



Extraction System Report

Active Creosote Extraction (ACE) Project

15/11/22

**Electricity
Distribution**

nationalgrid

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Executive summary

An experimental extraction system, capable of accepting 2.5m and 5.0m long wooden utility poles of maximum diameter 30 cm, has been developed. This unit is designed to investigate the ability of Supercritical Fluid Carbon Dioxide, SFCO₂, (CO₂ with a minimum temperature of 32 °C, and minimum pressure of 1070 psi) to extract and transport creosote from wooden end of life utility poles, therefore rendering them non-hazardous.

The design allows the parameters of target pressure, temperature and Carbon Dioxide flow rate to be controlled, while monitoring the quantity of creosote being transported out of the pole using Infrared spectroscopy, utilising a custom made high pressure observation cell capable of a working pressure of 5000 psi.

The design has the functionality to add modifier chemicals via a high pressure HPLC (High Pressure Liquid Chromatography) pump, capable of delivering up to 25 cm³ per minute at up to 8000 psi into the carbon dioxide flow stream. The modifier chemicals have been chosen to be Propanone (Acetone) and Methanol. By adding these modifiers, there is the an increased potential of mass transfer of creosote out of the poles, by altering its solubility and surface tension.

A further design feature is the addition of a piston accumulator, which can act as an oversized (60 litre) syringe, and gives the ability to rapidly reduce the pressure in the extraction vessel, creating a pressure differential between the core of the pole and its surroundings, forcing the creosote laden CSFCO₂ to exit the wooden pole and increasing the mass transfer.

The design incorporates regulatory safety devices throughout the system including safety relief valves and bursting disks which will release any contained over-pressure.

The design also includes a refrigerated carbon dioxide storage system capable of maintain up to a maximum 1200 kg of liquid at -17 °C., on hire from IGC Engineering.

The design also ensures that all exhausts from the system are depressurised into custom made traps which stop any creosote from being discharged to the atmosphere. The outlets from these traps are then directed out of the building via pressure tested and trace heated 2 inch diameter stainless steel piping, with the heating ensuring the exhaust remains in the gaseous phase without solidifying, which is essential when a recycling plant is incorporated into the system.

The extraction vessel was constructed by Vessco engineering of Bridgend, and the pipework was installed by R&D of Neath Abbey Business Park, Neath. The exhaust pipework was installed by Control Gear of Pontypridd, who also designed and supplied the pulse system.

The overall layout and safety design was completed by Glamorgan Process Engineering Ltd.

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Extraction system overview

Liquid carbon dioxide storage tank.

As carbon dioxide is the basis of the extraction media it has to be delivered to the pumps as a liquid to ensure efficient pumping. To reduce the volume of carbon dioxide that has to be stored, it is more efficient to store it as a liquid, gaseous carbon dioxide has a volume ratio of approximately 600 litre of gas (at room temperature) for 1 litre of liquid.

There are two methods of storing, and delivering the liquid carbon dioxide:

- Cylinders
 - For the cylinder to deliver liquid, as opposed to gas, the cylinders have dip tubes installed down the inside which extend to the bottom and are connected to a cylinder outlet. Therefore, the liquid carbon dioxide has gaseous carbon dioxide above it and the pressure exerted by this gas forces the liquid carbon dioxide up the tube and out when the outlet valve is opened. These cylinders are at room temperature (generally between 10 °C – 20 °C) and have an internal pressure of approximately 50 Bar (730 psi).

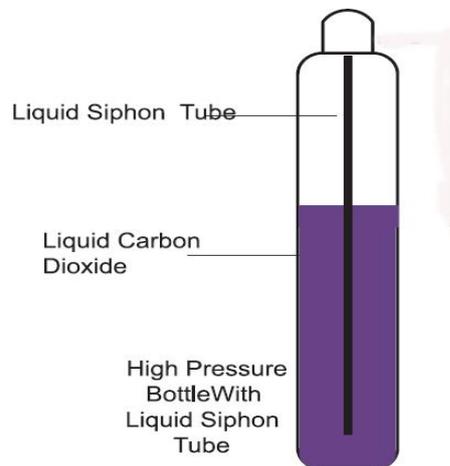


Figure 1 Liquid carbon dioxide cylinder delivery system

- Storage tanks.
 - These are refrigerated tanks, chilled at approximately -17°C. When stored and maintained at this lower temperature, the internal pressure within the tank is approximately 260 psi, more than two and a half times lower than the cylinder pressure. Further details are explained in the later sections.

The tank is approximately 2 meters in height and diameter and is insulated to reduce heat transfer and has associated fill and supply lines used to top up and feed the liquid to the pumps. The main tank can be seen in figure 2.



