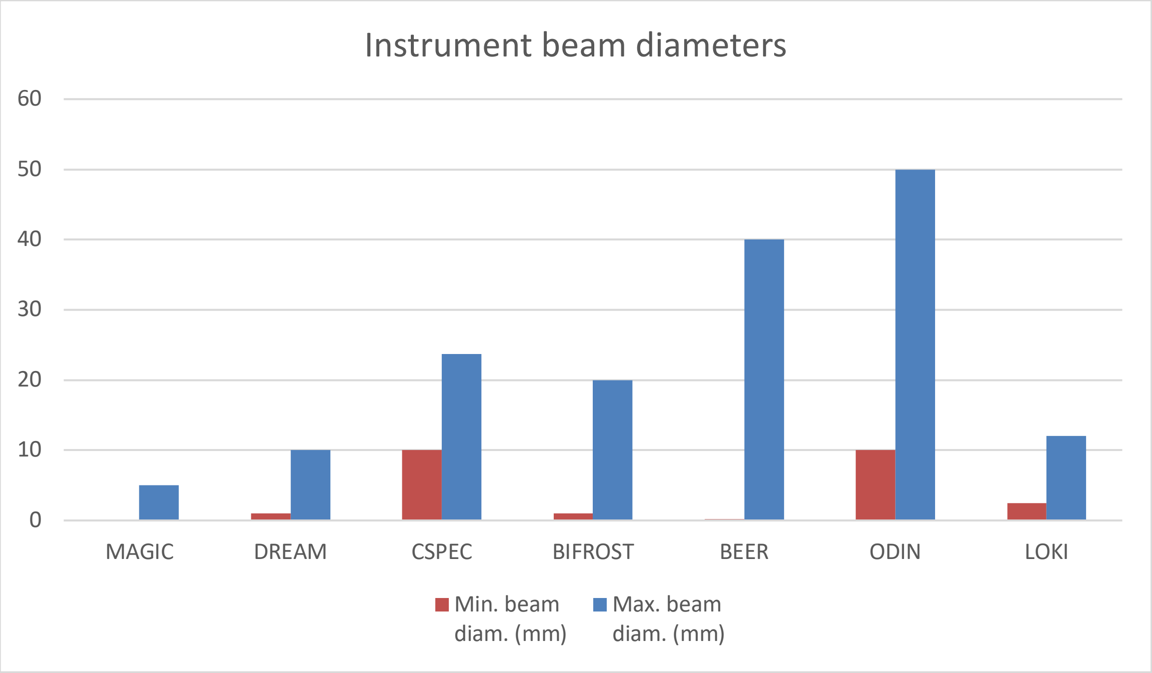

Figure 1 Beam diameters baseline suite of 8 instruments

Although the ESS beams are expected to be extremely bright, many early science goals depend on both measuring very weak signals or measuring strong signals very quickly. Therefore, large sample volumes must be supported. Figure 1 illustrates the range of expected beam sizes for the reference suite of 8 instruments. This can then be compared with range of currently supported sample sizes at existing facilities. In light of these considerations, we define a base sample diameter of 10 mm (cell inner diameter, ID), which then gives rise to the series of diameters - ***7.0, 5.0, 3.5 mm*** etc – each corresponding to a halving of volume (rounded to the nearest 0.5 mm)

Sample height Another important design criterion is the height of the sample. This is not limited by cell design and so other facilities tend to have large aspect ratios R (= height/diameter), often exceeding 10. However, Figure 1 makes it clear that the ESS will not benefit from these as the maximum beamsizes are relatively small. There appears to be little benefit in samples that are taller than 25mm, and some instruments will benefit from this (apart from DREAM, MAGIC and LOKI whose maximum beamsizes are too small). We, therefore, propose 25mm to be the standard height for ESS cells.

Note this may be revised if other instruments come online with larger beam sizes.

2.6. Maximum Operating Pressure

Several requirements work together to define maximum operating pressure including determined by administrative controls (i.e. safety factors), materials used and neutron scattering considerations.

