MICE HYDROGEN SYSTEM

R&D Test Report

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Contents

Figures

1 Goals

The MICE requirement for the hydrogen system is to safely store and deliver enough hydrogen gas to the AFC to fill the absorber module with LH2.

As operation of the hydrogen system requires exclusion from the Hall in a similar fashion to beam operations, the R&D programme was limited in scope to avoid excessive impact on the overall MICE schedule. The stated goals of the hydrogen system test programme were as follows:

- Safely charge the hydride bed for the first time
- Successfully complete Hydrogen Fill and Empty sequences
- Prove that liquid hydrogen was produced
- Characterisation and optimisation of the major system components
- Leave the system in a safe state for decommissioning

2 Test programme

2.1 Hydride bed charge

The hydride bed charge procedure was the first time that the bed had been opened since delivery in 2006. Predictions on charge time were based on test data provided by the manufacturer, Treibacher. The Treibacher data suggested that the intended charge of 16,000 litre @ s.t.p. (equivalent to 2 standard bottles of H2) would take around 6 hours. In the Treibacher setup, the bed was cooled to -5˚C and used an H2 pressure of 1.6 bara.

MICE used a similar setup but with a lower temperature in the bed of -15˚C. The charge completed in 2 hours, representative of this lower temperature. No data is presentable from this sequence as the charging line does not incorporate a flow meter; the only indication of progress was a visual reading of the bottle pressure.

It should be noted that 16,000 litres does not represent a full charge; the bed’s capacity is 32,000 litres with the right charging conditions. The MICE absorber will eventually require at least 20,000 litres (equivalent to 22 litres of LH2 plus the buffer tank volume). However, a charge of this capacity would have required an intermediate bottle change and so the decision was made to run the tests with a partial charge.

2.2 Hydrogen fill (first attempt)

The first hydrogen fill sequence started with the cryostat purged and at room temperature. Figure 1 shows pressure and temperature plots of the sequence progression, as well as a trace of the control valve operation.