Figure 2 Liquid carbon dioxide storage tank

Before filling, the tank must be cooled and all the air displaced with carbon dioxide, to ensure that no ice forms and blocks the outlet pipes. The tank is capable of storing up to 1200 kg of liquid CO₂ and has an integrated refrigeration system to maintain an internal temperature of -17 °C and a pressure of 280 psi. It has built in overpressure system, which allows the liquid carbon dioxide to controllably vent in the case of refrigeration failure as seen in figure 3.

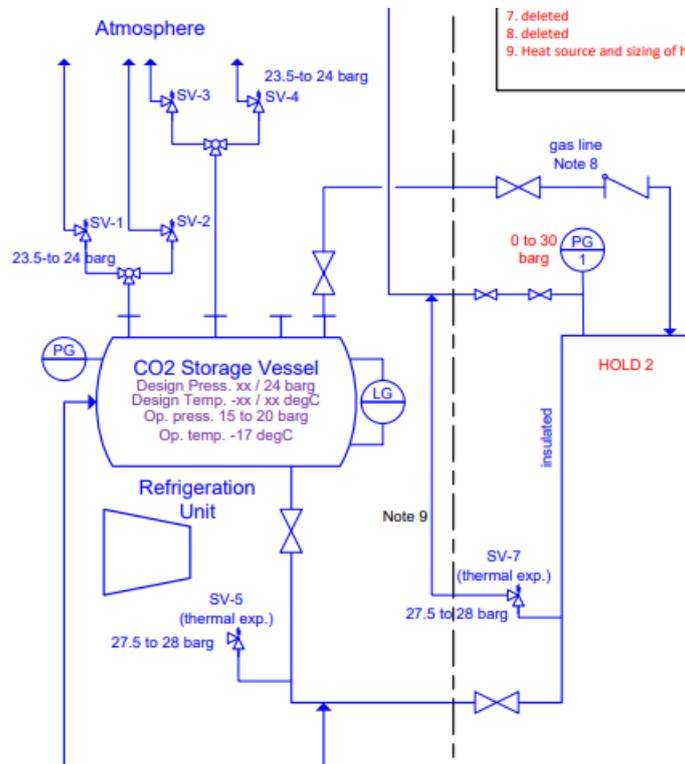


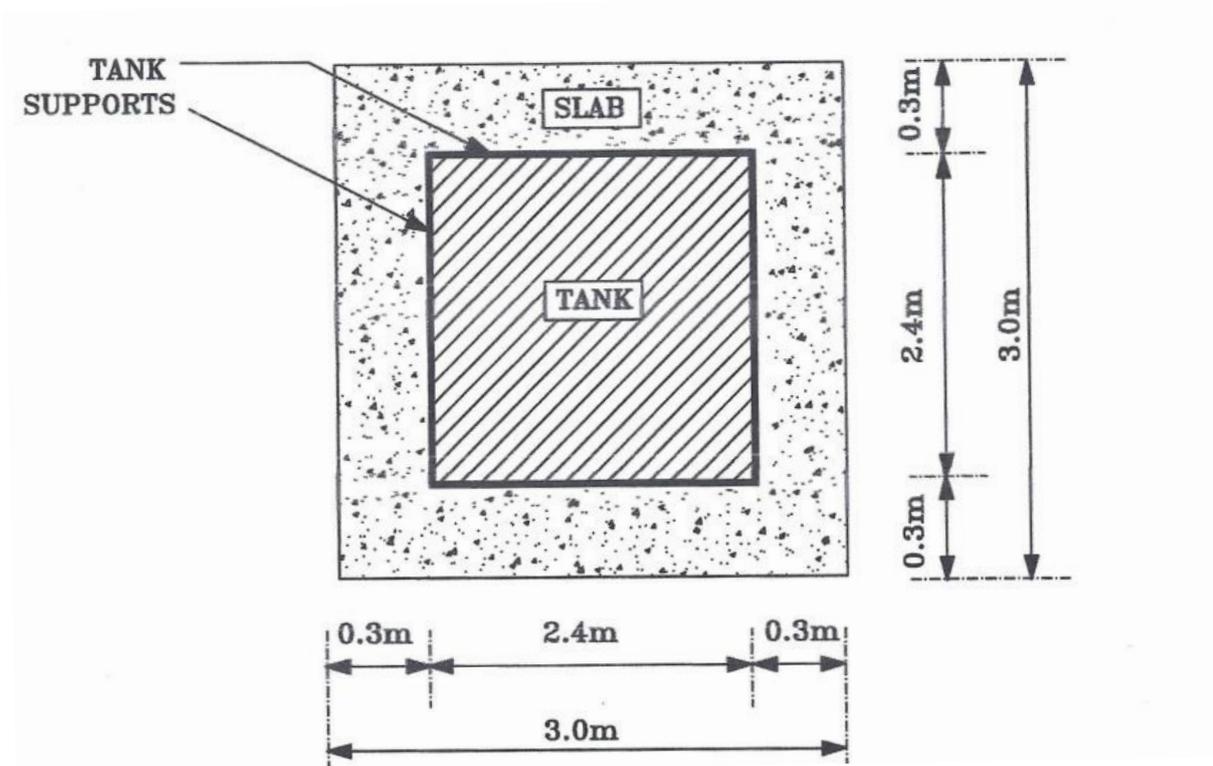
Figure 3 Storage tank safety pressure relief diagram