Neutron scattering considerations

Cell outer diameter For thick-walled cylindrical pressure vessels, increasing the outer diameter (OD), will increase the strength both YP for a given ID. However, YP rapidly plateaus as a function of wall ratio K. Moreover, this leads to increasing problems with neutron attenuation and cell background. A typical maximum practical value for K is 3 [Klotz 2013]

At this preliminary design phase, it is assumed the priority will be data quality. If we assume a maximum acceptable attenuation of 50%, Figure 2 shows a corresponding maximum pathlength of ~ 20 mm for CuBe and TiZr. In principal, the low attenuation of Al permits significantly longer pathlengths, however, the increased scattering must also be considered leading to larger backgrounds.

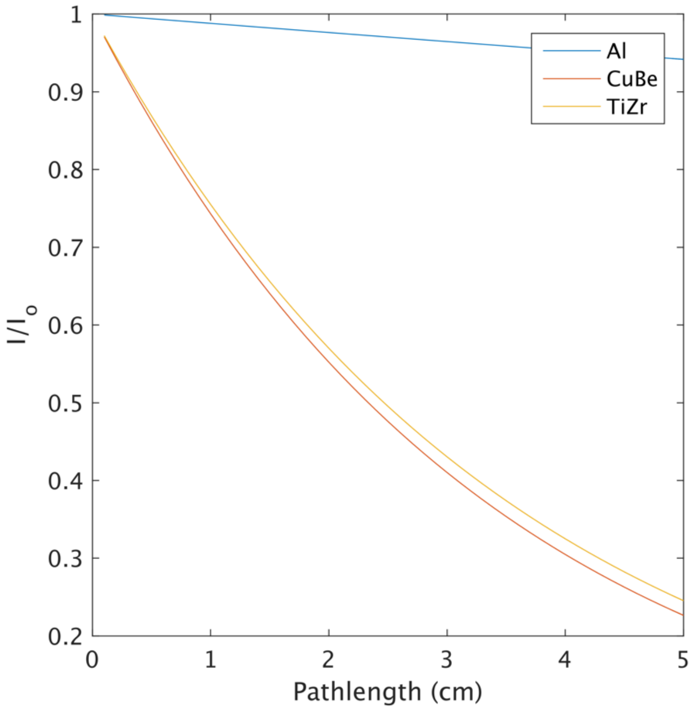

Figure 2 Attenuation of different cell materials at 1.798 Å(note these are only illustrative calculations based on materials with no impurities)

Proposed K values for different cells

Here we propose maximum K-values for different cells and sample volumes

Material	Sample ø (mm)	Sample vol. (cc)	Tot. path-length though wall (mm)	Cell outer ø (mm)	K
TiZr	10	2.0	20	30	3
	7	1.0	14	21	3
	5	0.5	10	15	3
CuBe2-TF	10	2.0	20	30	3
	7	1.0	14	21	3
	5	0.5	10	15	3
Al	10	2.0	50		
	7	1.0			
	5	0.5			

Maximum Pressure for different cells

The internal pressures corresponding to initial plastic yield (IPY) at the surface of highest stress (internal layer of cylinder) and burst pressure (BP) are a function of the mechanical properties of the cell material and the wall ratio K using the ***Von Mises Criterion (check).***

Preliminary calculations for thick-walled cylinder under internal pressure for CuBe2-TF, TiZr and Al 7049 are given in Appendix X

Material	0.2% yield strength or proof stress (N/mm2)	K	Initial plastic yield, IPY (MPa)	Burst pressure, BP (MPa)	Max operating pressure, MOP – normal use (MPa)	Test pressure, TP (MPa) taken as 1.4*MOP
CuBe2	1215	3	623	1541	770[2]	1079
TiZr	690	3	354	875	437	613
Al 7049A-T6	570	3	293	723	362	506
Al 7075	460	3	236	584	292	408

We note that the MOP in all cases exceeds the IPY, which may require that the re-machining of cells after use or even single-use cells. For this reason it is viewed as essential that ESS aquires in house experience and ability to fabricate and maintain these kinds of pressure vessels.

