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Design Description for the Seeded Ultrafiltration Plant at Sudbury

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Introduction

This preliminary design description of the seeded ultrafiltration plant at Sudbury was first written on the 8th of September 1994. It will be periodically updated as the design continues to evolve. A general description of the technique of seeded ultrafiltration, its role in the D_2O system and the criteria the SUF plant has to satisfy can be found in Refs. [1], [2], [3] and [4].

The aim here is to describe the components and modes of operation of the SUF plant. At the simplest level, the plant consists of two identical and independent SUF rigs which are surrounded by a number of shared components. The shared components are shown in flow sheet N2-93-93 where the two SUF rigs appear as black boxes, each with a D_2O inlet and outlet and with connections to H_2O and D_2O head tanks, as well as a cover gas system and a bottle of compressed Nitrogen (at 5 and 10 psi). The D_2O inlets and outlets are connected to the D_2O system via a manifold which allows the rigs to be used separately or together both in series or in parallel. This manifold uses a D_2O pump (200 l/min at 60 psi) to run the two rigs in series. It can also be used for running a rig in a (small volume) closed D_2O loop on its own, which may be useful for full procedural blanks.

The internal components of a SUF rig are shown in flow sheet N2-93-92 and lay-out drawing N0-93-96. The combination of these internal components and the external (shared) components described above allows each rig, without interfering with the other rig, to perform all the basic functions of seeded ultrafiltration: D_2O filtration, acid elutions of the membrane, secondary concentration of the eluates, alkali washes of the membrane, HTiO priming and deuteration.

In each rig there are two sets of isolation points (in the glove box) which are used to physically disconnect the UF membrane from either the D_2O system or the H_2O chemical stocks. At all times during the operation of a rig at least one of these sets of isolation points is disconnected, so that the risks of contaminating the D_2O with H_2O or other chemicals, or of losing some of the D_2O, are minimised. A further safety measure is included in the H_2O and D_2O head tanks which have a maximum capacity of 12 litres and cannot be emptied until they are full and their inlets shut-off.

Shared Components External to the Rigs

- **Head Tanks:** Maximum of 12 litres for both H_2O and D_2Ohead tanks. Used to fill the mixer tanks (or recirculation reservoir directly) by gravity. Located above the mixer tanks. Float valve so that a tank can only be emptied once it has been filled and the inlet shut off.

- **Compressed N_2 supply:** Delivered at both 5 and 10 psi from the same N_2 cylinder. In both cases the maximum flow of N_2 is restricted to $\sim$ 10 l/min so that N_2 doesn't rush too fast into the pipework and internal sprays of gas/liquid mixtures are avoided in the system.

- **Cover Gas System:** This cover gas system comes into contact with D_2O, H_2O and various acid and alkali solutions such as 0.5M HCl and 0.1M NaOH. It is not clear whether it should be part of the larger cover gas system for SNO or whether it should be a small system, particular to the SUF plant only in order to avoid chemical contamination. It is not in direct contact with the D_2O solution during D_2O filtration (valved-off) and so, in theory, has a higher radon tolerance.

- **D_2O Manifold:** This 2" piping system allows the two rigs to be used one at a time or together in series or in parallel. It receives D_2O from either the D_2O recirculation system or the initial fill system, and, after routing through the SUF rig(s), returns it to the appropriate line. It is assumed that the input flow of D_2O is driven by pumps external to the SUF plant which deliver D_2O at 60 psi pressure and at a variable flow between 100 and 200 l/min. When the two rigs are being used in series a further D_2O pump which is part of the SUF plant is used to boost the pressure between the two rigs back to 60 psi.

Internal Components of the Rigs

- **UF Membranes:** Each rig will house two 4ft Amicon cartridges (connected in parallel), either H53P30-20 cartridges (10 nm pore) or H26MP01-43 cartridges (0.1 micron pore), which should deliver, respectively, 100 and 200 l/min D_2O flow at 10°C and 60 psi trans-membrane pressure. Either set of two cartridges has a hold-up volume of about 1.4 litres when drained by gravity, and about 0.7 litres when drained by gravity with the assistance of 10 psi gas pressure on the permeate side and a flushing flow of gas on the concentrate side.