In case of failure of the refrigeration system, venting lines, with integrated safety relief valves, (labelled SV-1 to SV-4 and set at 23.5 Bar g which is below the failure pressure of the tank) are

attached to ensure the safe release of gaseous carbon dioxide. Attached to the insulated feed line is an isolation valve which is cryogenically rated to withstand the low temperatures of the liquid carbon dioxide, safety valves (SV-5 and SV-7) are placed within this line to vent any trapped carbon dioxide.

Because of the high cost of purchasing a storage tank this item will be leased from IGC engineering, which are an engineering company based at Chorley, Bolton, who specialise in liquid carbon dioxide storage tank supply.

The tank will be situated at the rear of the unit and has to be positioned on the prepared concrete base, surrounded by appropriate safety fencing. The vent lines have to be extended to roof level to ensure adequate dispersal. Figures 4a and 4b show the design process of the concrete slab.



**Weight of Tank Full = 4 Tonnes (40kN) Unfactored
uniformed distributed over 4 No Supports**

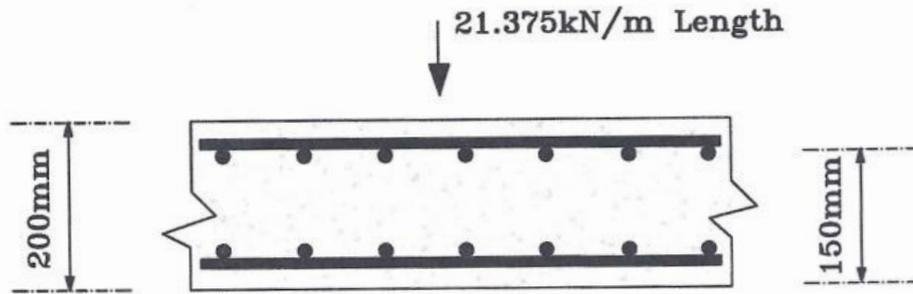
UD Load from Tank on Slab

$$= \frac{40\text{kN}}{3\text{m} \times 3\text{m}} = 4.5\text{kN/m}^2 \text{ (} 5.0\text{kN/m}^2 \text{ Say) (Unfactored)}$$

UD Load from Slab

$$\text{Floor Slab } 200\text{mm} = 5\text{kN/m}^2 \text{ (Unfactored)}$$

Figure 4a Storage tank safety pressure relief diagram



Factored Load on Slab/m Width 3m Strip
 $= 7.5 + 6.75 = 14.25\text{kN/m}^2$
 Total Load on Slab $= 14.25 \times 3.0 = 42.75\text{kN/m}$
 $R_1 = 21.375\text{kN/m}$ $R_2 = 21.375\text{kN/m}$
 \therefore KE Load $= 21.375\text{kN/m}$ (Factored)
 Effective Depth $d = 150\text{mm}$
 \therefore Shear Stress/m $= \frac{21.375 \times 10^3}{1000 \times 150} = 0.1425\text{N/mm}^2$
 Percentage Reinforcement $= \frac{393 \times 100}{1000 \times 150} = 0.26\%$
 Allowable Shear Stress $= 0.51\text{N/mm}^2$ Satisfactory
 \therefore 200mm Thick Slab with A393mesh Top and Bottom Satisfactory

Figure 4b Design details for concrete slab

Liquid carbon dioxide pumps



Figure 5 Liquid carbon dioxide pumps

These are specifically designed piston pumps by CAT pumps (Minneapolis, USA) who have a reported 25 years' experience in the field of liquid carbon dioxide pumping. In order to pump cold liquefied gases without cavitation in the pump heads, cavitation is the formation of bubbles in the liquid which grow and then collapse, which reduce the compressibility of the liquid leading to inefficient pumping and also possible corrosion. To reduce cavitation the pump heads are initially cooled, before pumping, with liquid CO₂ being allowed to flow through them for approximately 30 minutes which reduces the temperature of the pump heads to -17 °C.

