Design Criteria for Purification and Assay of Heavy Water by Seeded Ultrafiltration

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Introduction

In order to measure the neutral current signal in SNO it is necessary to purify the heavy water plus neutral current additive to an activity level of one decay per day per tonne of D_2O (DDT) for the thorium chain and 20 DDTs for the uranium chain. It is equally important to be able to monitor these activity levels, by some form of radioactive assay. From the point of view of purification the critical isotopes are 2 year ^{228}Th in the thorium chain (see Fig. 1) and 1600 year ^{226}Ra in the uranium chain (see Fig. 2); whereas for monitoring, the critical isotopes are respectively ^{224}Ra and ^{222}Rn. However, in addition to monitoring ^{224}Ra and ^{222}Rn, the assay of ^{212}Pb and ^{228}Th in the thorium chain and ^{226}Ra in the uranium chain are very important for investigating possible disequilibria in the two chains, which may arise if the dominant sources of ^{228}Th and ^{226}Ra are not in the D_2O solution.

The purpose of this document is to outline the design criteria of a seeded ultrafiltration (SUF) plant located down the mine at Sudbury which is able to accomplish all of the above tasks except for the assay of ^{222}Rn. It might be preferable to consider two separate SUF plants for purification and assay, but the overlap in design criteria between the two is so great that it is more economical to combine these into one. As a simple example, a filtration rate of 200 litres/min is required to turn over the volume of the detector on a reasonable timescale for purification (4 days) and also to assay a sufficiently large volume of water (100 tonnes) in a time (8 hours) which is comparable to the half-life of ^{212}Pb (10.6 hours).

Both tasks of purification and assay by SUF would be considerably simpler if the SUF plant were located on the concentrate side of the reverse osmosis (RO) plant which could then be used as a preconcentrator, reducing a flow of 200 litres/min to one of 10-20 litres/min. However, the uncertainties arising from backgrounds from the RO membrane are felt to be too much of a risk at present to rely on such a scheme. To illustrate this point let us consider a ^{212}Pb assay experiment in which a 100 tonne sample of D_2O is monitored at the 1 DDT level. A considerable fraction of the ^{212}Pb activity from ^{228}Th embedded in (or plated onto) the $\sim$ 100 Kg of microporous membrane material will find its way into the D_2O via the mobility of the intervening 1 minute ^{220}Rn. For a signal to noise of 10:1 we require the RO plant to produce less than 10 ^{212}Pb decays per day, which is equivalent to a level of $\leq 3 \times 10^{-13}$ g/g ^{232}Th in the membrane (assuming all the ^{220}Rn from the membrane

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finds its way into the water). By comparison a SUF plant for 200 litres/min flow has only ~ 1 Kg of membrane so that the requirement becomes $\leq 3 \times 10^{-11}$ g/g ^{232}Th in the membrane.

Perhaps even more important than the difference in membrane mass between the RO and SUF plants is the difference in ability to clean the membrane from plated out ^{228}Th. This is a very serious concern given the propensity of ^{228}Th to plate out onto the large surface areas provided by both the RO and SUF membranes. The SUF membrane will be considerably easier to clean because it is compatible with a wider range of cleaning agents (e.g. 0.5 M HCl and 0.1 M NaOH), and because there is a provision for on-line cleaning (and background checking) after every run, whereas the RO membrane can only be cleaned by removing the membrane from the plant to a separate rig which will be able to clean a few of the fifteen membrane cartridges at a time. Lastly, the relatively low cost of replacing all the SUF membrane cartridges, $\sim 4,000$ pounds sterling, is a safeguard against the possibility of uncleanable and excessive ^{228}Th plating which might occur in the early stages of the operation of the D_2O system.

The method of seeded ultrafiltration has been detailed elsewhere [1], [2], but it will be briefly summarised here with the aid of Fig. 3. A fine suspension of Hydrous Titanium Oxide (HTiO) is deposited and retained on the surface of an ultrafiltration membrane. D_2O is then passed at 200 litres/min through the primed membrane which extracts ^{228}Th, ^{226}Ra, ^{224}Ra and ^{212}Pb from solution by adsorption onto HTiO. After a suitable volume of D_2O has been processed (100 tonnes or more) the membrane is drained and then eluted with about 6 litres of 0.03 M HNO_3 which pulls off the ^{224}Ra and ^{226}Ra from the HTiO and brings them back into solution, whilst leaving the ^{228}Th and ^{212}Pb on the HTiO and also dissolving about 4% of the HTiO (into Ti ions). This acid solution is then drained out of the UF membrane. Next, a stronger acid elution of 0.5 M HCl is used to elute ^{228}Th and ^{212}Pb off the HTiO, whilst also dissolving 25% of the HTiO, and is drained into a separate reservoir. The two acid solutions are then neutralized (separately) by the addition of NaOH which re-precipitates the dissolved Ti into HTiO; and this HTiO re-absorbs the eluted radioactive species. These neutralized solutions are then filtered by much smaller secondary UF membranes which can be eluted with 10-20 mls of 0.1 M HNO_3 in the case of ^{224}Ra and ^{226}Ra and 50-100 mls of 0.5 M HCl for ^{228}Th and ^{212}Pb. These acid solutions are then mixed (separately) with a liquid scintillator which accept aqueous solutions so that they can be counted by β-α coincidences [3], [4]. Further elutions of the main UF membrane with 0.5 M HCl are used to dissolve the remaining HTiO, and then washes with 0.1 M NaOH restore the membrane to optimum condition, so that it is ready for another D_2O filtration run which, once again, starts with the deposition of fresh HTiO on the membrane.