- **D_2O Plumbing:** The plumbing consists of 2" and 1.5" pipes for delivering D_2O to the concentrate side of the UF membrane and back from the permeate side. This is optimised for minimal flow impedance so that as little as possible of the 60 psi inlet pressure is wasted in getting the D_2O to the membrane and away from the membrane. For this reason connections are made to the top and bottom ports of both the concentrate and permeate sides of the membrane, which is used as a dead-end filter. There are 6 D_2O valves in the rig and two D_2O disconnection points inside the glove box which are used to isolate

the membrane from the D_2O system. This isolation is mandatory for all H_2O chemical processing of the membrane.

- **H_2O Recirculation Plumbing:** 1" pipe for performing all of the following chemistry operations: HTiO priming, acid elutions, alkali washes and deuteration. This plumbing has been optimised to minimise the pipe volumes whilst still allowing flows of up to 100 l/min (H_2O). It allows recirculation of the concentrate stream in either direction (up or down the inside of the hollow fibres) and also recirculation of the permeate stream (if required). It includes 11 valves, 4 non-return valves, three pressure gauges (up to 80 psi), a sight pipe (transparent piping), a pH meter and a recirculation reservoir with a conical bottom as an outlet and a T-piece as an inlet. Two of the non-return valves are located on the water and gas inlets of the recirculation reservoir. These only allow gas or liquid into the reservoir and are very useful for draining the rig, when gas from the 5 psi line can only exit the H_2O recirculation plumbing via the drain point Hdrain. The other two non-return valves are used as a precaution for not allowing HTiO to flow into the permeate side of the membrane during HTiO priming. If it were allowed to accumulate there it could later release itself into the D_2O during D_2O filtration.

- **Recirculation Pump:** Must deliver at least 80 l/min H_2O at 8 psi output pressure. It would be considerably more practical if it were an adjustable pump, but this is not absolutely necessary since there are valves immediately downstream of the pump which can be throttled down to control the 'effective' output of the pump. The dead-volume of the pump after draining it in the forward direction must be less than 100 ml. Because it is always valved off from the 1,000 tonne D_2O solution, its radon requirement is not as severe as for other pumps in the D_2O system, but some care must still be taken. At present there is a choice of two pumps: i) a Totton magnetically coupled centrifugal pump which is adjustable and has a very small hold-up volume but could be a radon problem and is known to produce RF interference in nearby instrumentation, ii) Osmonics 1" Diaphragm pump which is good for radon and RF but may have a significant hold-up volume (Is it adjustable?).

- **Stock Tanks:** removable 12 litre tanks to hold the following chemicals: 10M HCl, 5M HNO_3, 2,500 ppm HTiO, 10M NaoH. The HTiO stock must be vigorously stirred before dispensing to the HTiO mixer tank. Located next to their respective mixer tanks.

- **Mixer Tanks:** For making the following 12 litre dilute solutions: 0.5M HCl, 0.03M HNO_3, 250-500 ppm HTiO, and 0.1M NaOH. All of these solutions must be thoroughly stirred before introduction into the recirculation plumbing. Automatic dispensers are used to pass the concentrates from the stock tanks into the mixer tanks. The mixer and stock tanks are located on a rack above the glove box. The mixer tanks are connected to the H_2O cover gas (is there a problem with acid fumes?) and to the recirculation plumbing via a 1/2" pipe manifold. This manifold passes through the glove box so that all

of the above tanks (stock, mixer and head) can be isolated from the recirculation plumbing by physically disconnecting the 1/2" pipe. This isolation is mandatory whenever the SUF rig is connected to the D_2O system.

- **Elution Drain Tanks:** Two 12 liter tanks, located below the recirculation plumbing, for recovering 0.5M HCl and 0.03M HNO_3 acid elutions. Both connected, via automatic dispensers, to 10M and 1M NaOH neutralisation tanks, so that the acid elutions can be neutralised in situ. Mixing and pH meters are required as well as connections to the cover gas system. Connected by 5mm bore flexible piping to peristaltic pumps in the glove box.