Note, as stated in §2.4.2, operation exceeding MOP, up to but not exceeding the test pressure is envisaged in special cases. In light of this, the test pressure represents the highest possible operating pressure for the cell.

2.7. Proposed gas, liquid and clamp cell suite

The provisions considered above suggest the following matrix of pressure cells options for supporting early science on the initial ESS suite of 8 instruments (Table 1).

2.7.1. Gas and liquid cells

Cell class	**material**	**ID (mm)**	**OD (mm)**	**K**	**MOP (MPa)**

---

[2] The envisaged test facility will enable testing up to 1000 MPa. Therefore, where the test pressure (defined by 1.4*0.5*BP = 0.7*BP) exceeds this, it will be truncated to 1000 MPa. Correspondingly the MOP cannot exceed 714 MPa

Gas	TiZr	10	30	3	437
	TiZr	7	21	3	437
	Al	10	30	3	362
	Al	7	21	3	362
	CuBe2	7	21	3	714
Liquid	TiZr	10	30	3	437
	TiZr	7	21	3	437
	Al	10	30	3	362
	Al	7	21	3	362

These specifications should be considered as starting points to be finalised during a subsequent detailed design stage.

2.7.2. Clamp cells

TiZr MOP 1.0 GPa 0.3cc

CuBe MOP 1.5 GPa 0.3cc

McWhan cells are not presently envisaged

The pressures and volumes noted here are comparable to those used routinely at other facilities. However, we note that assumed administrative limits imposing a safety factor of 2 to burst pressure is incompatible with these specifications. In particular, even by increasing K to a value of 6 or 7, we would expect BP of 1.55 GPa or 1.66 GPa for Al and CuBe cells respectively, equating to a safety factor of only 1.5-1.7. It is for this reason that the specialised operational scenarios, as discussed in §2.4.2, will be necessary. The exact policies remain under development at this stage.

3. PARIS-EDINBURGH SYSTEMS

3.1. Introduction

This document details concepts regarding the requirements and subsequent specification of Paris-Edinburgh (PE) systems related to the ESS high-pressure sample environment pool. There are three separate sections, which are each here described separate in this document:

1) The Paris-Edinburgh cell suite
2) The Paris-Edinburgh gas-loading device
3) The Paris-Edinburgh cryostat

The principles guiding the requirement selection are to enable the broadest range of high-pressure science across the first 8 instruments in the ESS suite. This document is based on the current (Feb 2018) baseline of first 8 instruments summarized in Table 4.

Table 4 Baseline first 8 instruments at ESS

Instrument	**Class**	**Primary geometry**[3]	**Demand for PE systems (3=high, 1=low,0=none)**
MAGIC	DIFF (SXL)	L	2
DREAM	DIFF (PWD)	T or L	3
CSPEC	SPECT	L	2
BIFROST	SPECT	L	3
BEER	ENGINEERING	T or L	1
ODIN	IMAGING	L	1
LOKI	SANS	L	1
ESTIA	REFLECT	NA	0

The primary stakeholders are perceived to be the powder diffractometer (DREAM), and the extreme-conditions spectrometer (BIFROST). There will also be some demand from the single-crystal diffractometer (MAGIC) and CSPEC) and the requirements have subsequently been chosen to optimize performance on these instruments. Some applications are also envisaged for MAGIC, LOKI, BEER and ODIN. We noted that cryogenic operation of the PE devices will also be critical.

It is envisaged that this document will be superceded by a subsequent detailed design stage, which will finalise the requirements and specification of the various systems.

3.2. Sub-System 1: The Paris-Edinburgh Cell Suite

Each Paris-Edinburgh system will include both the PE load-frames and the sample assemblies which comprise: backing discs, seats, anvils, gaskets and a series of alignment rings.

3.2.1. Paris-Edinburgh load frame

The PE load frame comes in two distinct styles, the older V-series and more recent VX-series, shown in Figure 3. Within each series, a range of standardised designs with capacities from 50 to 400 metric tonnes are available (Table 5).

