Optical & Th Measurements of RPT Acrylic Samples.

E. D. Earle, R. Deal & E. Gaudette

SNO-STR-94-010
April, 1994

Section 1: Introduction:

The optical, mechanical and radioactivity properties of the acrylic sheets purchased from Polycast have been measured[1]. The material satisfied the SNO specifications and the sheets were graded as vessel, spare or qualification grade. The qualification sheets are the poorest and are being used to qualify the RPT fabrication procedures. RPT has to thermoform, machine, bond and sand the sheets. The properties of the acrylic after RPT handling must be checked.

Samples handling by RPT in their California plant have been sent to CRL where optical and neutron activation analysis measurements have been performed. In addition, sheets prepared in California were used to make an eight panel wall and three samples from this wall have been examined. Two sheets have been thermoformed in the new Grand Junction oven and samples from these two sheets have also been measured at CRL.

Section 2: Results:

2.1 California Sheets.

Two shipments of California handled acrylic were received. The first contained three samples, each 6' by 1' by 2", from sheets 49-03 and 47-02. The sample from 47-02 had a bond down the middle and the two from 49-03 were just thermoformed and machined samples. The second shipment contained samples from sheet 09-12 with and without a mark-off from the mold. It had been noticed by Peter Doe that sections of the mold used in thermoforming were leaving an orange peel appearance on the surface of the acrylic which could be contributing Th and degrading the optical property of the acrylic.

2.1.1 Th content.

The Th concentrations in pg/g and in pg/in^2 are listed in Table 1. The table lists the CRL identification and details of the coupon including the sheet identification, whether it was in contact (outside) or away (inside) from the mold, the presence or absence of a bond or a mark-off, its weight, its optical surface area and the duration of the gamma count. Figs. 1 & 2 at examples of the spectra, one with a small error and one with a large error.

Table 1. Th concentration in RPT coupons from California.

ID	Sample	wt in g	hrs	surf area in2	ppt	pg/in2
211	4903 inside	933	24	48	0.44 ±.06	8.6
212	4903 outside	913	23	48	0.49±.05	9.5
213	4903,4702 inside	913	40	48	0.16±.05	3
214	4903,4702 outside	954	24	48	0.33±.07	6.7
223	4903 inside	931	23	48	0.18±.05	3.5
224	4903 outside	902	28	48	0.08±.03	1.5
225	4702 with bond,inside	406	36	27	0.39±.19	7.3
226	4702 with bond,outside	519	29	27	0.95±.3	18.3
230	4702 with bond,no surface	504	22		0.28±.17	
239	4903 with mark off	216	22	18	0.48±.13	5.8
245	4903 A sample	882	46	48	0.53.03	9.7
246	4903 B sample	939	28	48	0.2±.03	3.9
247	4702 with bond,surface	514	22	27	0.31±.2	5.9
248	0912 with mark off	112	68	11	2.2±.2	22
249B	0912 with mark off	104	24	11	0.7±0.14	6.6
249A	0912 no mark off	415	64	22	0.04±.026	0.75

The following observations can be made.

- There is significant variability between coupons in the Th per gm of material and in the Th per unit surface area. This makes generalizations uncertain.

- The fabrication procedures adds Th to the acrylic. The bond surface has the most, followed by the bond itself (or the machined surfaces) and then the thermoformed surfaces. While any additional Th is bad the amount is well below the specifications and should be considered acceptable. A very rough ratio might be virgin acrylic - 0.06 ppt, thermoformed - 0.25 ppt, bond core - 0.30 ppt, bond surface - 0.7 ppt. SNO would certainly like to have this Th reduced.