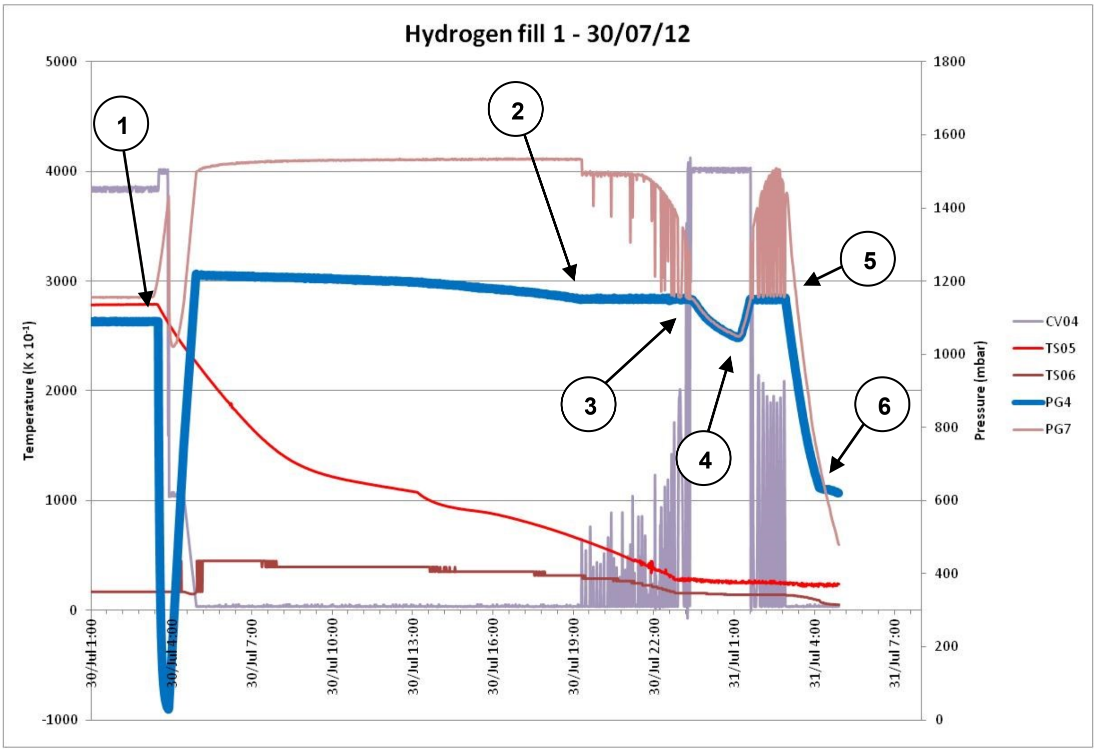

Figure 1: Hydrogen Fill 1

1. **Sequence starts**. The dip in pressure just after this point represents the helium from the purge sequence being pumped out and the system being repressurised with hydrogen. The cooldown from room temperature to condensing temperatures can be observed from the TS05 and TS06 traces. Both sensors represent the temperature of the condensing pot heat exchanger; TS05 is a high-temperature PT1000 and TS06 is a low-temperature Cernox.
2. **Control valve loop starts**. At this point, the hydrogen in the cryostat has cooled enough to reduce the pressure in the buffer tank to 1150mbar, at which point the CV04 control loop initiates. This maintains the pressure at 1150mbar. The back pressure from the hydride bed (PG7) can be seen dropping as the control valve opens.
3. **Bed pressure drops.** As the bed evolves hydrogen, a higher temperature is required to continue evolving at a constant pressure. During this sequence, the heater-chiller was under manual control and the rapidly dropping bed pressure was not immediately attributed to an insufficient bed temperature. This led to the bed pressure dropping below the 1150mbar required to maintain the pressure in the buffer tank. As such, the control valve opened fully and the pressure throughout the system dropped. The 20W heater was ramped up to 100% duty cycle in an attempt to arrest the pressure drop but did little to slow the process.
4. **Heater-chiller setpoint increased.** The bed temperature was finally increased, resulting in the system pressure recovering and the CV04 control loop resuming.
5. **Sequence ended.** At this point, TS06 was showing 14.1K. As the freezing point of hydrogen is 14K, the data was (incorrectly) interpreted as showing the system was icing up. As such, the fill sequence was prematurely ended and put into ‘operational mode’. This closed the control valve, put the hydride bed into ‘absorb mode’ and opened a relief line to the bed.
6. **Freezing occurs.** As the supply of warm gas from the hydride bed had been cut off, the self-regulating heat flow into the heat exchanger from hydrogen condensation slowed. This caused the heat exchanger temperature to drop steadily, as can be seen from the trace of TS06. This resulted in clear evidence from the level sensors of frozen hydrogen on the heat exchanger. At this point, an empty sequence was run to return the hydrogen in the system to a gaseous state and provide the basis for the start of a new fill sequence.

For 7 days following this point, no sensor data exists. This is because resetting of the data logger results in stored files being overwritten and if a manual download of the data prior to resetting the system is not carried out, the data is lost. The problem has since been rectified by archiving the PLC process variables using the global MICE EPICS system.

2.3 Hydrogen fill (second attempt)

The empty sequence started at the end of the last section was aborted once the temperature readings suggested that all solid/liquid hydrogen was boiled off. This caused some issues with the control system due to some bugs in the ‘abort’ command. However, the necessary conditions were forced and a new fill sequence was started. During this, the hydride bed temperature and heater duty cycle settings were manually controlled.

Initially, the 50W heater duty cycle was adjusted to maintain the heat exchanger temperature at around 18K. However, this did not result in condensation. The heater duty cycle was gradually decreased from 70% to 40% until TS06 read around 14K, at which point it was believed that hydrogen would begin to freeze on the heat exchanger. Even at this temperature, there was little evidence of condensation, so the temperature was allowed to drop further. At a heater duty cycle of 20% (10W equivalent), the condensation rate eventually increased and TS06 stabilised at around 11K. The system was allowed to operate in this setup for around 24 hours, with the hydride bed temperature being increased as required.

Unfortunately, during a shift period without an expert present, the bed pressure began to drop. This was not registered by the shifter present at the time and the temperatures and pressures dropped in the same manner as before, again resulting in hydrogen ice building up on the heat exchanger. However, the system was recovered shortly after and the fill sequence completed with approx 10,000 litres of gas having flowed from the bed. The system was then left in 'operational mode' with the heater duty cycle set to 50% to stop further condensation.