The pumps have a limited suction (pump inlet) pressure range, in this case between 240 and 300 psi, this means that supplying carbon dioxide to the inlet side of the pump at pressures higher than 300 psi can seriously damage the pumps. Therefore, referring back to the carbon dioxide storage pressures, cylinders are stored at room temperature and 50 Bar 730 psi (higher the temperature the higher the pressure) while carbon dioxide stored at -17 C has a pressure within the prescribed input pressure range for the pumps, therefore supplying carbon dioxide from cylinders, which would have been easier logistically, was not an option.

The pumps take the liquid carbon dioxide from the inlet (suction) pressure of 280 psi and raise to our target pressure of 1200 psi. The smaller pump will offer more control, and the larger pump higher pump up rates, the time (rate) to achieve the target carbon dioxide pressure.

The pumps are controlled using an Invertek Optidrive variable frequency controller which allows the pump speeds to be altered between 20% to 100% capacity, of their highest rate. Prior to extraction the amount (flow rate) of carbon dioxide to achieve extraction can only be estimated from earlier work, the ability to achieve fine control over the amount of carbon dioxide required to flush the extracted creosote from the pole is essential.

Heat exchangers.

For the liquid carbon dioxide to become a supercritical fluid it has to achieve the critical conditions of a minimum pressure of 1072 psi and a minimum temperature of 32 °C. The CO₂ leaves the pump at -17 °C, to reduce cavitation, and has to be heated to a minimum of 32 °C. In order to achieve this temperature rise, a heat exchanger has to be placed between the pumps and the extraction vessel. The heat exchangers are constructed from seven lengths of six meter ½ inch diameter high pressure stainless steel tubing, constructed by R&D engineering. They will be connected together before having heating tape attached which will allow temperature control and can be seen in figure 6.



Figure 6 Extraction System Heat Exchangers

As the liquid carbon dioxide travels through the heat exchangers, its temperature is raised because of the differential temperature between the liquid and the heated tube. In addition, liquid carbon dioxide cools on expansion due to the Joules – Thompson effect, which is the cooling, on expansion from a high pressure region to a low pressure region, and increases with increasing pressure differential. This occurs as it expands out of the pump, and also the back pressure regulator. Because of this, solid CO₂ can form (dry ice) which can cause temporary blockages.

For this cooling to be minimised as well as positioning the heat exchangers between the pump and the extraction vessel, all the pipework will be fitted with trace heating and lagged to maximise the

heat transfer into the liquid. Maintenance is minimal, but will involve regularly checking the unions to ensure no leakage is occurring, any solid or liquid carbon dioxide will become a gas on standing, to ensure this does not produce a pressure which could cause damage safety relief valve SV-10 is placed in this delivery line.