Fig. 1. Spectrum 941220 ID 246

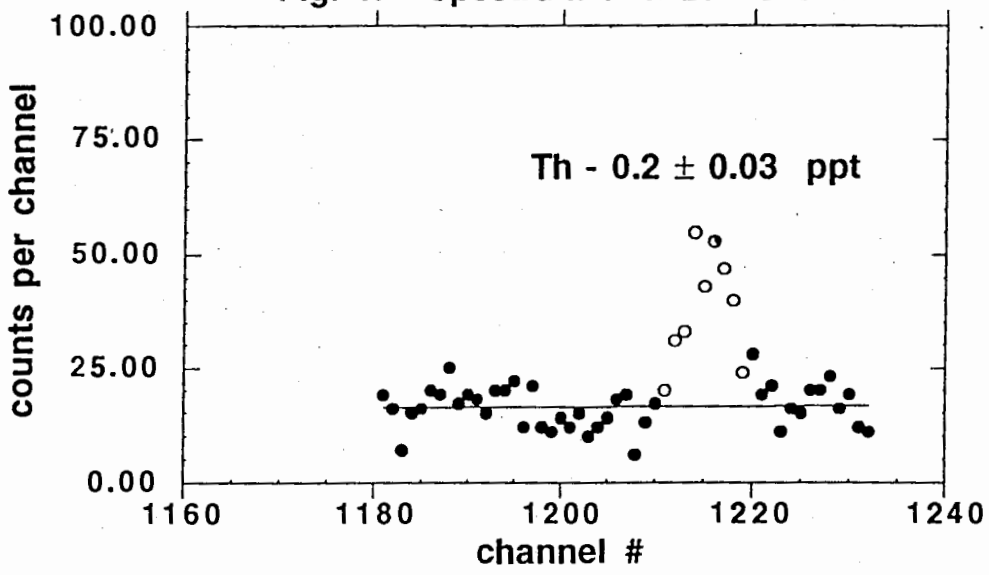
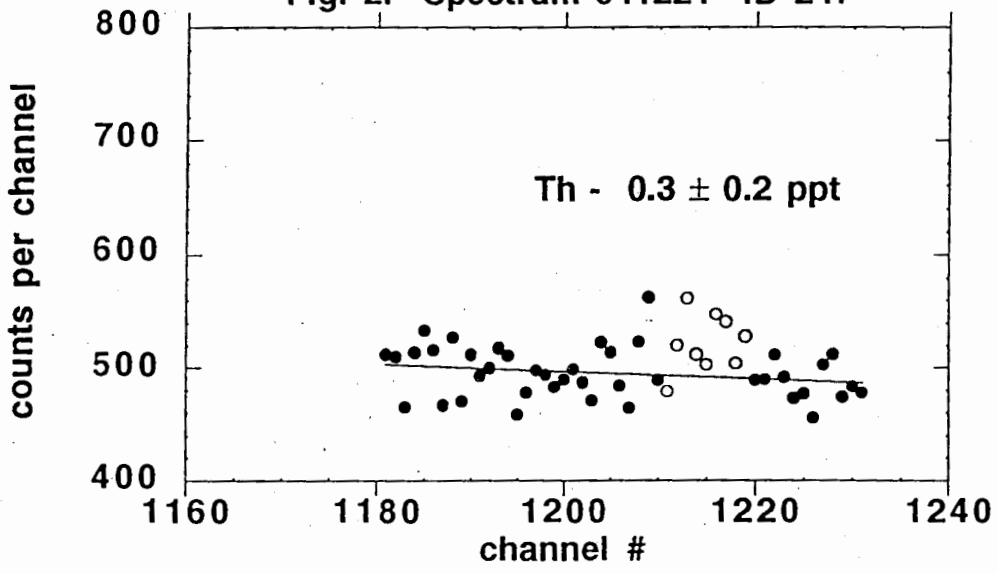


Fig. 2. Spectrum 941221 ID 247



- The most obvious effect is that the coupons with a bond in them or a mark-off have much higher errors. This means that contamination has been introduced into these coupons which gets neutron activated and increases the background under the Th peaks. It does not mean that there is more Th in these coupons or that this extra material is bad for SNO. This material may not be in the syrup, it may be due to machining, sanding and mark-off.