---

[3] Transverse (T) means beam enters along load axis; Longitudinal (L) means beam enters perpendicular to load axis.

Table 5 Certain specifications for various standard PE load frames.

Type	Capacity	Mass (kg)	cols	Rear access	Ø piston (mm)	Height (mm)
VX1	50	10	2	Yes	50	188
VX2	50	10	2	Yes	50	188
V3	250	50	4	No	114	309
VX3	200	50	2	Yes	114	315
V4	250	50	4	Yes	114	309
VX4	200	50	2	Yes	114	315
V5	150	35	4	No	92	263
VX5	130	35	2	no	92	242

Customised variations of the standard design are also available, for example, extended cell bodies (allowing additional volume for specific sample assemblies) or modified pistons with through-holes for either neutron or optical beams to enter and exit.

At present, all instruments at the ESS are foreseen to benefit from the design of the VX-series. This design provides a greatly enhanced angular access (160° horizontal), and a larger and more flexible volume for sample assemblies. The result is an extremely versatile design well suited to the demands of a multi-instrument sample-environment system within the ESS pool.

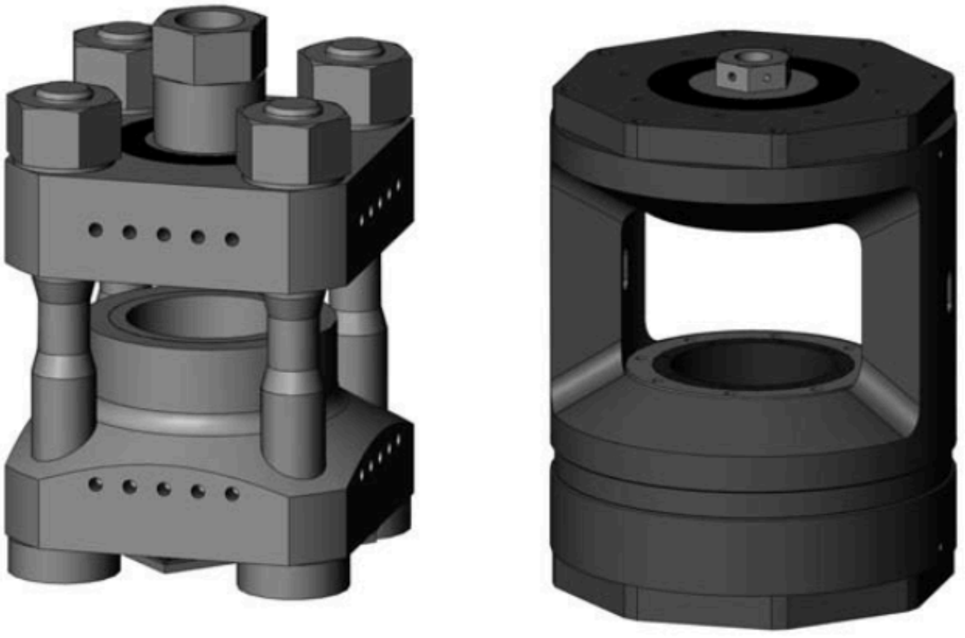

Figure 3 PE load frame (left) shows the V-series cell, characterised by a 4-fold symmetry along the load axis. (right) shows the VX-series cell, characterised by a 2-fold symmetry along load axis.

The required capacity is ultimately determined by a combination of the sample size and maximum pressure requirement. The characteristics for two main “standard” anvil geometries in use at existing facilities (e.g. ISIS, MLF and SNS) are single and double-toroid type designs as detailed in Table 6.