2.4 Hydrogen empty

Prior to starting the first full hydrogen empty sequence, there was some concern over the bed's ability to balance absorption rate with boil-off rate to maintain a safe pressure. As such, a conservative approach to implementing the control loop was taken.

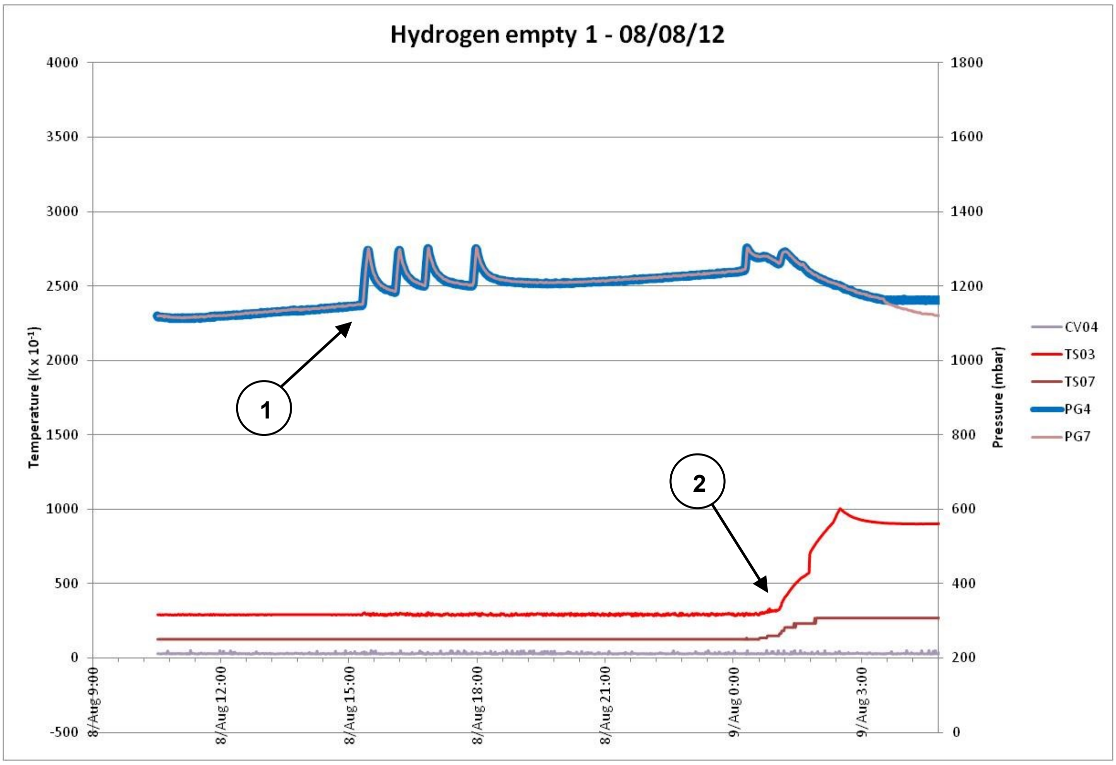

Figure 2: Hydrogen Empty

1. **Absorption rate characterised**. An initial pressure setpoint of 1100mbar was chosen, at which point the absorber heater would switch off. This worked initially but as hydrogen was taken up by the bed, mere cooling was no longer sufficient to force absorption. The pressure setpoint was

incrementally raised to 1400mbar, at which point the bed absorbed the remaining boil-off.

2. **Boil-off complete.** Up to this point, the temperature of the absorber, TS07, was stabilised as the hydrogen boiled off. With the boil-off complete, the temperature immediately began to rise. The divergence in pressures a few hours later indicates the bed being isolated at the end of the empty sequence.

2.5 Hydrogen fill (third attempt)

For the second hydrogen fill sequence, a temperature control loop was implemented, using the heaters to maintain a set temperature on TS06. Manual control of the heater-chiller was still required but, unlike previous sequences, the non-expert shifters were instructed to monitor the system and make adjustments accordingly.