Design Criteria

1. **Plant Configuration:** Two independent SUF units will be built side-by-side, see preliminary drawing N2-93-90. They will each be able to filter at least 100 litres/min D_2O at 10°C (which is equivalent to 200 l/min H_2O at 20°C), but the optimum rate is 200 l/min D_2O each. The plumbing will allow the two

units to be operated together in parallel or in series, or either of the two units on its own. Isolation valves on the inlet and outlet of each unit (4 valves in total) will allow a unit to be physically disconnected from the rest of the D_2O system. These isolation valves will be located inside a glove box, and will be disconnected every time a chemistry operation (acid elution, HTiO priming, alkali cleaning etc.) is performed in a unit.

2. **Pore Size:** $\leq 0.2\mu$m, must retain $\geq$ 99% of the HTiO at all times during the filtration run, which is up to 1,000 tonnes of D_2O processed. The particle size distribution of HTiO is given in Fig. 4 from which it would seem that a 0.1-0.2 μm pore size will comfortably retain the HTiO. Indeed, numerous tests with 0.2 μm pore size have shown $\geq$ 99.5% retention of the HTiO. However there is still some concern about the possibility of the HTiO breaking down into smaller particles (peptization) over the course of a 1,000 tonne extraction, particularly at low ionic strength (i.e. D_2O without 0.2% $MgCl_2$). It should be noted that there is quite a tight UF membrane (20,000 Daltons NMWC $\simeq$ 10 nm pore size) between the SUF plant and the acrylic vessel, which acts as a final barrier for breakthrough of fines. However, we do not want to rely on this 'back-up' UF rig in the design of the SUF plant. In this way we keep the back-up UF rig as an extra safety factor and also avoid the risk of fouling the back-up UF rig with HTiO.

3. **Membrane Fouling:** The pure water permeate rate of most UF membranes drops-off to about half its initial value quite soon after rinsing out the glycerol solution used during shipping. If it is then periodically cleaned with 0.5 M HCl and 0.1 M NaOH solutions it will keep this permeate rate of half the initial value, provided there is no accumulation of insoluble particulate matter on the membrane. This is to be expected in a system of high purity water such as the D_2O system, but there is a concern that trace quantities of TiO_2 will form in the stock bottles of HTiO. TiO_2 is insoluble (by the above cleaning solutions) and would thus foul the membrane. The remedy is to avoid TiO_2 formation by keeping HTiO stock bottles in the dark, and by establishing a shelf-life for the HTiO.

who, where prepared ?

4. **Mechanical Integrity:** The maximum trans-membrane pressure allowed for hollow fibre membranes is 60 psi. This is based on establishing, in water tests, that the burst pressure for the membrane is > 100 psi. At present this has only been measured by the manufacturer, but soon burst tests will be carried out at Oxford at up to 150 psi water pressure. The integrity of a UF membrane can be tested quite easily by standard procedures, involving a few psi gas pressure, which can detect even the smallest rupture of the membrane. These tests will be carried out on a routine basis for all membranes in the SUF plant. The membrane is housed in a polysulphone cartridge which is connected, through end-flanges and adaptors, to standard polypropylene piping. If necessary, these

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end-flanges and adaptors will be modified so that no rupture or leakage occurs with 150 psi water pressure inside the cartridge. The rest of the plumbing system (piping, pumps, adaptors, valves, instrumentation) will be required to comply with the same specifications as the rest of the D_2O system (as specified by CRPP). Indeed, many of the components might originate from CRPP.

5. **Extraction Efficiency:** $\geq 90\%$ extraction of ^{226}Ra, ^{224}Ra, ^{212}Pb and ^{228}Th at all times during the filtration run which is 1,000 tonnes for ^{228}Th and ^{226}Ra purification and 100 tonnes for ^{212}Pb, ^{224}Ra and ^{226}Ra monitoring. This requirement determines the amount of HTiO deposited on the membrane (currently expected to be 0.5 gTi/m^2). The SUF plant will have the ability to run with two SUF units in series. This will provide an independent check on the extraction efficiency from the ratio of counts between the two units. It has been shown [5] in tests with stable Barium spiked with ^{133}Ba that the ion exchange capacity of HTiO is 3 milliequivalents of Ba per gram of Ti, or 0.07 moles of Ba per mole of Ti, or 0.2 grams of Ba per gram of Ti. This capacity is many orders of magnitude in excess of what is required to purify the D_2O from all thorium and uranium chain isotopes, but the selectivity of HTiO is such that significant quantities of any transition element will saturate the HTiO and interfere with the absorption of Ra in particular, but also, to a lesser extent, Pb and Th. To illustrate the scale of the problem, let us look at Fe III which would be selected by HTiO in preference to Ra. The amount of HTiO used to process the entire 1,000 tonnes of D_2O is about 2.5 g of Ti. This has a capacity for absorbing 0.125 g of Fe, so that 125 ppt of Fe in the D_2O would saturate the HTiO. Thus, a limit of about 10 ppt total is allowed for all transition elements in the D_2O. This includes ^{208}Pb, which has been suggested as a carrier for determining ^{212}Pb extraction efficiency by measuring ^{208}Pb recovery. Less stringent requirements are imposed on the alkaline-earth elements (other than Ra) which will not be selected before Ra, but which can nonetheless interfere with Ra absorption onto HTiO if they are present in excessive quantities, e.g. 0.2% MgCl$_2$ solutions do not interfere, but 5% MgCl$_2$ solutions do exhibit some interference and CaCl$_2$ solutions show substantial interference at the 5% concentration level. In view of all these constraints on the chemical purity of the D_2O it is strongly recommended that a general purpose ion-exchange bed be added to the D_2O receiving system, before the SUF plant, otherwise the performance of the HTiO may be impaired to a degree that it is ineffective.