Table 6 Details of different standard anvil types

Anvil type	**Anvil material**	**Sample volume (mm^3) (encapsulated)**	**Typical Maximum pressure (GPa)**	**Required load (met. Tonnes)**
Single-toroid	WC	55.5	10	100
	ZTA	55.5	7	70
Double-toroid	SD	16.8	20	200

It seems likely that the ESS will benefit from a low-moderate capacity press, such as the VX5. The 130 tonne capacity of the press is compatible with all current designs of single-toroid anvil. In addition, its low mass of 35kg will be advantageous for low temperature applications and generally makes the easier handling. It is also considered highly likely that the beam brightness of the ESS will enable smaller sample volumes, likely bringing the higher pressure of current double-toroid designs within reach of 130kg capacity press.

Meanwhile, given the importance of low-temperature operation *and* the likely smaller sample sizes (and, thus, lower force requirements), the smallest capacity VX 1/2 designs are attractive for use at the ESS. We note that, increasing, these cells are being used in the ILL (primarily to exploit their easier cooling). Lastly, small variants are also substantially less expensive than the larger cells.

Based on the above considerations, the preliminary conclusion is to procure a suite of

- 1 of VX6
- 2 of VX2

3.2.2. Sample assemblies

The sample assemblies for the PE system contains backing discs/spacers, seats, anvils gaskets and a series of centring devices as shown in Figure 4.

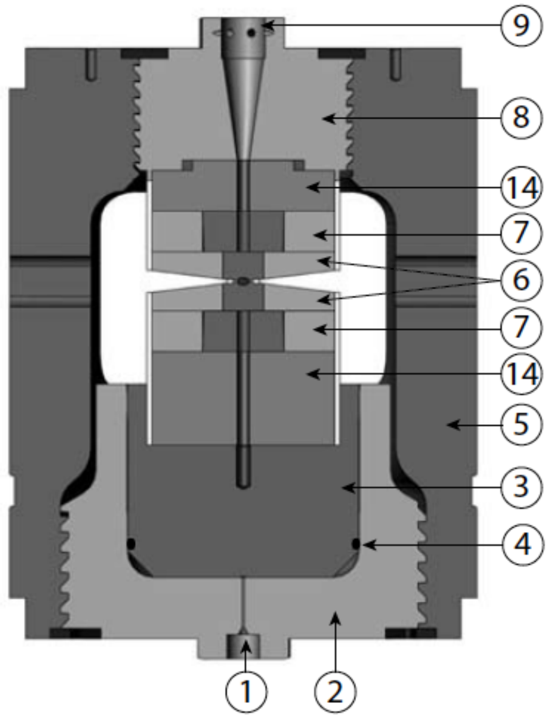

Figure 4 Illustration of PE system components. The sample assembly includes steel spacers (14), WC seats (7) and anvils (6). Also shown, but not labelled, are the gasket and sample itself and aluminium centring rings that align the various parts together.

By design, the anvils and seats are exchangeable: different materials and geometries being required for different neutron measurements. By choosing a variety of assemblies, we can maximize the versatility of the PE systems for use in the ESS pool. A further consideration is it is possible for anvils (and, to a lesser extent, seats) to fail during use and they should be considered consumables replaced at certain intervals.

3.2.2.1. Anvils

Current materials used in manufacturing anvils include several hard ceramics and sintered materials: tungsten carbide (WC), sintered diamond (SD), Zirconium-toughened alumina (ZTA) and cubic Boron Nitride (cBN). The anvils are also available in two standard profiles: single and double toroid profiles. *We note that alternative new designs of anvil are being pursued in separate projects and they are not considered here*.

Each material has its own distinct advantages from the mechanical, neutronic and cost perspectives. These properties are summarized in Table 7.

Table 7 some properties of common anvil materials

Material	Neutronic properties	Maximum pressure	Cost
WC	High absorption	10	Moderate
ZTA	Low absorption	7	Moderate
cBN	Very high absorption – beneficial for L geometry	9	moderate
SD	Medium absorption, simple background, large Bragg edges	20	high

Consultation of the baseline instrument suite (Table 4) shows that there is significant demand for longitudinal geometry, and this would be best catered for with single-toroid cBN anvils. For transverse geometry, single-toroid ZTA anvils provide the best performance due to high transmission up to 7 GPa. Lastly, for early science success, ESS should be able to match the current maximum pressure capabilities of alternative neutron sources and, therefore, double-toroid SD anvils must also be available.