During this sequence, the flow meter total registered around 12,000 litres. The remaining 4000 litres (the bed was originally charged with 16,000 litres) can be accounted for via the following:

- 1000 litres – H2 in buffer tank at start of fill sequence
- 1000 litres – 2 helium purges with 500 mbar present in the 1 m^3 buffer tank
- 2000 litres – unevolved from bed due to conservative temperature cap

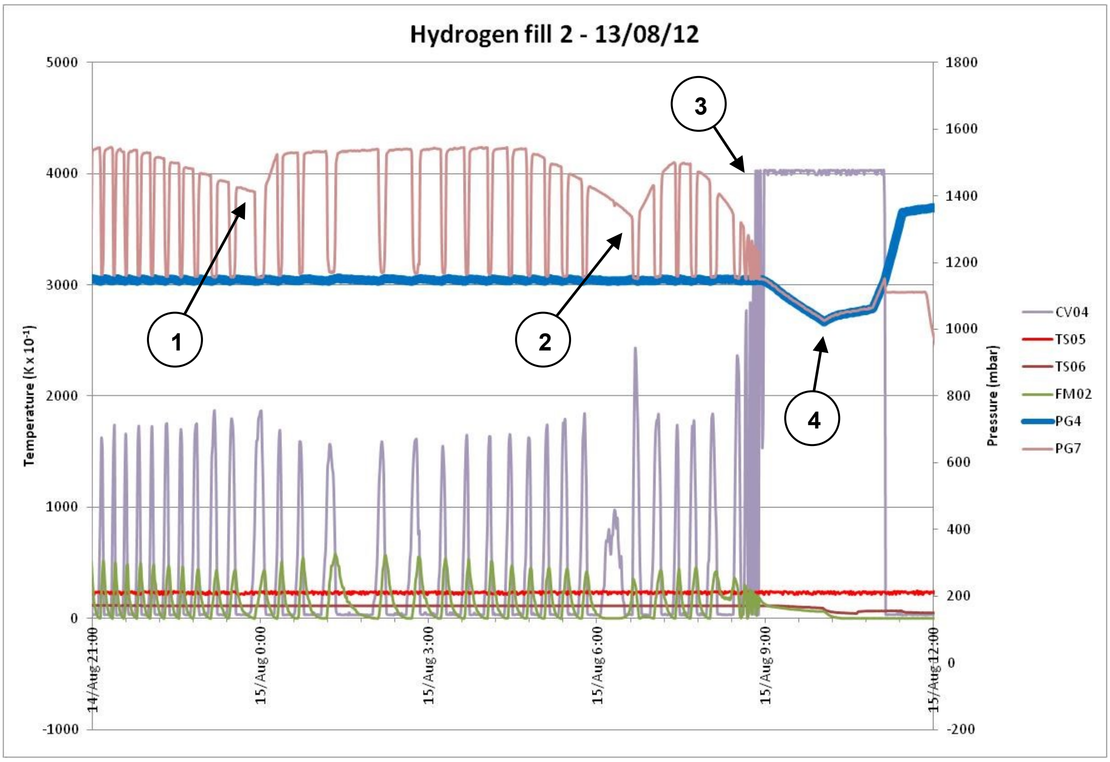

Figure 3: Hydrogen Fill 2

1. **Heater-chiller adjustment.** This point shows an example of the heater-chiller setpoint being adjusted in response to a decrease in pressure.
2. **Heater-chiller temperature maxed.** At this point, the heater-chiller was set to its high point of 60˚C. This is not the maximum capability of the system but merely a conservative cap based on the hydride bed specification.
3. **Bed pressure decreasing.** In a similar pattern to the first hydrogen fill sequence, the bed pressure can no longer satisfy the pressure setpoint for CV04 to close. However, with the temperature control loop on the heat

exchanger, there was less anticipated risk of freezing and so the system was allowed to proceed in an attempt to maximise the output from the bed. The trace from FM02 can be seen slowly dropping from this point on.

4. **Freezing reduces performance.** This point appears to show icing of the heat exchanger, although in a more controlled fashion than previous fill sequences. This may be due to the flow from the bed dropping off to a point where the heat flow into the heat exchanger from condensation is no longer sufficient to regulate the temperature, even with the heater on. As such, the temperature drops and hydrogen plates on the fins of the heat exchanger. This insulates the heat exchanger from the hydrogen atmosphere, halting any further freezing or condensation and as such the pressure begins to rise until the sequence is ended.

2.6 Hydrogen empty

The subsequent hydrogen empty sequence ran as expected, with no significant alterations to that described in Section 2.4. All liquid was boiled off within approximately 16 hours.