- **Neutralisation Tanks:** 10M and 1M NaOH tanks (12 litres each) which are next to the elution drain tanks and connected to both via automatic dispensers. Demountable.

- **H_2O Waste Drain tank:** 100 litre tank for collecting the H_2O solutions which are used to clean the UF membrane, i.e. NaOH and HCl solutions as well as H_2O rinses. Demountable and connected to cover gas.

- **D_2O Drain Tanks:** Two 100 litre tanks for collecting the high and low isotopic purity D_2O waste from the recirculation rig. The high isotopic tank is used to drain the pure D_2O in the rig at the end of a filtration run and may be contaminated with trace amounts of HTiO and so must be filtered before re-introduction into the bulk of the D_2O. The low isotopic tank is used to collect waste solutions during deuteration of the membrane, and will thus have to be upgraded. It might also contain trace amounts of HTiO, and should also be filtered. Both tanks are demountable and connected to the cover gas.

- **Glove Box:** Houses the D_2O and H_2O isolation points. Contains the SUF Rig for secondary concentration of the neutralised acid elutions: peristaltic pumps, small UF membranes (150-700 cm^2 area), acid bottles (filled from the stock tanks). The resulting acid eluates from these secondary SUF rigs are mixed with liquid scintillator in a counting jar which is sealed before removing from the glove box via a side-port with air interlock. Connected to cover gas.

- **Glove Box Waste Tank:** A couple of tundishes feed this tank from the glove box, so that waste solutions from the glove box can be disposed of. It is connected via an air return line to the glove box, to facilitate draining from the tundishes. Demountable.

Modes of Operation

Usually, a SUF rig will be run in a repeating cycle of six operations:

1. D_2O filtration
2. acid elution
3. secondary concentration

4. alkali washes

5. HTiO priming

6. deuteration

However, other cycles are possible, e.g. no stage 6 if all chemicals used in the rig are already deuterated, or the order of 5 and 6 being swapped if DTiO is used but other chemicals are not deuterated, or only a sub-set of the full cycle of operations is performed to measure a specific background. We will attempt to describe these operations in a semi-independent way, so that as many different permutations as possible are allowed.

1. **D_2O Filtering:** It is assumed that before this operation is begun the membrane has already been primed with HTiO, deuterated and drained. One of the overriding concerns in many of the steps described below is to disturb as little as possible the deposited layer of HTiO. Sudden rushes of water, especially if they are backflushes, might remove some of the HTiO, which might then find its way into the D_2O drain tanks. Nevertheless, this is not a serious problem, as these drain tanks will be filtered (by fine UF or maybe even RO) before re-introducing the D_2O into the 1,000 tonne solution. The more serious problem of allowing HTiO into the permeate stream (and hence into the 1,000 tonne solution) does not occur except if HTiO goes through the membrane.

   (a) Close all valves in the H_2O recirculation plumbing and disconnect the H_2O isolation point in the glove box.

   (b) Re-connect the D_2O isolation points inside the glove box.

   (c) Open and close valves on the D_2O manifold according to how the rigs are to be used, i.e. only one rig or both in series or in parallel. It will now be assumed that there is enough pressure on the D_2O inlet line (of the relevant SUF rig) to push D_2O to the top of the rig (just below the mezzanine floor), but not too much pressure so that opening the inlet valve will create a sudden rush of D_2O.

   (d) Fully open the four D_2O valves named Dconctop, Dconcbot, Dpermtop, Dpermbot.

   (e) Crack open the D_2O inlet valve Din, to the point where a small flow of D_2O enters the rig. This flow will continue until all the trapped gas in the rig is at the same pressure as the D_2O inlet line.

   (f) Crack open the valve Hconctop so that D_2O slowly fills the concentrate side of the membrane, until it starts to flow past the sight pipe and into the recirculation reservoir. At this point Hconctop is firmly shut.

   (g) Crack open the valve Hpermtop so that D_2O slowly fills the permeate side of the membrane, until it starts to flow past the sight pipe and into the recirculation reservoir. At this point Hpermtop is firmly shut.

(h) Fully open the D_2O valves Din and Dout and, if the other SUF rig is being used in this run, then repeat all of the above steps with the other rig. The rig is now ready to filter D_2O and the D_2O pumps can be switched on.