In considering the exact number and type of anvils to procure, we must take account of both demand amongst the baseline first 8 instrument suite and also estimate regularity of anvil failure. This latter can be minimized by operating the anvils significantly below (say, 75% of) their maximum operating pressure (MOP). However, early scientific success at the ESS may require some operation at 100% MOP in certain cases.

Anvil type	Maximum operating pressure (MOP)	Cycles to failure 75% MOP	Cycles to failure 100% MOP
Single toroid WC	10	40	15
Single toroid ZTA	7	40	15
Single toroid cBN	9	40	15
Double toroid SD	20	20	10

The preliminary conclusion of the above considerations is that the provision of the following sets of anvils will be sufficient to support early science on the first 8 instruments at the ESS:

- Procure 4 pairs cBN single-toroid anvils
- Procure 3 pairs ZTA single-toroid anvils
- Procure 2 pairs SD double-toroid anvils

To facilitate use with H_2/D_2 gas, the binding rings for anvils should be H_2 compatible

3.2.2.2. Seats

Seats are generally robust and can be used multiple times, although failure is possible in the event of a high load anvil failure. At present, we only consider using well-tested WC designs for the ESS, both standard and conical seats (for use with the SD double-toroid anvils). *See later section on cryogenic operation of the PE cell for consideration of special thermally-insulating seats.*

Preliminary conclusion is that ESS should procure:

- 2 pairs standard WC seats for the VX6
- 2 pairs conical WC seats for the VX6
- 4 pairs of standard WC seats for the VX2

To facilitate use with H_2/D_2 gas, the binding rings for seats should be H_2 compatible

3.2.2.3. Spacers and centring pieces

Standard hardened steel spacers and Al centring rings will be procured to complete the sample assembly.

3.2.2.4. Gasket assemblies

As gaskets are consumables, we will develop the expertise to manufacture gaskets locally in Sweden.

3.3. Paris-Edinburgh gas-loading system

Gas loading is the process whereby gases are introduced to the sample chamber of the PE system at sufficient density to prevent volume collapse upon subsequent pressurization. This is critical in the case where the sample itself is gaseous under ambient conditions, and is also essential to enable hydrostatic conditions above ~7 GPa freezing pressure of liquid media. In order to achieve sufficient densities, such a device must pre-compress the gas to ~3000 bar with even higher pressures being advantageous for very-compressible gases such as He and H_2/D_2. The complete gas-loading system should be portable

The gas-loading system includes several key components:

1. A gas compressor able to generate the required pressure of gas to load the cell
2. A pressure chamber able to accommodate the sample assembly while it is surrounded with pressurized gas.
3. A driving and clamping mechanism to seal the pressurized gas within the sample volume.
4. The gas system linking compressor to chamber and enabling some other processes
5. Ancillary components including anvil clamping system and space fillers.

Commercial compressors are available to deliver the required gas pressures, while an existing prototype system for the pressure chamber and driving/clamping mechanism serves as the preliminary design for the ESS system

3.3.1. Gas compressor

Requirements for the gas compressor are:

- safely operating pressures of up to 3000 bar
- must be hydrogen compatible.
- The compressor will be multi-purpose and it should be simple to disconnect it to use in other SESs.

3.3.2. Pressure chamber and loading mechanism

The preliminary design for the pressure chamber and loadings mechanism is to duplicate an existing and well-tested design used at ISIS (shown in Figure 5).