6. **Magnesium Chloride:** The performance of the SUF plant must not be affected by the addition of 2 tonnes of MgCl$_2$ to the 1,000 tonnes of D_2O. This essentially means no significant change in the above extraction efficiencies. Thus the same purity limits as above on transition elements are imposed on the 0.2% MgCl$_2$ solution, so that an elemental analysis is required of the purified 5% MgCl$_2$ solution before it is added to the D_2O. Further tests, in small scale laboratory SUF rigs, are required to check that 0.2% MgCl$_2$ in H$_2O$ (made from purified 5% MgCl$_2$ stock) behaves exactly as expected, without

any signs of interference with Ra absorption onto HTiO.

7. **EDTA:** The performance of the SUF plant must not be affected by the addition of EDTA. If small scale laboratory SUF rigs show that the extraction efficiency for any of the three important elements: Pb, Ra or Th drops significantly below 90% at the concentration of EDTA which is anticipated in SNO then it must be demonstrated that the EDTA can be removed from the D_2O system to a concentration level where it no longer affects the extraction efficiencies.

8. **Acid Elution:** Not more than ten litres of acid should be required to elute the HTiO. This acid has to gain access to all the HTiO (all the membrane surface) and should be recoverable after five minutes of recirculation. The five minute time constraint is motivated by the desire to minimize the amount of HTiO dissolved per elution. Different acid strengths are envisaged for eluting the different elements: 0.03 M HNO_3 for ^{226}Ra and ^{224}Ra, and 0.5 M HCl for ^{212}Pb and ^{228}Th, but in all cases the elution efficiency should be $\geq$ 80% (including the loss associated with undrainable dead-volumes). Elution efficiencies can be checked by re-eluting the membrane immediately with fresh acid and comparing the counts from the two elutions. Acid elution is just as important for purification as for assay because purification is only achieved by removing the long lived ^{228}Th and ^{226}Ra from the D_2O recirculation system and not just from the D_2O solution.

9. **Secondary Concentration:** Further concentration of the above eluates is required in order to reduce the volume down to a size that can be counted by β-α coincidences ($\leq$ 20 ml for ^{226}Ra and ^{224}Ra, and $\leq$ 100 ml for ^{212}Pb and ^{228}Th). This secondary concentration by seeded ultrafiltration (or other techniques) should take no longer than 2 hours for ^{212}Pb and ^{228}Th, and 3 hours for ^{226}Ra and ^{224}Ra. The 2 hour time constraint comes from the 10.6 hour half-life of ^{212}Pb (and should ideally be shorter), whereas the 3 hour time constraint is motivated by the desire to enable one operator, in one 6 hour shift down the mine, to elute the two main UF units twice each, perform the secondary concentrations and start the β-α counters. The efficiency for the secondary concentrations should be at least 80%.

10. **β-α Counting:** At least twelve β-α counters will be located down the mine: 6 radium counters with an acceptance of up to 20 mls 0.1 M HNO_3 for ^{226}Ra and ^{224}Ra counting, and 6 lead counters with an acceptance of up to 100 mls 0.5 M HCl for counting ^{212}Pb and ^{228}Th. Both sets of counters will count thorium chain decays (^{212}Bi) with 50% efficiency, and the radium counters will also count uranium chain decays (^{214}Bi) with 80% efficiency. All counters will be operated for at least 2 weeks after each extraction, which, for the radium

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counters, will allow the growing in of ^{222}Rn from ^{226}Ra (uranium chain) and the growing in of ^{212}Pb followed by the decay of ^{224}Ra (thorium chain), and, for the lead counters, the decay of ^{212}Pb followed by the growing in of ^{224}Ra from ^{228}Th (thorium chain). By fitting the time spectrum of counts to the half-lives of the above elements we can deduce the amount of ^{226}Ra, ^{224}Ra, ^{212}Pb and ^{228}Th introduced into the counters. The intrinsic background of the above counters should be less than 1 count per day for the thorium chain and less than 10 counts per day for the uranium chain.