(i) There may be some remaining gas trapped on the concentrate side of the membrane, which would decrease the water permeability of the membrane by reducing its effective surface area. To flush this gas out, valve Hconctop should be cracked open just enough to allow for a small flow of D_2O past the sight pipe. During this bleeding, the D_2O valves Dconctop and Dconcbot should be alternatively closed and opened. After a couple of minutes, Hconctop is firmly shut and Dconctop and Dconcbot fully opened.

(j) During the D_2O filtration run it may happen that gas from elsewhere in the D_2O system comes down the line and is trapped on the concentrate side of the membrane. If sufficient gas is trapped, then the flow rate will drop and step 1(i) should be repeated to remove the gas.

(k) After the required volume of D_2O has been filtered, the D_2O pumps are switched off, and valve Din is closed. If possible, the membrane is then drained through valve Dout, by opening the valves Hpermtop and Hconctop to allow gas (from the cover gas) into the rig. This will only work if there is minimal back pressure on the D_2O outlet line. After draining, close valve Dout.

(l) Drain the remaining D_2O in the rig into the high isotopic tank by opening all the valves on the H_2O side of the membrane.

(m) Close all four valves Dconctop, Dconcbot, Dpermtop and Dpermbot. Disconnect the D_2O isolation points in the glove box.

(n) Close Hpermtop and Hpermbot and open valve Gasperm. This will further drain the membrane by squeezing a bit of D_2O out of the walls of the membrane and into the concentrate side.

(o) Open valve Gasconc which will also further drain the membrane, by pushing D_2O down the inside of the hollow fibres. During this flushing it is useful to alternatively close Hconctop and Hconcbot which will force the gas alternatively through the recirculation pump and membrane. After a couple of minutes, close Gasconc, Hrev1-Hrev4 and Hdrain and open Hconctop and Hconcbot. The membrane (+ system) is now fully drained and will contain about 800 ml of D_2O.

(p) To recover the 800 ml of D_2O, introduce 12 litres of H_2O into the H_2O Head tank. Re-connect the H_2O isolation point inside the glove box and introduce the 12 litres of H_2O into the recirculation reservoir.

(q) Switch on the recirculation pump and recirculate at about 20 l/min with less than 5 psi backpressure for 10 minutes. This should mix the 12 litres of H_2O and 0.8 litres of D_2O inside the walls of the membrane. Then switch off the pump, open the drain valves and allow the 6% D_2O

solution to pass into the low isotopic D_2O tank. Open valve Gasconc and alternatively close Hconctop and Hconcbot, as before, to facilitate draining. Close Gasconc and the drain valves, and open Hconctop and Hconcbot. This de-deuteration step recovers 750 of the 800 ml of D_2O, leaving behind 50 ml in the membrane, which is the accepted loss per D_2O filtration run with a SUF rig. The rig is now ready for the H_2O chemical operations, which usually start with acid elutions.

2. **Acid Elutions:** For these operations, it is assumed that the rig is connected to the H_2O stock tanks and disconnected from the D_2O system. Usually two acid elutions, 0.03 M HNO_3 and 0.5M HCl, will be performed in quick succession, but sometimes the same acid strength will be used twice to check for elution efficiencies from the ratio of counts in the two elutions. In terms of circulation flow there are two ways of eluting the membrane. The first, standard method, is better understood and relies on recirculating acid through the membrane (permeate valve open), and also along the inside of the hollow fibres (concentrate valve open). In this way excellent contact is achieved between the acid and the HTiO (deposited on the membrane surface). An alternative method, which has not yet been investigated, would be to only recirculate inside, but not through, the hollow fibres (concentrate valve open and permeate valve shut). This is expected to be just as efficient and has the advantages of i) less acid is required (perhaps 3-4 litres less) and ii) It is a simpler operation (less opening and shutting of valves). We will first describe the better understood standard method:

   (a) Close the following H_2O valves: Hrev1-4, Hpermtop, Gasconc, Gasperm and Hdrain. Open Hconctop, Hconcbot and Hpermbot.