Extraction vessel

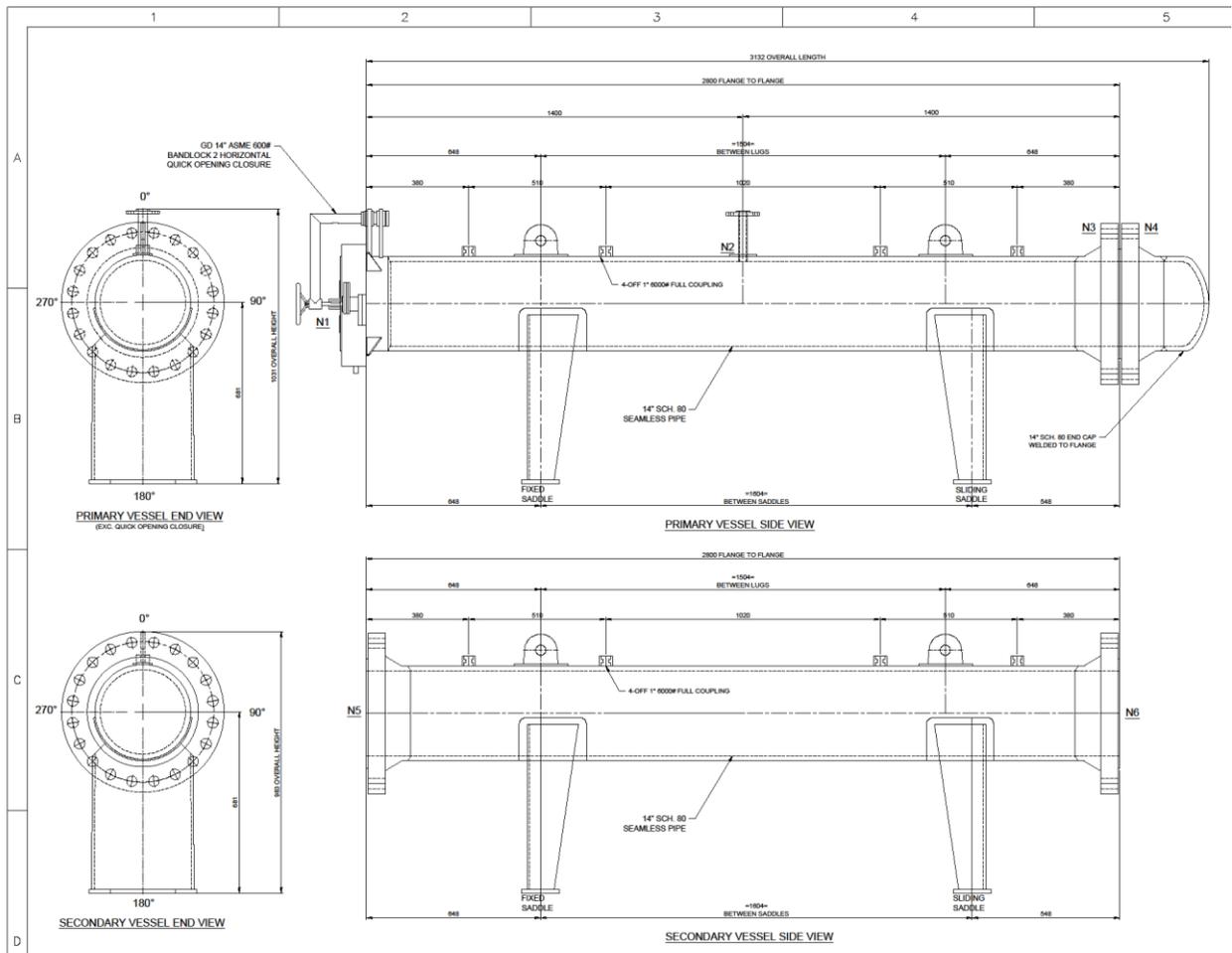


Figure 7 Extraction vessel design

Before design and construction of the vessel, the diameter of poles capable of being inserted into the extraction vessel was determined, with most urban poles having a diameter of between 25 and 28 cm which gave an indication of the required vessel diameter.



Figure 8 Extraction vessel showing the Bandlock 2 quick opening enclosure and prior to the application of heating tape, insulation and aluminium shell

The extraction vessel is constructed from seamless 14 inch Schedule 80 steel pipe with a wall thickness of 11.3 mm and is designed to operate at 1200 psi and has been hydrostatically pressure tested to 137.9 Barg (2000 psi). It has an internal diameter of 300 mm and an internal length of 2800 mm flange to flange, resulting in an internal volume of 287 litres or 0.287 m³. The total weight of each extraction vessels is 1109 kg.

Each section has 4 inlet/outlet ports which are 1 inch diameter NPT threaded. These ports will be used as:

- x1 inlet port for the pressurised carbon dioxide,
- x1 for the pulse system,
- x1 for the modifier inlet,
- x1 outlet port to exhaust the carbon dioxide via a back pressure regulator.

One of the vessels has a fifth port to allow a bursting disk, or safety relief valve, to be fitted. This is a safety device which has its pressure limit set at 96.5 Barg with a tolerance of 10% and is designed to release the pressure in case of a malfunction before reaching the vessel upper design pressure, and vents the carbon dioxide to the exterior of the building via a 2 inch vent pipe.

After construction the vessel was fitted with trace heating, which has a heating capacity of 66 watt per meter as seen in figure 9. In order to protect the trace heating from being damaged, an aluminium casing has been designed and applied to the extraction vessel as seen in figure 10.



Figure 9 Extraction vessel with heat tracing (red) applied



Figure 10 Completed extraction vessel

The vessel is fitted with a fast opening door at one end and a domed flange on the other fitted with 20, 1 and 3/8 x 12 inch long bolts to ensure adequate torque on the metal seal fitted between the vessel and the domed flange. The Bandlock 2, fast entry door is made externally by Celeros, and allows one person to open it as opposed to opening a domed flange end and its 20 location bolts. The vessel is elevated on legs to a height of approximately 600 mm.