11. **Backgrounds:** The system itself must not contribute more than 10% of the expected signal for a 100 tonne assay at 1 DDT thorium chain and 20 DDT uranium chain activity. Since the overall efficiency for thorium chain monitoring is given by the following product: 90% (extraction) x 80% (elution) x 80% (secondary concentration) x 50% (β-α counting) = 29% (total), then the total system background from chemical reagents, membrane elution and β-α counting must be $\leq$ 3 counts/day (expected signal 30 counts/day). Similarly, the overall efficiency for uranium chain monitoring is given by: 90% (extraction) x 80% (elution) x 80% (secondary concentration) x 80% (β-α counting) = 46% (total), so that the total system background must be $\leq$ 90 counts/day (expected signal 900 counts/day). The total system background (for both chains) can be determined by a full procedural blank which involves all of the above steps (HTiO priming, acid elution, secondary concentration and β-α counting) with the obvious exception of processing the 100 tonnes of D$_2$O. It is important to carry out full procedural blanks on a regular basis: certainly after every change of chemical stock bottles, but also to keep a check on whether ^{228}Th or ^{226}Ra contamination is building up in the system. Subsystem background checks can be performed by omitting certain stages of the full procedural blank, e.g. by-passing the membrane in the elution stage, or eluting a membrane which has not been primed with HTiO. These sub-system background measurements can be used to track down the origin of an increase in the system background to any one of the following: reagents (acids and alkalis), membrane (plus recirculation plumbing), HTiO, secondary concentration rig or β-α counters (including scintillator).

12. **Cleaning:** After the five minute acid elutions of the main UF membrane (used for eluting ^{226}Ra, ^{224}Ra, ^{212}Pb and ^{228}Th with minimal HTiO dissolution) two more elutions with 0.5 M HCl will be performed for at least one hour each. These washes are necessary for dissolving the rest of the HTiO, which is not re-usable after it has been exposed to acids, and also for removing any plated out ^{226}Ra or ^{228}Th which might otherwise build-up on the membrane and become a source of background in the system. A water rinse occurs next, and then a wash with 0.1 M NaOH which is an excellent cleaning agent for polysulphone membranes and is very important for maintaining high permeate rates. For this purpose it is strongly recommended by the manufacturers to use the NaOH in a high flow recirculation mode with the permeate side of the UF cartridge full of fluid and valved shut. The recirculation flow establishes a

pressure gradient along the length of the hollow fibres which results in effective backflushing of the downstream half of the fibres. By switching the direction of flow, both halves of the fibres can be cleaned. The NaOH wash is also useful for removing certain chemical species of ^{228}Th, such as silicates, which are insoluble in acids.

13. **Closed System:** The entire SUF plant, including the apparatus required for performing all of the above operations (HTiO priming, D_2O filtration, acid elution, alkali cleaning and secondary concentration), will be a closed system (see preliminary drawing N2-93-90). The output of the system will be sealed jars containing a mixture of scintillator and acid eluate from the second UF stage. These jars will be prepared in a glove box and removed through a standard double-entry side port. The only other exposed operations will be the replacement of empty stock bottles of chemical reagents (which should last for at least 20 runs) and the replacement of UF membranes which is not anticipated to be more than once a year, but might be more frequent in the first year of operation of the D_2O system if excessive ^{228}Th plating is occurring on the membranes during initial purification of the D_2O.

14. **Radon:** If the SUF plant were in continuous use then practically all of its equilibrium level of ^{222}Rn emanation would be mixed into the 1,000 tonnes of D_2O. Thus the ^{222}Rn emanation from the SUF plant must be kept below a level of 1,000 ^{222}Rn decays per day which would correspond to 5% of the allowed 20 DDT uranium chain activity level in the D_2O. This requirement can be relaxed by a factor of up to four if a small volume of D_2O is recirculated in a loop through the SUF plant and monitor degasser before each D_2O filtration run, just after HTiO priming. In this way the ^{222}Rn is initially purged from the SUF plant and only grows back into equilibrium with its 4 day half-life.

15. **Potassium:** A consensus seems to have emerged from the SNO working group on backgrounds that a limit of 2 ppb g/g of natural K in the D_2O is required for the beta-neutron look back scheme for measuring thorium chain contamination directly in the D_2O. This corresponds to 2 grams of natural K in the D_2O. In order to be sure that K leaching from HTiO is not a significant source of K in the D_2O it is necessary to measure the amount of K found in the standard production HTiO. As long as it is less than 100 ppm then it would be impossible for 20 Kg of HTiO, the maximum conceivable amount of HTiO used over the lifetime of the detector, to leach 2 grams of K. This level of 100 ppm K in HTiO is unlikely to be difficult to establish, given that the dominant source of K contamination in the production of HTiO is from NaOH which is now commercially available with $\leq$ 2 ppm K.