   (b) Prepare 10-12 litres of the required acid in the relevant mixer tank and introduce this acid into the recirculation reservoir.

   (c) Switch-on the recirculation pump, then adjust the pump and throttle valve Hconctop so that Flowconc = Flowperm = 20-40 l/min and the trans-membrane pressure is in the range 5-10 psi. After the relevant elution time (5 minutes for 0.5 M HCl and 10-15 minutes for HNO_3) switch-off the recirculation pump.

   (d) Shut Hpermbot and open Gasperm.

   (e) Open Hdrain and Gasconc and allow the acid to drain into one of the elution drain tanks.

   (f) Close Gasperm, Gasconc and then Hdrain.

   At this point the system is ready for another acid elution, i.e. go back to step 2(a). For the alternative elution method use steps 2(a)-(f) with the following changes: 2(a) open Gasperm and close Hpermbot, 2(b) use about 3 litres less acid, 2(c) recirculate at 10-20 l/min with less than 5 psi backpressure, 2(d) miss this step, and 2(f) close Gasconc and then Hdrain.

   After the first two elutions, it is customary to perform two more elutions with 0.5M HCl for one hour each, using the standard elution method and draining

to the H_2O waste tank. These are used to dissolve the HTiO which is impaired by the first acid elutions and cannot be re-used. However, it may be possible to regenerate the HTiO with alkali washes. This would significantly reduce the acid and HTiO consumption of the rigs, but unfortunately, some early attempts at regeneration were unsuccessful and, at this point in time, HTiO regeneration is not an anticipated operation.

3. **Secondary Concentration:** Usually, the first two elutions of the SUF rig will be drained to the acid elution drain tanks for secondary concentrating. This secondary concentrating can take place at the same time as the third and fourth HTiO dissolving elutions of the primary membrane. In most SUF runs the first elution will be 0.03M HNO_3 for eluting Ra and the second elution will be 0.5M HCl for eluting Pb and Th. Because of the 10.6 hour half-life of ^{212}Pb it is important to process the 0.5M HCl elution as quickly as possible. Hence, there are two acid elution drain tanks, so that the 0.03M HNO_3 elution can be stored whilst the 0.5M HCl elution goes through the secondary concentration process first.

   (a) Neutralise the 0.5M HCl elution to pH 8-9 by first adding 10M NaOH, then 1M NaOH, from the neutralisation tanks.

   (b) Filter the neutralised solution with a secondary UF membrane of area 700-1400 cm^2, depending on how much HTiO was deposited on the primary UF membrane at the beginning of the run (3-6g). A possible secondary membrane is the Filtron ultrasette or minisette cassette, which has 700 cm^2 area and is remarkably compact.

   (c) Elute the secondary filter with 50-100 cc of 0.5M HCl.

   (d) Dilute the eluate with the required amount of 0.5M HCl so that re-precipitation of the dissolved Ti does not occur. Do we know how much this is?

   (e) Mix the eluate with liquid scintillator (in a counting jar) in a prescribed ratio. Seal the jar and remove it from the glove box.

   (f) Count the β-α coincidences [5], [6] for two weeks.

   For concentrating the 0.03M HNO_3 primary acid elutions follow steps 3(a)-(f) with the following changes: 3(b) membrane area 100-200 cm^2, possibly Mediakap 10, but these have been known to burst at only 2 bar pressure, 3(c) elute with 10-20 cc, 3(d) not required.

4. **Alkali Washes:** These are used to recondition the membrane as NaOH is a very effective cleaning agent for membranes which restores permeate rate to high values. It is also possible that the NaOH removes some Th species which are not eluted by acids. Almost invariably these alkali washes will occur after acid elutions. The technique that shall be used is a high flow recirculation cleaning with the permeate valves shut and the cartridges full on both sides of the membrane which is why 12 litres are required. A first wash will be performed with the concentrate flow in the upward direction, then a second

with the flow in the downward direction. In this way the whole length of the hollow fibres are effectively backflushed.

(a) Rinse the rig with 12 litres of H_2O using the standard elution method for recirculation (see steps 2(a)-(f)). The obvious change is that the H_2O is drained to the H_2O waste drain tank. This step is to remove excess acid from the rig which might otherwise have neutralised the alkali.

