SNO-STR-91-049

STATUS OF THE ACRYLIC R&D PROGRAM

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INTRODUCTION: The following is a summary of the status of the radioactivity (Earle), mechanical, and optical (Doe) aspects of the R&D program for the acrylic vessel.

RADIOACTIVITY: Progress has been slow since the Vancouver meeting for a number of reasons which will become clear under each section.

1) NEUTRON ACTIVATION (NAA): The NRU reactor has been shut down for an extended period and no new samples have been irradiated at CRL. We have redesigned the irradiation holder and are putting the protocol in place to do some irradiations in the lower flux of NRX. One sample of acrylic was machined into a beaker shape at CRL, shipped to Guelph for irradiation and gamma counting and then shipped back to CRL for vaporization and mass spectroscopy (MS). The NAA result was 14 ppt and the MS value was 17 ppt.

2) ALPHA SPECTROSCOPY (AS): Several more spiked samples and standards have been analyzed giving evidence for factors of three variations in recovery efficiencies. Steps are being taken to make these more consistent. These variations do not effect the earlier reported conclusions that the 232Th to 228Th ratio in acrylic samples is 1 +-0.5 and that the 226Ra to 238U ratio appears to be less than 1. With improved recovery efficiencies it should be possible to decrease the uncertainty in the 226Ra to 238U ratio and to make meaningful comparisons between measurements by the three methods of MS, NAA and AS.

3) MASS SPECTROSCOPY (MS): The number of samples analyzed was low this quarter because;

A) We installed a second fume hood in the vaporization room.

B) We purchased and installed a band saw dedicated to acrylic cutting.

C) There was a staff change in the MS section followed by a realignment of protocol for the MS. Previously samples were sent for TIMS as they were vaporized and there was a good turn around time for the results but only at the rate of three samples per week. We have recently arranged for NRC to do ICPMS on a contract basis, involving about 10 samples at a time but the turn around is two to

three weeks. We hope to make use of both the TIMS and ICPMS to improve our net throughput.
D) We obtained some poor quality acids which made some of our measurements less interesting.
The results (Fig 1 and 2) that we do have continue to imply that much of the acrylic is in the range of 5 to 10 ppt Th which is a factor of 2 to 3 higher than the Guelph NAA results on six samples from the one sheet from one supplier. The average values of some sheets are lower than others.
The factor of 1.5 to 2 difference between the measurements on five samples made by NAA at Guelph and by MS at CRL continue to worry us and we have not yet completed our exploration of possible contamination during CRL handling of the acrylic. Our intention is to approach the acrylic suppliers to explore ways to reduce the Th content but this action has been delayed awaiting a measuring technique in which we have confidence.

4) MEASURING TECHNIQUES: Of the three techniques the MS route is the only viable one for many samples and for the SNO program.
The AS technique has a sensitivity for Th and U at the 0.5 ppt if 10kg samples are vaporized which takes one week to do. The Th, U and Ra electroplating takes several weeks and the counting time is also several weeks. Per sample this is therefore expensive, the turn around time is long (more than a month) and unless facilities are duplicated many times the throughput is low (one sample per month).
The NAA technique also has a sensitivity at the 0.5 ppt level for Th but up to an order of magnitude worse for U due to half life differences. Sample preparation takes only a day but then we have to wait two weeks for short lived background reduction before a two or three day counting session. Turn around time is therefore poor (several weeks) and the throughput with one gamma counting facility is not good (one sample per week). This technique appears to be the most reliable however.
The MS technique also has a sensitivity at the 0.5 ppt level for Th and U for 1 kg samples. Backgrounds from the acids and containers require such large samples but then we want to average over some reasonable sample of the acrylic so the 1 kg sample may not be a disadvantage. The sensitivity of the MS instruments is at least an order of magnitude better. Vaporization takes a day, rinsing the tube several times takes a day and the TIMS takes a day (ICPMS takes an hour). Turn around time is therefore less than a week and throughput with one set of facilities is one per day. If we had the man power,

two sets of facilities could be operational at CRL within a few weeks resulting in a throughput of up to 10 per week.