- There is no difference between coupons in contact with the mold (outside) and coupons not in contact with the mold (inside).

- The Th on the surface appears to vary by a large amount. This is probably a real effect but the measurements also have a systematic uncertainty perhaps of the same order as the statistical error. One has to place a larger uncertainty on the two smallest coupons(248 & 249B) with mark off. Sample 248 is high but 249B, while 10 times 249A, is like many others. There is no conclusive evidence that the mark off is causing a significant Th problem.

- The surface area of the thermoformed sheet is much larger than the areas of machined, bonded and sanded surfaces and so, if these measurements are indicative of all fabricated panels, the net Th inventory of the vessel will be determined by the extra Th added during the thermoforming and not during the machining, bonding and sanding. Efforts to reduce the Th inventory should, initially concentrate on the handling and thermoforming stage.

2.1.2 Optical properties:

Optical measurements were made on coupons from each of these samples. The figures[2)] of merit (F) are listed in Table 2 where the F's of the original Polycast sheet are shown for comparison. The experimental uncertainty on each number is about 0.004. Three different optical measurements through the bond region of two coupons are also tabulated.

Table 2. Figure of merit of RPT samples.

Sheet	Original F	RPT coupon	Comments
49-03	0.711	0.722	thermoformed
		0.725	thermoformed
47-02	0.695	0.697	thermoformed
		0.721, 0.719, 0.514	bond section
		0.525, 0.522, 0.388	bond section
09-12	0.74	0.731, 0.733	no mark-off
		0.732, 0.724	mark-off
		0.724, 0.737	mark-off

There is no deterioation of the optical properties from the RPT thermoforming even for coupons with a mark-off or orange peal

pattern but the optical quality of the bonds is inferior. These observations are consistent with earlier measurements on RPT material thermoformed and bonded in Santa Ana and underground at the 4600' level[2].

2.2 Grand Junction Samples:

Two samples from Grand Junction thermoformed sheets 49-11 & 49-12 have been received at CRL. The >1 micron dust levels, as measured by James Dodson[3], before and after the themoforming were 350k to 400k for 49-11 and 250k to 300k particles per cu ft for 49-12. These densities were on the low side of the range measured by Earle[4] during his visit to RPT in early Febuary '94 after the fan had been going for 15 minutes i.e. 200k to 500k. Note that the dust level for 49-12 was slightly lower than for 49-11. This dust level is almost two orders of magnitude higher than the dust level outside the Grand Junction building[4] and than the dust level measured in the California oven[5].

2.2.1 Th Content

Four 12" by 2" by 2" coupons were neutron irradiated on March 20. All non optical surfaces were machined off prior to vaporization and counting. The coupons were separated into inside and outside components (meaning in contact with the mold and not in contact with the mold). The measured Th levels in ppt are listed in Table 3.

Table 3. Th (pg/g) content in Grand Junction Coupons.

sheet	inside	outside
49-11	0.3 (.04)	0.51 (.03)
49-12	0.03 (.01)	0.15 (.01)

The average from 49-12 is 20% of 49-11. This difference is significant and it would be great if the low Th concentrations in 49-12 could be reproduced but the reason for the difference is not obvious. The dust levels during the 49-12 thermoforming may have been only slightly better and the dust levels are much higher than the California dust levels whereas the Th in the California themoformed sheets are similar to the Grand Junction thermoformed sheets. One should remember that the Th level in the dust may not be correlated with the total dust levels.

2.2.2 Optical Measurements

Only two optical measurements have been done on the Grand Junction thermoformed sheets. The figures of merit are listed in Table 4.

Table 4. Figure of Merit of Grand Junction Coupons.

sheet	before thermoforming	after
49-11	0.72	0.728
49-12	0.717	0.733

These differences are within the uncertainty of the measurements, a result consistent with many previous measurements which indicate that there is no significant deterioation after thermoforming.