16. **D_2O Downgrading:** Every full cycle of the system (HTiO priming, D_2O filtration, acid elution and membrane cleaning) should not downgrade the 1,000 tonne D_2O solution by more than 10 ppb, i.e. 10 g of H_2O contamination. At

the very most 100 cycles per year are anticipated, which would be a downgrading of 1 ppm per year, (cf) the 5,000 ppm downgrading allowed before we have to return the D_2O. At present, two methods for reaching the above downgrading criterium are being investigated: i) Using deuterated chemicals (DTiO, DCl, DNO_3, NaOD) in all of the above procedures which involve the main UF membrane, and ii) After D_2O filtration, switch to standard hydrated chemicals for all procedures until after the HTiO is deposited, then deuterate the membrane plus HTiO to less than 10 g H_2O contamination by repeated rinsing with D_2O (probably three times). The choice between the two schemes will be largely dictated by operating costs, as well as security of the D_2O. For the moment it is planned to design a system which can accomodate both schemes by having pure D_2O and pure H_2O head tanks, as well as D_2O drain tanks for high isotopic pure D_2O, high isotopic D_2O with dissolved salts (e.g. NaCl, DCl etc.) and low isotopic D_2O.

17. **Assembly:** Expected to take place at Oxford in a clean room of similar specification to the clean room at CRPP where the majority of the D_2O system will be assembled. The cleaning procedures before and during assembly will be identical to those used at CRPP, and after assembly (onto one or more skids) the plant will be sealed and packaged in similar manner to the rest of the D_2O system and delivered directly to the mine.

References

[1] Seeded Ultrafiltration Progress and Prognosis, S. Lilley, M. Moorhead and N. Tanner, SNO-STR-92-089.

[2] Seeded Ultrafiltration for Thorium Chain Assay in the D_2O, P. Trent, M. Moorhead, E. Hooper, R. Black, N. Tanner, B. Knox, M. Omori and S. Lilley, SNO-STR-92-090.

[3] The Liquid Scintillator β-α Coincidence Method, R.K. Taplin, M.E. Moorhead, SNO-STR-93-??.

[4] The Liquid Scintillator β-α Coincidence Method: Progress Report, R.K.Taplin and M.E.Moorhead, SNO-STR-93-??

[5] Ion Exchange Properties of Hydrous Titanium Oxide, K.H. Howard, Chemistry Part II thesis, Oxford University, 1994.

^{232}Th Decay Scheme

^{232}Th

$1.4 \times 10^{10}y$
$\alpha\ 4.08$

^{228}Ra

$5.77y$
$\beta^-\ 0.046$

^{228}Ac

$6.13h$
$\beta^-\ 2.14\quad \gamma 339, 911, 969$

^{228}Th

$1.91y$
$\alpha\ 5.52$

^{224}Ra

$3.66d$
$\alpha\ 5.79$

^{220}Rn

$55.6s$
$\alpha\ 6.40$

^{216}Po

$0.15s$
$\alpha\ 6.91$

^{212}Pb

$10.6h$
$\beta^-\ 0.57\quad \gamma 239$

^{212}Bi

$60.6m$
$36\%\quad \alpha\ 6.21$

$64\%\quad \beta^-\ 2.25\quad \gamma 727$

^{208}Tl

^{212}Po

$3.05m$
$\beta^-\ 4.99\quad \gamma 583, 860$

$0.30\mu s$
$\alpha\ 8.95$

$\gamma 2614\ (100\%)$

^{208}Pb

Figure 1: Simplified schematic of the ^{232}Th decay chain. Half-lives of all decays are given, together with Q-values in MeV of α and β^- decays and energies in keV of important γ radiations.