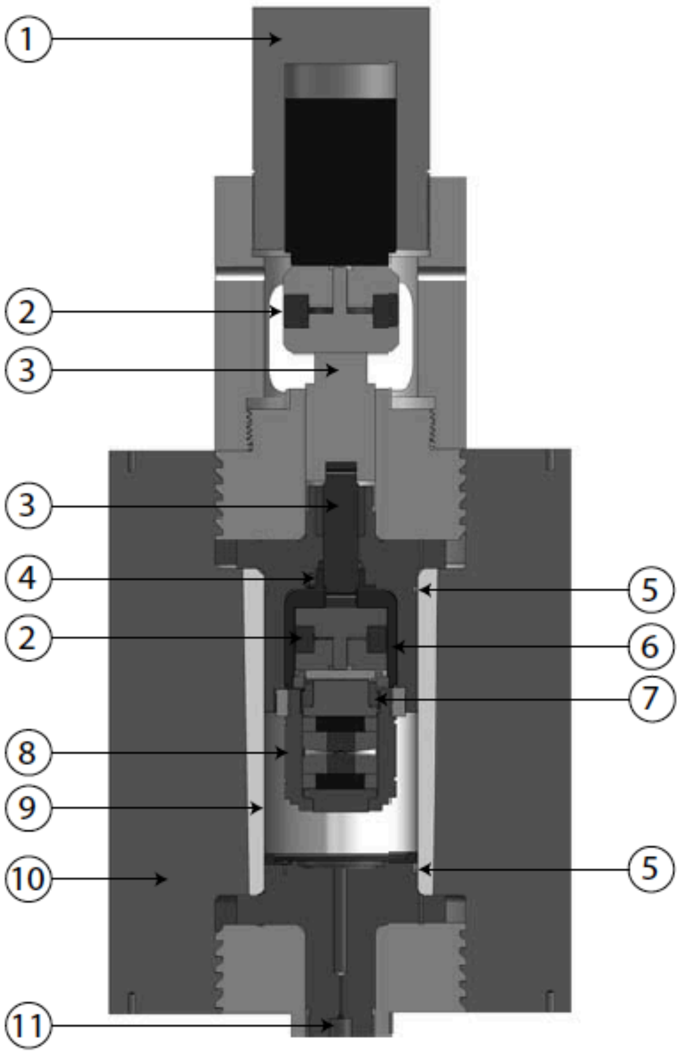

Figure 5 pressure chamber and loading mechanism

- We will examine adapting the design to ensure compatibility with other envisaged cells. This might be achieved by simple spacers and adapters to be defined during subsequent detailed design.

3.3.3. The gas system

The gas system connects the compressor to the pressure chamber, but also includes several other features including:

- **Pre-compression of the inlet gas**. Typically, high-pressure compressors will require a minimum inlet pressure to operate efficiently. Full gas cylinders will typically deliver sufficient pressure but, as they empty, operation becomes less effective and often significant residual gas is unusable. Pre-compression of the inlet gas to ~50 bar will avoid this problem.
- **Vaccum capability.** To avoid any possible contamination of the sample by moisture in the air, the system will also feature a vacuum pump, permitting full evacuation of the sample chamber prior to introducing pressurized gas
- **Gas recovery**. After loading, the chamber will contain a significant volume of compressed gas. In some cases, this gas can be extremely valuable (for example D_2). A gas recovery system will allow the used gas to be recompressed into a secondary cylinder for future re-use.
- **Safety features** Some safety features will need to be included in the design to ensure safety. Primarily, these will include the possibility of remote operation.

3.3.4. Ancillary components

In order to operate the gas-loading system, the anvils must be enclosed in load-carrying clamps. These should be able to withstand at least 7 met. Tonnes and have windows matching the VX-type PE cell apertures.

- We will require a clamp for each anvil pair
- In addition, we will need a set of machined volume filling spacers. This is necessary to minimize the volume that must be filled with the compressed gas. Distinct sets of spacers should be made that are compatible with both “standard” PE anvils, and DAC-type cells.

3.4. Sub-system 3: The Paris-Edinburgh Cryostat-system

It is a recognized priority for early science that pressure be combined with low temperature. Although a suite of cryostats will be developed by the TEFI platform at ESS, it is acknowledged that the physical size of the PE press will be incompatible with these.