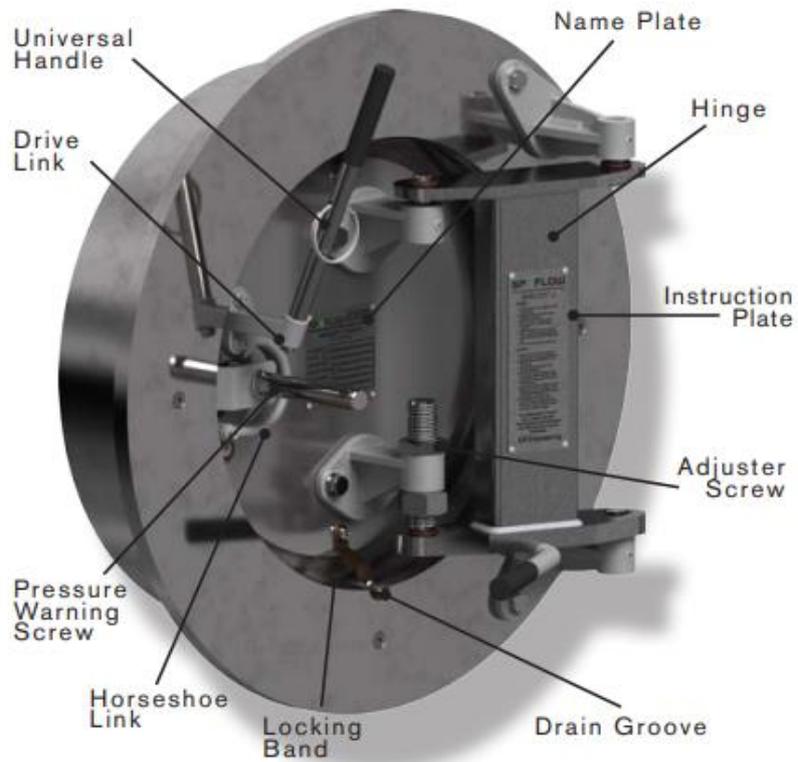


Figure 11 Celeros Bandlock 2 rapid opening enclosure

Back pressure regulator

The spring loaded back pressure regulator is fitted to the outlet of the extraction vessel and maintains the target pressure within the vessel.

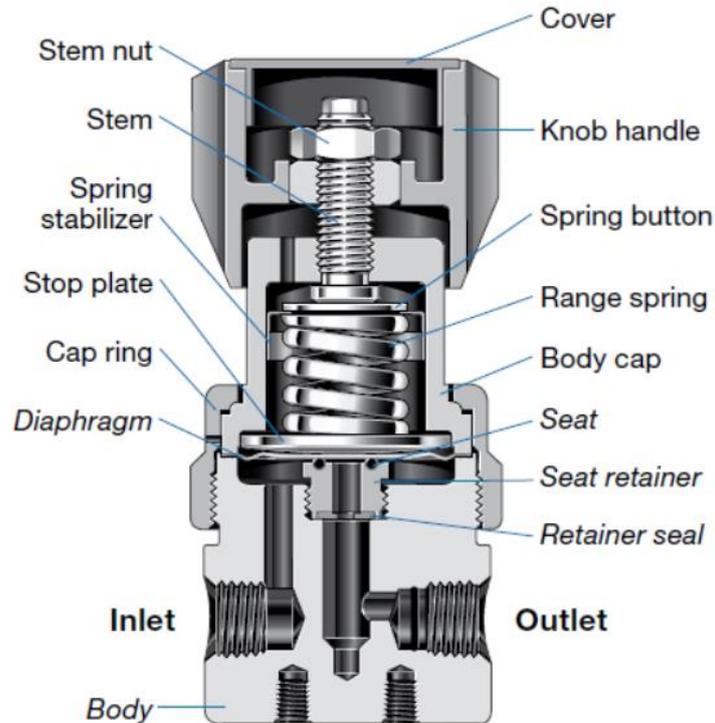


Figure 12 Swagelok spring loaded back pressure regulator

The regulator remains closed until the target pressure is reached, it then opens to maintain that target pressure while clean CO₂ is flushed through. Without a back pressure regulator, the pressure in the vessel would continue to rise while carbon dioxide was still being pumped in.

It is a spring loaded valve and has a rotatable handle (Green) which can be altered to change the set target pressure. While carbon dioxide is depressurised through the valve, there is an associated Joules-Thompson cooling effect, because of the pressure differential across the regulator, and so trace heating has to be applied to the valve, and pipework, to ensure no dry ice blocks the valve ensuring efficient venting.