MECHANICAL R&D: The short term mechanical properties of acrylic are well documented for our requirements. The mechanical R&D program focuses on those aspects which are special to the SNO project; long term water immersion, acrylic and bond properties. A major uncertianty in all this work is the simulation of the long term aging. This has been attempted by immersion in pure water at 60C for 40 days. Assuming that aging processes are accelerated by a factor of between 2 - 3 for every 10C rise in temperature, then this immersion is equivalent to 10 years at 10C. To demonstrate that this holds true for acrylic is non-trivial, therefore, wherever possible we rely on real long term data, even though the environmental conditions do not exactly duplicate SNO. This is generaly conservative since the SNO environment is considered to be benign for plastics.

1) AGED/UNAGED BOND STRENGTH: Acrylic from 3 different sources have been bonded by Reynolds Polymer using their proprietary techniques. These bonds were subject to flexural, tensile and compressive tests and had strengths of about 70% of the parent material - a respectable result. 50% of the samples were aged as described above. Strengths were reduced by 25% - 30% with no statistically significant difference between the acrylic suppliers. The results are described in UCI Neutrino-89-10 and confirm that the long term properties assumed for the acrylic in the design studies are conservative.

2) BOND STRENGTH VS. WIDTH: Because of the nature of polimerisation, it is expected that the bond strength is both a function of the thickness of the sheets being bonded and the width of the bond. UVT acrylic sheets of 1" and 2" thickness were bonded by Reynolds Polymer with 5 bond widths, between 1/32" to 1/4 ". For both sheet thicknesses the maximum tensile strength (70% of parent material) resulted from a 1/8" bond width. This has been incorperated into the vessel fabrication specification. Since the vessel will now contain some 4" (and possibly 3") material, these tests are being repeated for thicker sheets. The results are described in SNO-STR-91-3.

3) BOND STRENGTH VS. FINISH: Bond joints are usually finished by machining and polishing. Apart from a good optical finish (not essential for SNO) this also eliminates surface roughness and discontinuities which can act as stress risers, weakening the joint. This is a labor intensive, time consuming process which can

potentially introduce additional radioactive materials into the acrylic. Since the SNO vessel will have in excess of 1,000' of bonds, we would like to avoid this process. Jerry Stachiw has successfully demonstrated an idea of Hamish Robertson, whereby silicon rubber overlays are useu to contain the bond material. This technique conforms to dimensional variations of the acrylic sheet and provides a smooth finish. This technique is being further refined by Reynolds Polymer. In order to quantify the importance of surface finish and sheet misalignment, approximately 100 bonded flexural test samples are being fabricated by Reynolds. The results, expected in mid-July, will be incorperated into the fabrication specifications.

4) CREEP IN UNAGED/AGED ACRYLIC: Two, 250 liter, constant temperature water baths, each containing 32 tensile stress test coupons equiped with strain gages under computer readout are being use to study the creep of aged and unaged acrylic. Monitoring the creep of a material over several months allows the the prediction of its behavior over periods of years.

Time-to-failure envelopes and creep characteristics were obtained for unaged tensile test samples. Similar samples were aged for the equivalent of 10 years and instrumented with strain gages. It was found that the aged samples rapidly crazed and suffered premature failure when subject to tensile stress. This effect is now the subject of an independent study (see item 6 below).

As a result it is unlikey that the long term creep properties of acrylic which has been immersed in water will be avaliable for the detailed analysis of the vessel. Instead, existing 10 year data (Stachiw, June '89) from acrylic exposed to an outdoors environment will be used. This data is considered to be conservative. However, efforts to obtain creep data for acrylic aged in water are continuing.