The intrinsic issue with cooling the PE-press is its large mass. However, several successful approaches have been employed at other facilities. These are summarized here with some of their capabilities

Facility	System	Minimum T	Comments
ISIS	L-N_2	80	Entire cell cooled
	Double CCR	10	Only anvil assembly cooled
ILL	L-He cryostat	4	LN2 pre-cooling
SNS	CCR	10	LN2 pre-cooling planned

Additional requirements include appropriate optical windows on the vacuum vessels and transparent windows to enable aligning of the sample perpendicular and parallel to the beam.

There are several operational factors to consider in choosing between the wet (L-He cryostat) and dry (CCR) systems. These will be considered as part of a detailed design study.

Cryostat key requirements:

- Sub 10K base temperature
- Optimised cooling rate (e.g. LN2 pre-cooling)
- Optical windows for alignment
- Bulkhead feedthroughs for pressure lines

Other considerations:

- The ISIS system, which retains a warm cylinder on the cell allows oil as hydraulic fluid. If the entire cell is cooled, then special seals will be required on the PE-press to enable use of He as driving fluid.
- There is significant interest in going < 1K. A subsequent detailed design study will investigate this possibility, potentially by using clamp cells that fit in TEFI dilution fridges.

3.5. Certification requirements for PE systems

Considerations for certification are the same as those discussed in §2.4 for gas, liquid and clamp cells.

4. GLOSSARY

Term	Definition
<<YP>>	<<Yield pressure, the pressure of onset of plastic deformation >>
BP	Burst pressure, the pressure where the fully plastic state is reached
TP	Testing pressure
K	Cylindrical wall ratio = outer radius/inner radius
GPa	GigaPascal, unit of pressure equal to 10^9 Pa = 10,000 bar
MPa	MegaPascal, unit of pressure equal to 10^6 Pa = 10 bar
SES	Sample Environment System
SEE	Sample Environment Equipment
PE	Paris-Edinburgh

5. REFERENCES

[1] S. Klotz, *Techniques in High Pressure Neutron Scattering*, Taylor & Francis, Boca Raton, FL, USA (2013).

6. DOCUMENT REVISION HISTORY

Revision	Reason for and description of change	Author	Date
1	First issue	<<Malcolm Guthrie>>	<<2019-09-25>>

7. APPENDIX A

Table summarising published information from both the ILL and ISIS websites (ca Jan 2018).

Facility	type	material	max working P (GPa)	diam (mm)	height (mm)	vol (cc)
ISIS	gas	TiZr	0.56	7	40	1.54
	gas	7075 Al Alloy	0.45	7	40	1.54
	gas	Inconel	0.736	7	40	1.54
	gas	BeCu	0.736	7	40	1.54
	gas	BeCu TiZr layer(outside?)	0.8	7	40	1.54
	McWhan	Al2O3	2.5			
	BeCu clamp	BeCu	1			
ILL	clamp	TiZr	1	6	20	0.57
	clamp	BeCu	1	6	20	0.57
	clamp	TiZr	1.5	5	20	0.39
	clamp	BeCu	1.5	5	20	0.39
	McWhan	Al2O3	2.5	4	5	0.06
	gas	Al	0.1	12	57	6.45
	gas	Al	0.3	6	70	1.98
	gas	Al	0.5	16	50	10.05
	gas	Al	0.7	6	65	1.84
	gas	BeCu	0.1	12	57	6.45
	gas	BeCu	0.3	6	70	1.98
	gas	BeCu	0.7	6	70	1.98
	gas	TiZr	0.1	12	57	6.45
	gas	TiZr	0.3	6	70	1.98
	gas	TiZr	0.7	6	65	1.84
	liquid	Al	0.1	6	60	1.70
	liquid	Al	0.5	6	60	1.70
	liquid	Al	0.7	6	60	1.70
	liquid	CuBe	0.25	6	60	1.70

liquid	CuBe	0.7	6	65	1.84
liquid	CuBe	0.7	12	56	6.33
liquid	TiZr	6	6	60	1.70
liquid	TiZr	7	4	20	0.25
liquid	TiZr	7	6	62	1.75
liquid	TiZr	7	12	44	4.98