(b) Close the following valves: Hrev1-4, Hpermbot, Gasconc, Gasperm and Hdrain. Open Hconctop, Hconcbot, Hpermtop.

(c) Prepare 12 litres of 0.1M NaOH and introduce this into the recirculation reservoir.

(d) Recirculate at high flow (up to 100 l/min) which should produce about 10 psi backpressure. This pressure will cause the outer casing of the cartridges to fill with alkali (hence the need for 12 litres of alkali). When the casing is full, close Hpermtop. Recirculate for about one hour.

(e) Open Gasperm and reduce recirculation flow to 20 l/min. The backpressure will be less than the gas pressure and the alkali will permeate backwards through the membrane and the cartridge will empty. When it is empty, drain the rig to the H_2O waste tank by switching off the recirculation and opening Hdrain and Gasconc.

(f) Close Gasconc, Gasperm, Hdrain, Hconctop, Hconcbot and open Hrev1-4 and Hpermtop.

(g) Repeat steps 4(c)-(e) so that the membrane is washed again with fresh alkali flowing in the downward direction.

(h) Repeat step 4(a) twice to wash out the alkali from the Rig and bring the pH back down to 10-11 (it will further reduce during HTiO priming and deuteration and will approach pH 7 by the time the membrane is ready for D_2O filtration).

5. **HTiO Priming:** This operation is very similar to the standard acid elution method:

(a) Close the following H_2O valves: Hrev1-4, Hpermtop, Gasconc, Gasperm, Hdrain *and* Hpermbot. Open Hconctop and Hconcbot.

(b) Prepare an HTiO solution of suitable concentration and take a sample from the feed sample point before introducing into the recirculation reservoir. It is expected that 0.5 g Ti/m^2 will be required on the membrane so that 5g of Ti (in HTiO form) are required for the H53P30-20 cartridges (10 m^2 total) and 2.5g for the H26MP01-43 (5 m^2 total).

(c) Introduce the HTiO into the recirculation reservoir. It is very important not to let the HTiO get into the permeate side of the membrane and this objective is satisfied by having Hpermbot and Hpermtop closed, with double protection from the non-return valves Npermbot and Npermtop.

(d) Switch-on the recirculation pump, then adjust the pump and throttle valve Hconctop so that Flowconc = 20-40 l/min and the backpressure is about 3-5 psi. Only then should Hpermbot be opened to guarantee permeate flow in the right direction. As soon as possible adjust the pump and throttle Hconctop so that Flowconc = Flowperm = 20-40 l/min and the transmembrane pressure is in the range 5-10 psi. Recirculate for one hour. Shut Hpermbot, fully open Hconctop, switch the pump off and open Gasperm.

(e) Open Hdrain and Gasconc and allow the H_2O to drain into the H_2O waste tank, after taking a sample from the drain sample point.

(f) Close Gasperm, Gasconc and then Hdrain.

6. **Deuteration:** After the above HTiO priming operation there is about 800 ml of H_2O in the walls of the membrane. This amount has to be reduced to 10 ml, the acceptable downgrade per SUF run. The simplest method of deuteration, from an operational point of view, is repeated rinsing of the membrane with pure D_2O. In theory only two 8 litre rinses (using the standard acid elution recirculation method) are required before the 10 ml target is reached, however it seems prudent to have three such rinses. Also to reduce the cost of this process it is best to use already downgraded D_2O for the first rinse, e.g. 95-99% D_2O would be adequate. The question arises as to how many different grades of D_2O one is willing to keep track of? There has been an alternative deuteration technique proposed of using warm gas to dry off the 800 ml of H_2O. Unfortunately no tests have been performed so far, and it is possible that the HTiO would suffer irreparable damage in this drying step. Certainly HTiO has a reputation of being very sensitive to drying. Perhaps the drying stage could be performed before HTiO priming, which would then be replaced by DTiO priming. Yet another technique for deuteration is to introduce D_2O very slowly into the bottom of the membrane and displace the H_2O by gravity. Clearly these different deuteration techniques require careful study from which a decision could be made which will largely be based on cost, and security of the D_2O.

References

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