The material was examined visually on Mar 21. There were a significant number of blemishes, some black and some light in color, which could be dust embedded in the surface, in particular, the surface in contact with the mold. Some of these "stuff" was completely and partly removed by rubbing with a Q tip. Sample 49-11 appeared worse than 49-12.

2.3 Wall Samples:

Three 20" by 12" samples cut from the first acrylic wall fabricated by RPT were sent to CRL for optical and Th measurements. These samples are identified as #21, #22 and #23 by RPT and the vessel committee on drawings of the wall[7].
#21 was a sample containing the vertical bond joint between SQW-106-01 and SQW-106-02 (or 48-03 and 48-09), two of the panels in the top row of the qualification wall.
#22 was a sample containing the vertical bond joint between SQW-106-02 and SQW-106-03 (or 48-09 and 48-11), two of the panels in the top row.
#23 was a sample containing the vertical bond joint between SQW-101-01 and SQW-102-01 (or 93-01 and 49-03), two panels in the bottom row of the wall.

Sketches of the three samples are shown in Fig.1. The dotted line represents the location of the bond and the solid lines depict

coupons cut for optical (OP) and radioactivity (NAA) measurements. In every case one of the outside edges was machined before the wall was constructed and the other three edges were rough cut when the sample was removed from the wall. The orientation of these samples in the wall was not recorded and so there is uncertainty about which edge is the machined edge.

2.3.1 Th Content

A total of seven coupons were taken from the three samples for neutron activation and gamma counting. After irradiation all non optical surfaces were milled away before the coupon was cut into smaller pieces for vaporization and gamma counting. These coupons are identified in Fig. 3 as A, B and C. Two of the coupons, representing virgin material in samples 21 and 22, consisted of two pieces cut from different regions, and hence sheets, of the sample. The coupons were chosen to measure the Th in the virgin material, the sanded area and the bond joint. The Th content, in pg/g, of the coupons is listed in Table 5. .

Table 5. Th Content of Wall Coupons.

ID	Sample	Th	Identification
268	21A	0.14 (0.04)	virgin material
267	21B	0.7 (0.04)	sanded region
266	21C	4.5 (0.6)	sanded & bond region
257	22A	0.1 (0.02)	virgin region
256	22B	0.33 (0.04)	sanded region
254	23A	0.06 (0.02)	virgin region
255	23B	3.0 (0.3)	sanded & bond region

Several of these coupons had voids in the bond which extended to the surface of the sample. In those cases the milling after irradiation had to be deep enough to remove the void and thereby any trapped Th on the surface of the void.

A visual inspection of coupon 23B before it was sent for irradiation clearly showed several large black specks in the bond. It may be that the high Th content and very high background, two orders of magnitude higher than some of the others, was due to this

visually obvious contamination. On the other hand coupon 21C also had a high Th content and high background with no obvious source. The bond joint did contain bubbles.

Several of the coupons, in particular the three samples, 21 A, B & C had very high backgrounds on some of the edges after irradiation. These samples had to be stored for many days before they could be milled and processed for gamma counting.

Except for the dirty bond coupon, these Th levels are similar to those measured in other samples that have been handled by RPT. The previously observed increase in Th due to the thermoforming, machining, bonding and sanding performed by RPT is confirmed.

2.3.2 Optical Measurements:

Two coupons were removed from each sample for optical transmission measurements, one from the virgin sheet (E) and one from an area near the bond joint which had been sanded (D). Since the sample orientation in the wall is unknown the coupon pair can be from one of two Polycast sheets. The figure of merit (F), representing the fraction of Cerenkov light getting through the acrylic vessel, for each coupon is listed in Table 6 where it can be compared with the figure of merit of the original Polycast sheet[1].