^{238}U Decay Scheme

^{238}U

$4.47 \times 10^9 y$
$\alpha\ 4.27$

^{234}Th

$24.10d$
$\beta^-\ 0.26\ \ \gamma 63, 92$

^{m234}Pa

$1.18m$
$\beta^-\ 2.21$

^{234}U

$2.45 \times 10^5 y$
$\alpha\ 4.86$

^{230}Th

$8.0 \times 10^4 y$
$\alpha\ 4.77$

^{226}Ra

$1599y$
$\alpha\ 4.87$

^{222}Rn

$3.82d$
$\alpha\ 5.59$

α

^{218}Po

$3.05m$
$\alpha\ 6.11$

α

^{214}Pb

$26.8m$
$\beta^-\ 1.02\ \ \gamma 352$

^{214}Bi

$19.7m$
$0.021\%\ \ \alpha\ 5.62$

$> 99\%\ \ \beta^-\ 3.27$
$\gamma 609, \sim 1\%$ b.r. to $\gamma > 2200$

^{210}Tl

^{214}Po

$1.30m\ \ \beta^-\ 5.49$
$\gamma 800, \sim 1\%$ b.r. to $\gamma > 2200$

$164\mu s$
$\alpha\ 7.83$

^{210}Pb

$22.26y$
$\beta^-\ 0.06$

^{210}Bi

$5.01d$
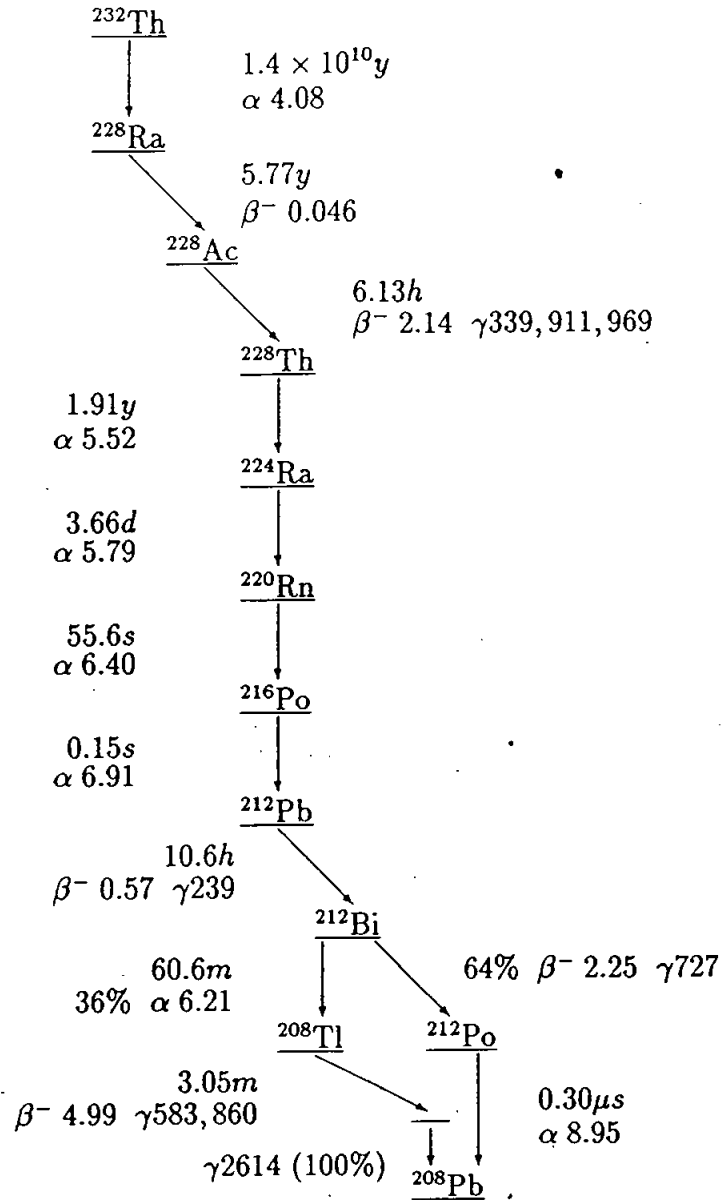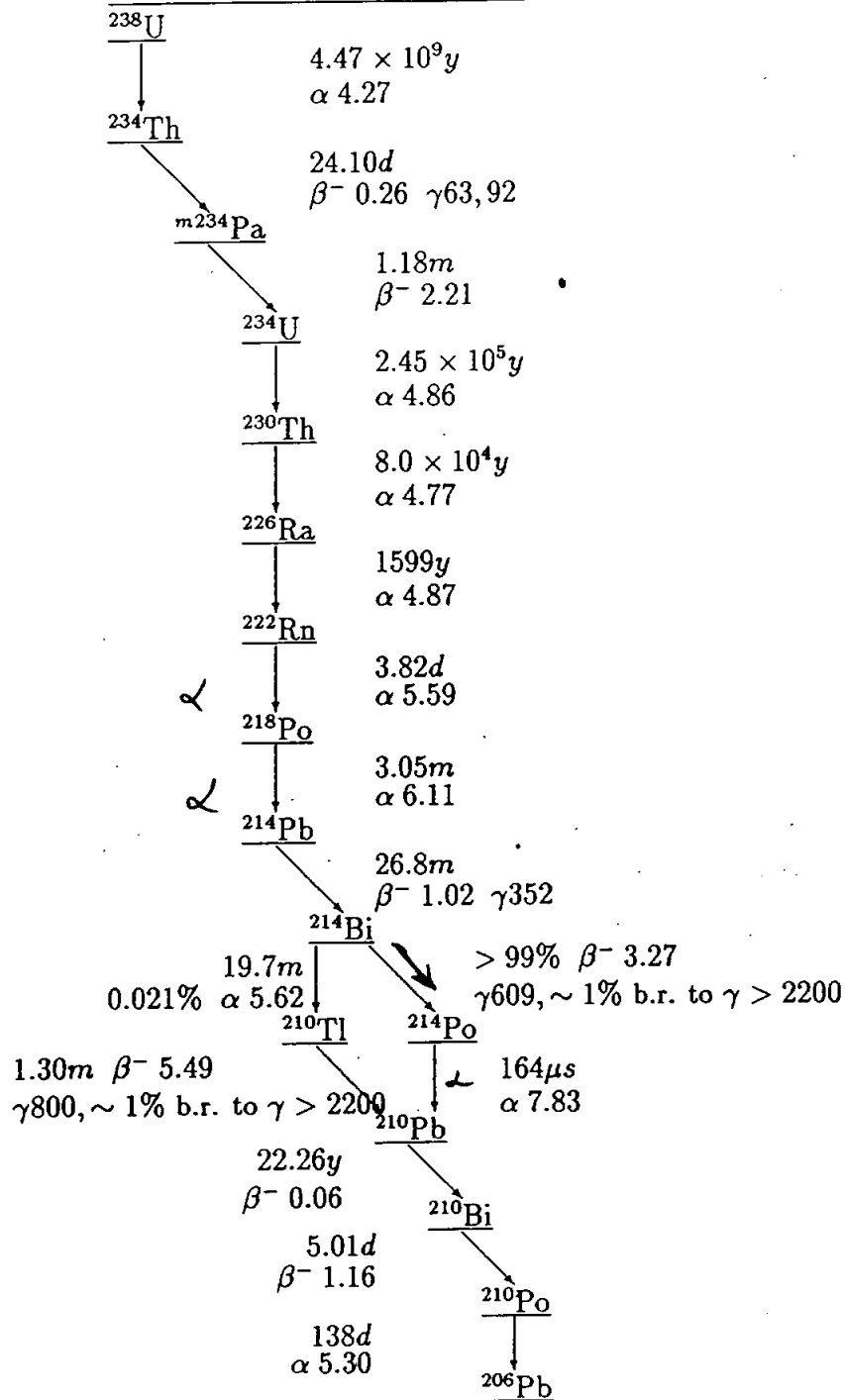$\beta^-\ 1.16$