It should be noted that during this empty sequence, the hydrogen detector in the vent line slowly rose. The pressure in the system was held at 1400 mbar, below the pressure relief valve rating of 1500 mbar. However, it appears that the flow of cold, boiled-off gas may cause a slight leak past the valve. The leak also seems to accumulate in the downstream vent line, suggesting that the nitrogen flow rate of 2 l/min is insufficient to purge the line. When the nitrogen flow was increased, the hydrogen detector reading briefly rose to 45% LEL before falling again, giving a clear indication of a substantial volume of stagnant hydrogen.

2.7 Hydride bed empty

Despite the design philosophy of the system stating that a charged hydride bed constituted a ‘safe’ system, the length of time between the R&D programme finishing and any AFC hydrogen tests was judged to be sufficient to justify returning the bed to an inert state.

The first step was to run a standard helium purge of the buffer tank. The bed temperature was then set to ‘evolve mode’ and the buffer tank filled with hydrogen via the bypass line. The pumping line was opened and the hydrogen throttled into it via the control valve to protect the pump. The bed temperature was gradually ramped up. Once the bed reached its maximum temperature, the pressure began to slowly drop. On reaching 5 mbar, the pump was isolated temporarily and the bed pressure subsequently rose to 300 mbar. This is because, at a constant temperature, the evolution rate depends on pressure. The pump draws hydrogen off the bed but when isolated, the pressure rises to a point where evolution stops. The bed was pumped on for a further 12 hours, decreasing the zero-evolution pressure to around 100 mbar.

At this point, a 3 bar argon blanket was applied and the bed was isolated.

3 Component characterisation

3.1 Heater-chiller

The heater-chiller unit suffered some problems during commissioning due to a underestimation of the volume of the hydride bed coolant circuit. This led to further problems during the R&D tests as the unit was topped up to ensure sufficient liquid level at its coldest state. However, the coolant expansion between this point and the maximum evolution temperature resulted in several litres overflow from the unit. This results in low-level warnings when the system is then reverted to evolution mode, requiring manual topping up of the level.

- ***Retrofitting of an external expansion tank to counter this is proposed.***

Independent of any effect from reactions in the hydride bed, the heater-chiller has a step change response time of around 10-15˚C/hour. This is too slow to reliably satisfy the design condition of automatically sending hydrogen back to the bed in the event of a failure.

- ***It may be advisable to reconsider whether venting hydrogen overpressure is a preferable default reaction to a fault scenario.***

3.2 Hydride bed

As described in Section 2.1, the first-time charge of the bed took less time than expected due to the charge being only partial, as well as a lower starting temperature.

Although the R&D programme was not a comprehensive test of the hydride bed, several aspects of its performance became evident. Firstly, the dependence of the rate of evolution on pressure increases in sensitivity as the bed empties and vice versa for absorption. This is particularly important during the hydrogen empty sequence, where the pressure cannot be increased beyond the relief pressure of 1500 mbar, lower than the pressure used in the charge sequence. As a result, each empty sequence and subsequent purge will result in significant quantities of hydrogen being vented to atmosphere, meaning regular top-up charges will be necessary.

- ***These regular charges may negate the benefit of fewer bottle operations when using a hydride bed instead of filling directly from a bottle.***

The manufacturer states that a fully charged bed will sit at approximately 3.5 bar at room temperature. As the system's pressure gauge was downstream of the regulator, this was impossible to verify.

- ***A pressure gauge upstream of the regulator is a recommended retrofit to the system.***

The bed's nominal capacity is 32,000 litres. MICE requires a minimum of 20,000 litres. As it comfortably absorbed 16,000 litres during the charge sequence and subsequently evolved 14,000 litres of this during the final fill sequence, there is no reason to believe the bed will fail to meet the MICE requirements.

3.3 Cryocooler and heaters

Hydrogen liquefaction requires a stable temperature of between 14K - 20K. The cryocooler model used is rated to 4K, at which hydrogen freezes and could potentially block the fill and return pipes. It was initially anticipated that the 20W and 50W heaters on the heat exchanger would need to be used to prevent this happening. However, the heat flow in from a high condensation rate appears to be sufficient to regulate the temperature. It appears that the heaters only become necessary when the hydride bed evolution rate drops as it approaches the empty state.