5) WATER ABSORPTION: Acrylic absorbs water, and in doing so swells. Accelerated water absorption tests (simulating 10 years at 10C) have been carried out on Rohm, Polycast and Reynolds Polymer material. The rate of water absorption was roughly equal for all 3 materials. Interestingly enough, the "slush cast" material of Reynolds Polymer became opaque in the accelerated aging tests, as did the bonding material. This does not happen in real life where aquarium windows have been in service for 10 years, and is an indication of the suspect validity of the accelerated aging process. If there is a difference in the absorption of heavy and light water, (and therefore swelling of the acrylic) this could result in stresses developing in the shell of the vessel. Acrylic from 3 different sources has been exposed to light and heavy water for approximately

2 years with no discernable difference in the water absorption. The earlier results can be found in UCI Neutrino-89-10, a full report is being written

6) HYGROTHERMALELASTICITY: This impressive title referes to the stress that develops when a homogeneous solid (acrylic) absorbs an impurity (water). This is what caused the aged samples in item 4 to craze. We can expect a similar effect when the vessel dries out between the light water and heavy water fill. The magnitude of this stress is presently unknown and may be a problem for the vessel.

To study this problem, strain gages are being slush cast into a blocks of acrylic which will be subsequently exposed to water and the stress build-up monitored during absorption and desorption. The samples will be ready by the end of June and the tests will take 6 weeks. It is not known if these tests will provide the neccessary data since no-one has conducted this type of test before in acrylic.

If there is a problem with stress building up during the light water - heavy water change over then it may be nessessary to keep the humidity in the cavity high. This in turn could impact the design of the water treatment system, in which case we will need to know if there is a problem by fall this year.

7) LOCKED IN BOND STRESS: When the bonding material polimerises it contracts by 10% - 15%. If the panels being bonded are unable to move (as in SNO) stress will develop. This has been demonstrated in earlier bonded spheres which always developed crazing at the bond joints. The exact level of stress is unknown, except it is probably in excess of 1,000psi long term crazing studies by Stachiw.

In order to quantify the stress level, strain gages are being embedded in a bond joint which contains metal shims to prevent the panels from moving. The strain gage will be read out as the bond polimerises.The samples will be ready by the end of June and the tests will take 1 week. It is not known if these tests will provide the neccessary data since no-one has conducted this type of test before in acrylic.

8) SUSPENSION ROPE: The long term creep of Vectran and Kevlar rope has been measured for unaged and aged samples. The decision was made to go with Kevlar on the grounds that its general properties (creep, tensile strength etc.) met the projects needs and a lot of long term data already existed for the rope. A report is being written on the results of the rope test, its finding are generaly in agreement with existing commercial test data. The remaining uncertianties for the rope suspension are the radioactivity and optimisation of the rope groove, for which a photoelastic model may be made.

OPTICAL R&D: If it is decided that the normal mode of operation of the vessel will be in compression, then it will be neccessary to thicken the walls to prevent the shell buckling, this in turn will place a new emphasis on the optical transmission of the acrylic. The acrylic for the SNO vessel will be both thermoformed and annealed. These excursions to elevated temperatures can effect the transmission (see SNO-89-11). Samples of acrylic from prospective suppliers must be subject to the temperature cycles of potential fabricators. In order to achieve thermal stability certian chemicals are added to the acrylic,which may influence the radioactivity. A temperature concontroled oven and other hardware have been aquired by CRL for this purpose. This information is essential in the selection of the acrylic supplier and should be inhand and understood by this fall.

A program has been written at CRL which culculates a figure of merit for a given vessel thickness. It treats multi thicknesses in a simple fractional way. If the number of photons detected from a Cerenkov event with no water attenuation and with the purchased PMT's is 133 then the numebr detected in the heavy and light water in place (attenuation as reported in SNO-12-87) is 100. The number detected with 2" of the best heat treated acrylic is 75. Thicker acrylic or acrylic with poorer transmission will worsen this figure of merit.

OUTSTANDING ITEMS: A major item for which a game plan has not been formulated is monitoring the vessel. Several options are avaliable;