^{210}Po

$138d$
$\alpha\ 5.30$

^{206}Pb

Figure 2: Simplified schematic of the ^{238}U decay chain. Half-lives of all decays are given, together with Q-values in MeV of α and β^- decays and energies in keV of important γ radiations.

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                        │    100 Tonnes of D₂O    │
                        └─────────────────────────┘
                                    │
┌──────────────────────────────────────────────────────────────────┐
│                  Primary Seeded UF Module                          │
│  2 x Amicon H26 MP01-43 - Membrane area = 5m² total, 0·1μm pore   │
│  Throughput = 200 l/min D₂O - Seeded with 0.5g/m² = 2.5g HTiO     │
│             Extraction Efficiencies >90% Ra                        │
│                              >90% Pb                               │
│                              >90% Th                               │
└──────────────────────────────────────────────────────────────────┘
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²²⁶Ra,²²⁴Ra Monitoring   ²²⁸Th ,²¹²Pb Monitoring

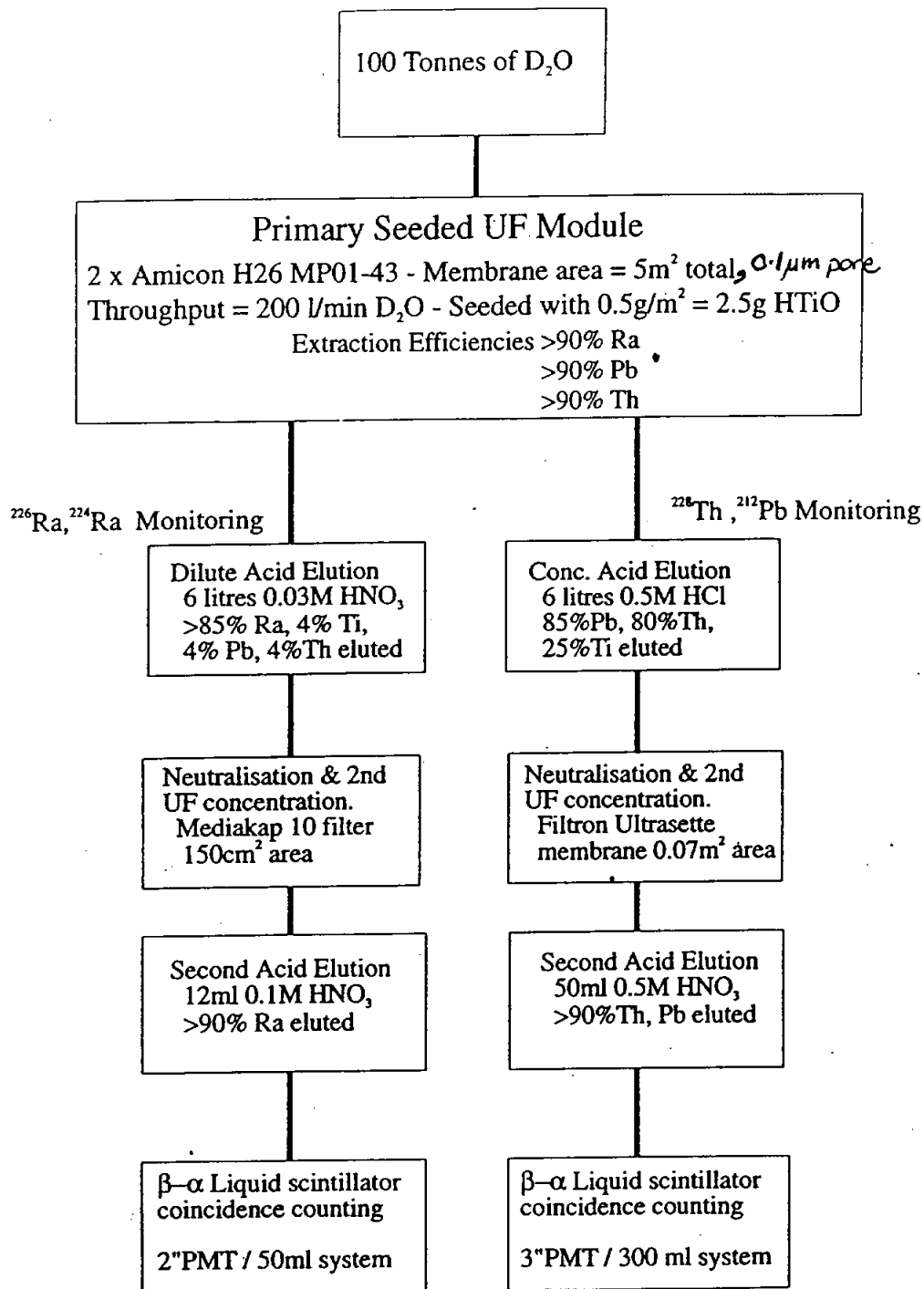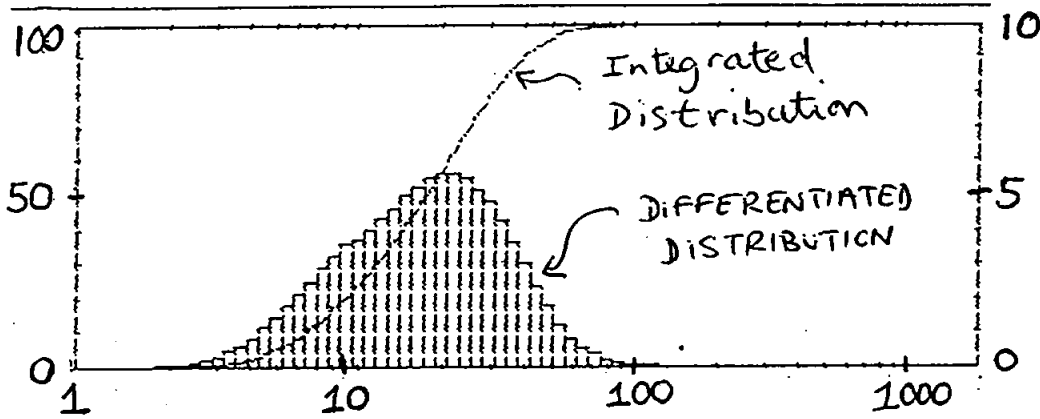|	
Dilute Acid Elution 6 litres 0.03M HNO₃ >85% Ra, 4% Ti, 4% Pb, 4%Th eluted	Conc. Acid Elution 6 litres 0.5M HCl 85%Pb, 80%Th, 25%Ti eluted
Neutralisation & 2nd UF concentration. Mediakap 10 filter 150cm² area	Neutralisation & 2nd UF concentration. Filtron Ultrasette membrane 0.07m² area
Second Acid Elution 12ml 0.1M HNO₃ >90% Ra eluted	Second Acid Elution 50ml 0.5M HNO₃ >90%Th, Pb eluted
β–α Liquid scintillator coincidence counting 2"PMT / 50ml system	β–α Liquid scintillator coincidence counting 3"PMT / 300 ml system