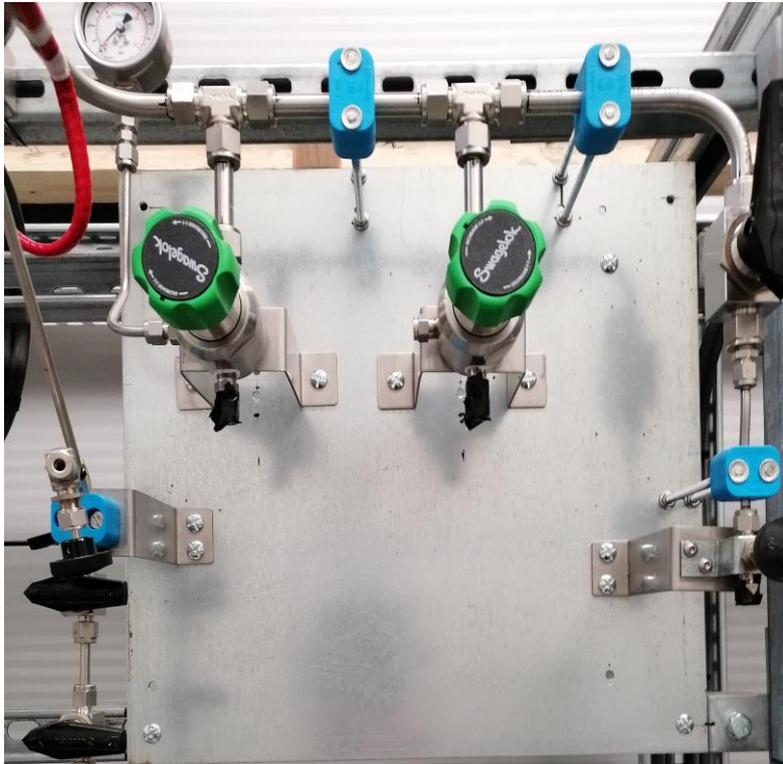


Figure 13 Swagelok high pressure back pressure regulators

Safety relief valves

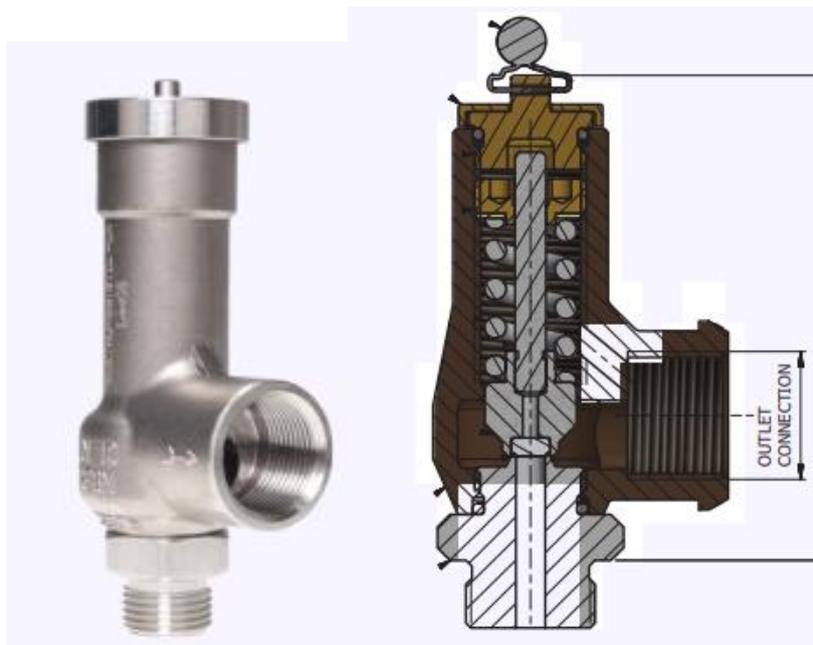


Figure 14 Seetru Safety relief valves

Due to the high pressures involved in the extraction process it is mandatory that pressure relief valves and bursting disks are placed at strategic points throughout the system. These safety relief valves are set to release the pressure before the pressure rating of the fittings, and the extraction

vessel, are reached the spring loaded valve is opened and the pressure released. When the pressure reaches 15% below that of the set value the valve will reseal. The output port of the valves are connected to a vent line which channels the vented gases to the exterior of the building.

Fike Bursting Disk

In addition to the pressure relief valve, the system will be fitted with bursting disks. As indicated in the title the bursting disk is positioned in a holder and will burst and release the pressure before permanent damage is done to the system. One is fitted to the extraction vessel and another to the traps. The outlet from the bursting disk must be connected directly to the heated exhaust.

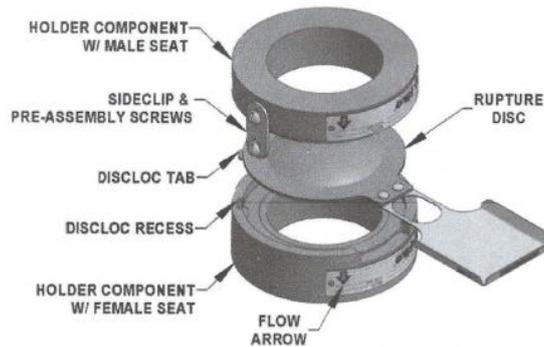


Figure 15 Bursting disk holder



Figure 16 Bursting disk

Creosote Traps

The exhaust gases from the extraction process and any overpressure issues, carbon dioxide and any extracted creosote, have to be vented via a pair of 1 metre long 15 cm diameter tubes constructed from Schedule 40 steel pipe with a wall thickness of 7.1 mm.