Acoustic Emmission

Ultrasonic Imaging

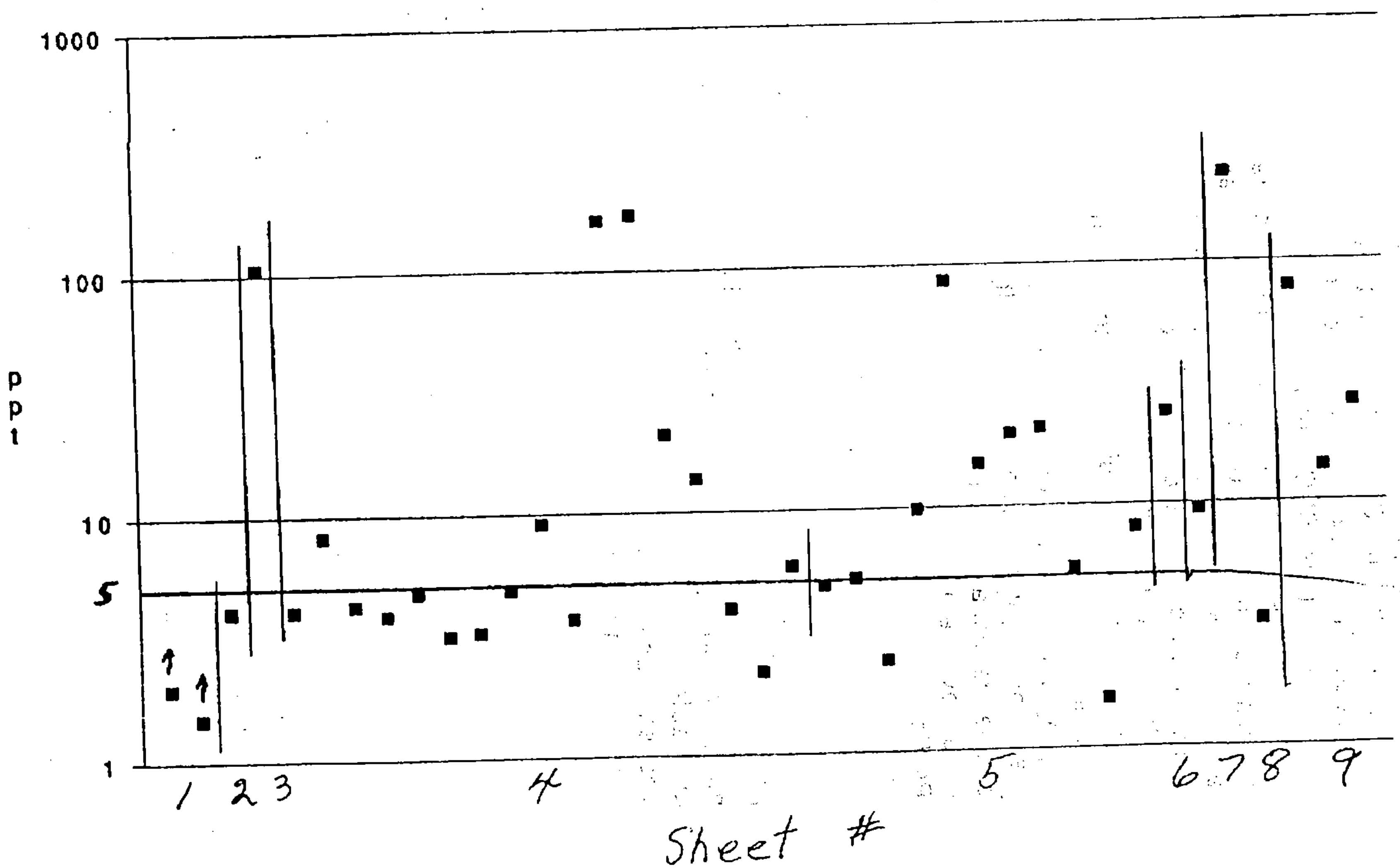
Heavy/light water concentrations

Selected strain gage location

No serious attempt has been made to evaluate these options, look at how they may compliment one another, how they would be integrated in the data acquisition system or what is commercialy avaliable. The timescale for this work is not critical.

FABRICATOR R&D: Considerable R&D is required of the vessel fabricator. A technique has to be developed to make the vertical and horizontal bonds - current techniques usualy bond in a horizontal plane. It is desirable that these techniques produce bond joints that do not require further finishing - this has radioactive as well as dollar implications. It would be a big advantage to start this R&D now by sole sourcing the fabrication contract. The acrylic vessel committee has issued a recomendation concerning this.

Th in Rohm acry. by MS Oct 89-Apr 91



Th in Poly. acry. by mass spec.,Oct 89-Apr 91