Fig. 3

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Dispersant  : Water
Additives   : None
Ultrasound  : None
Pump Speed  : 60
Stir Speed  : 50
Notes       : HTiO in Water
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3503 stnd 1u10547s

MALVERN   MasterSizer   SB.RD   Easy Mode   Wed 19 Jan 1994   3:25 pm

Percentage of total in Integral distribution

Percentage of total in differential distribution

Integrated Distribution

DIFFERENTIATED DISTRIBUTION

Particle size in microns (µm)

Upper	in	Lower	Under	Upper	in	Lower	Under	Upper	in	Lower	Under	Span 1.85
				124	0.1	101	99.8	11.6	7.8	9.48	21.7	
				101	0.3	82.3	99.5	9.48	6.9	7.78	14.9	D[4,3]
				82.3	0.6	68.3	98.9	7.78	5.3	6.39	9.6	20.90µm
				68.3	1.5	56.1	97.4	6.39	3.9	5.24	5.6	
600	0.0	492	100	56.1	3.1	46.0	94.4	5.24	2.6	4.30	3.0	D[3,2]
492	0.0	404	100	46.0	5.4	37.8	89.0	4.30	1.5	3.53	1.5	12.86µm
404	0.0	332	100	37.8	7.9	31.0	81.1	3.53	0.8	2.90	0.7	
332	0.0	272	100	31.0	10.0	25.5	71.0	2.90	0.4	2.38	0.4	D[v, 0.9]
272	0.0	224	100	25.5	11.2	20.9	59.9	2.38	0.2	1.95	0.2	38.00µm
224	0.0	183	100	20.9	11.1	17.1	48.7	1.95	0.1	1.60	0.1	
183	0.0	151	100	17.1	10.3	14.1	38.5	1.60	0.1	1.32	0.1	D[v, 0.1]
151	0.0	124	99.9	14.1	9.0	11.6	29.4	1.32	0.1	0.50	0.0	6.51µm
Source = :Sample				Beam length = 2.4 mm				Model indp				D[v, 0.5]
				Residual = 0.249 %								17.55µm
Focal length = 300 mm				Obscuration = 0.2508				Volume Conc. = 0.0488%				
Presentation = stnd				Volume distribution				Sp.S.A 0.4665 m²/cc.				Shape OFF

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Fig. 4: Particle Size Distribution of
HTiO, measured by diffraction of
light.