Remembering that the carbon dioxide only acts as a solvent for the creosote while it is in the supercritical fluid phase, on depressurisation it is no longer a supercritical fluid, and so no longer capable of having creosote dissolved in it.

The first trap is where the depressurisation of the extract will be maximised, and is where the majority of the creosote should be deposited, the second will be partially filled with activated carbon to adsorb any remaining creosote to ensure it isn't vented to the atmosphere. Both traps will have flanges fitted to allow removal of any extract. Even though these are vented to the atmosphere, to ensure no build-up of toxic gases (CO₂ and creosote), the first trap will also be fitted with a pressure transducer to measure the pressure in the trap. This will be used to incorporate into the design of the recycling plant.



Figure 17 Dual Exhaust traps

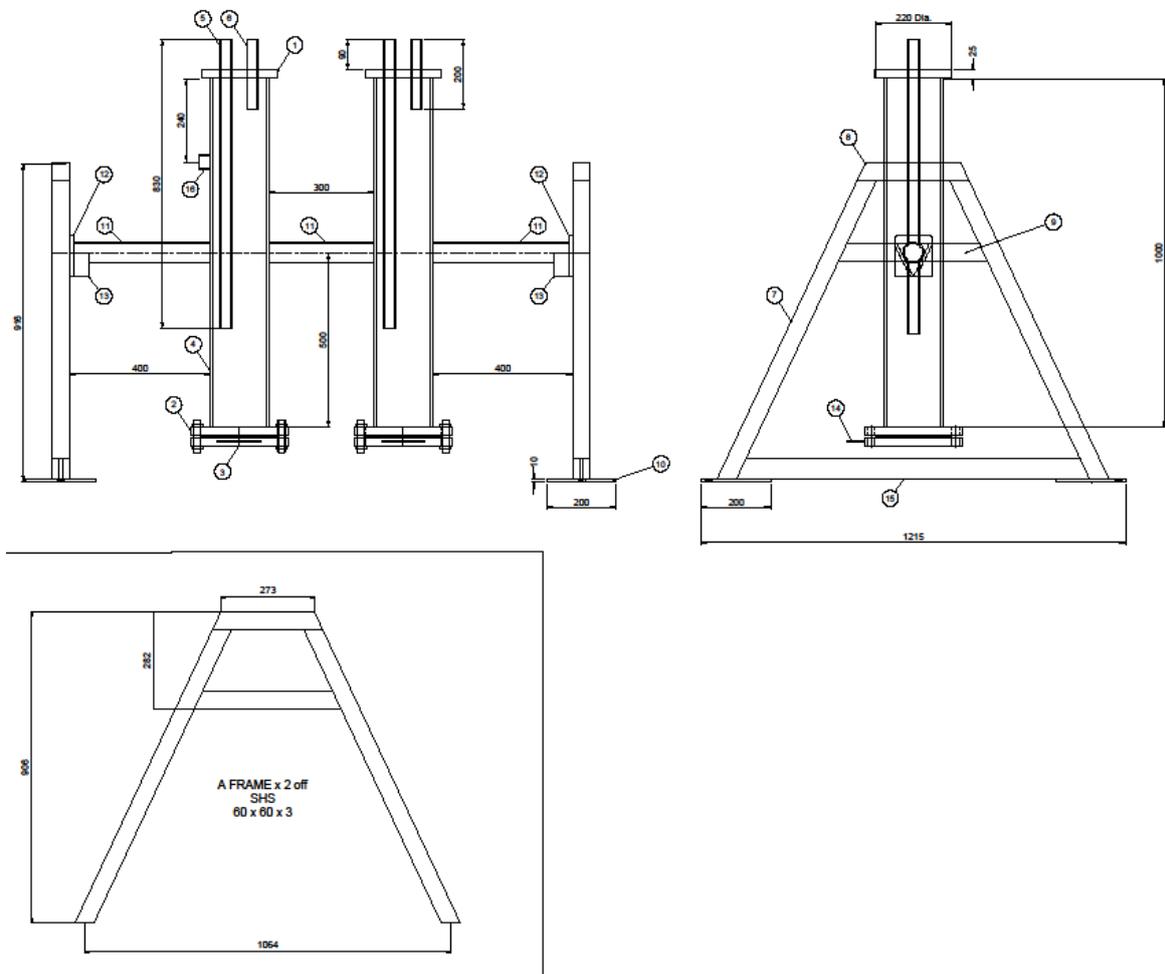


Figure 18 Exhaust traps diagram