Table 6. The Figure of Merit of Wall Coupons (E & D)

Sample	Polycast ID	Polycast F	E	D
21	48-03, 48-09	0.72, 0.70	0.72	0.66
22	48-09, 48-11	0.70, 0.71	0.72	0.63
23	93-01, 49-03	0.68, 0.71	0.70	0.65

The optical quality of the wall coupons taken from an area away from the sanded area are all similar to the original sheet but the coupons from the sanded areas are all of poorer optical quality. The deterioation is about 10% and is similar to the deterioation measured in sanded samples previously received from RPT[2]. SNO should have RPT minimize the sanded surface areas in the vessel.

Section 3. Conclusions

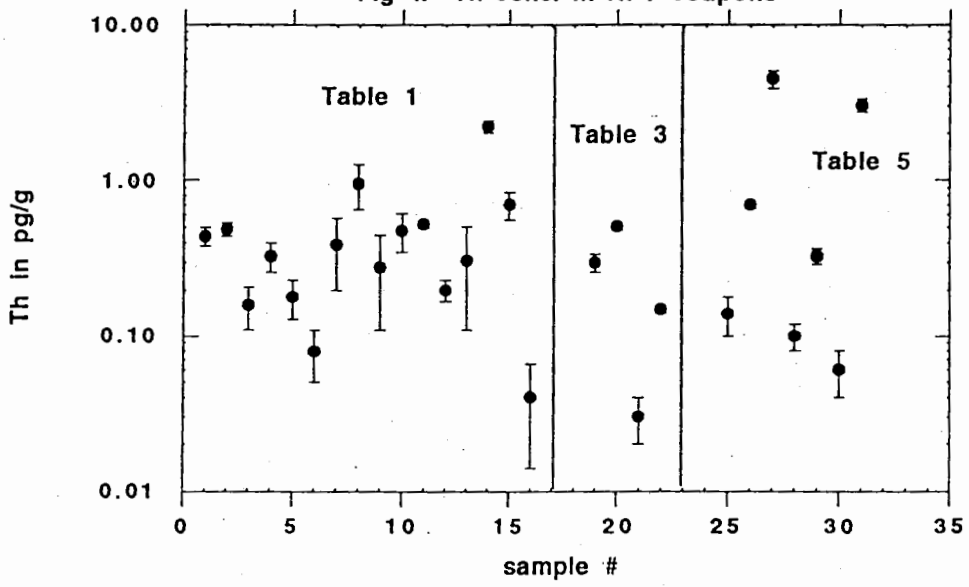
- Areas sanded by RPT have 10% poored light transmission than unsanded areas. Such area should be kept to a minimum. The light transmission of the bonds is variable and can be quite poor.

- The thermoforming, machining, bonding and sanding introduces additional Th into the acrylic. The measured concentrations listed in Tables 1, 3 & 5 are plotted in Fig. 4. However this Th, while undesirable, must be considered acceptable when integrated over the entire vessel. The supposition that airborne dust in the oven during the thermoforming is the most serious source of this Th contamiation has not yet been confirmed experimentally.

- There are isolated examples of inferior workmanship from a dirt point of view which the fabrication team must strive to eliminate. Visual inspection of bonds and surfaces show significant variability in the quality of the finished product and the large variations in Th content may also be a reflection of poor quality control.

- Only a small fraction of the test samples have been measured. SNO should consider ways to make its inspection program more extensive.

Fig 4. Th conc. in RPT coupons



1) Polycast Acrylic Sheets.
E. D. Earle, R. Deal & E. Gaudette
SNO-STR-93-042, revised and expanded Jan '94

2) Evaluation of Optical Properties of Acrylic Samples from Different Suppliers. E. Bonvin & E.D. Earle, SNO-STR-92-068

3) Memo to Earle from Dodson, Mar 8, 1994

4) Memo to vessel committee from Earle, Feb 15, 1994

5) Measurement of Airborn Dust at the RPT Facility in California, B. Brewer & P. Doe, SNO-STR-93-053

7) Memos from Doe and RPT concerning all wall samples.