Nevertheless, a control loop which is tied to the temperature of the heat exchanger is utilised throughout the hydrogen fill sequence. A setpoint within the theoretical temperature range does not result in significant condensation; it was found that a setpoint of around 10K was reasonable.

3.4 Ventilation system, purge gases and bottle cabinet

While connecting hydrogen bottles, an inadvertent hydrogen leak was caused. The system response was as expected and a near-miss report filed for the incident.

- ***General recommendations include having non-sparking specialist tools tethered in the bottle cabinet and reviewing the connector materials (consider brass bottle connector)***

During the fill sequence, the hydrogen level in the vent line approached 50% LEL. It is suspected that this is due to the N2 flow through the line being insufficient to clear any leakage through the relief valve.

- ***Increasing the nominal N2 flow rate from 2 l/min to 5 l/min may solve this.***

3.5 Temperature sensors

As described in Section 3.3, a heater control setpoint temperature within the theoretical condensation range results in minimal condensation. It is possible that this is due to either faulty temperature sensors or a thermal gradient between the sensor location and the condensing surface.

The sensors in question are Lakeshore Cernox models, generally considered to be reliable if calibrated correctly. A small error was found in the calibration for one but this made little difference to the absolute reading. However, it was noted that the PLC voltage range is optimised for helium temperatures, rather than hydrogen. Higher temperatures result in smaller resistance changes in the sensor and subsequently, the PLC resolution in the range of interest to only 4 or 5 data points. General trends are still evident but detail is lost.

- ***A voltage amplifier should be added to the control rack to increase resolution in the temperature range of interest.***

Despite the lack of resolution, the absolute values remain anomalous to expectations. Although not certain, it is most likely that this is due to the physical location of the sensors with respect to the condensing surface. As the R&D instrumentation is unique to the test cryostat though, further analysis is without merit.

3.6 Level sensors

The three level sensors in the test cryostat comprise of up to 7 simple carbon resistors arranged on a fibreglass rod. When covered with liquid, an individual resistor will stop responding to the change in ambient conditions associated with the control valve operation. As such, the sensors do not give an instantaneous level reading as such but the level can be determined by interpretation the trend over a period of several hours.

Due to the daisy chain wiring method of the sensors, a failure in the IS barrier could result in the IS power threshold being exceeded. As such, only one resistor on each sensor can be energised at any one time. This severely limits the functionality of the sensors to the extend where the sensors where virtually ignored during the R&D programme, particularly for their primary purpose as this was more easily determined from the inlet flow total. That said, the level sensor in the recondensing pot did provide evidence of suspected hydrogen ice build up.

The AFC uses a different system of paired Cernox sensors, giving instantaneous readings, so no further development is necessary.

3.7 Relief valves

As mentioned in Section 3.4, the hydrogen level in the vent line was observed to approach 50% during the hydrogen empty sequence. This is suspected to have been due to cold hydrogen boil-off causing water ice to form on the valve sealing face. Machining an O-ring groove in the sealing face could solve the problem but the leakage isn't necessarily a problem in itself – it only contributes to the general loss of hydrogen from the system during operation.

- ***As such, the pragmatic approach may be to take no action other than increasing the N2 flow rate to avoid accumulation.***

The relief circuit from the insulating vacuum experienced a significant leak of air into the cryostat during vacuum commissioning. This was arrested using an upstream check valve, although on the strict premise that the solution would be temporary.

- ***A more permanent solution will involve either retrospective modification of the various components or complete removal of the valve and replacement of the current rupture disc with a welded assembly. This will be considered at the post-R&D design review.***

3.8 Other recommendations

For general improvement of the system, the following recommendations will be considered at the design review.

- ***Addition of a flow meter to detect hydrogen flow into the hydride bed.***
- ***Movement of PG07 to the bed-side of the regulator to determine the pressure inside the bed.***
- ***Removal of the cryostat relief line check valve and implementation of a permanent solution to the leaking relief valve.***
- ***Fitting of upstream valves to all vacuum sensors to allow maintenance while at vacuum.***
- ***Insertion of a drain valve to the heater-chiller coolant circuit.***
- ***Replacement of all copper gaskets with silver-coated nickel gaskets where flange is not protected from rain.***
- ***Replacement of helium supply regulator dial gauge (broken).***
- ***Movement of heater-chiller and auxiliary chiller to external location to avoid effects of stray magnetic fields.***