

SND-STR-90-25

MINUTES OF THE ACRYLIC RADIOACTIVITY  
GROUP

C.R.N.L. Tues Jan 17, 1990

January 19, 1990

PRESENT:

E.D. Earle, Chairman  
R. Deal  
T. Clifford  
G. Milton  
H. Lee  
P. Jagam  
J. Taylor  
W. Edwards  
M. Hurteau  
L. Green  
N. Elliot  
F. Miller  
E. Cooper

1 Morning

Earle: Reported on discussions with Los Alamos chemists. Fowler has suggested that free O<sub>2</sub> in the acrylic may be making stable Th compounds at high temperatures that do not dissolve.

Milton: Spiked runs should show the same effect.

Elliot: Has acrylic production process been examined?

Earle: A visit have been made to the Polycast. Some possible problem areas have been identified, for example the storage tanks for the monomer are made of carbon steel. A preliminary Q.A. plan has been laid out.

-: It is planned to irradiate the monomer with neutrons.

Milton: We should try to incorporate a spike of Th in the acrylic before polymerization to give a more realistic measure of our recovery efficiency.

Earle: We should try vapourizing neutron irradiated acrylic (with high Th content *i.e.*, 50 ppt) to get a positive comparison between the neutron irradiation and the mass spectroscopy methods.

-: Los Alamos plan and capabilities. They can do neutron irradiation and  $\gamma$ -counting. They have a well-type Ge detector and an anti-Compton shield. They are going to try to work with 6 g samples. They have a Re filament mass spectrometer and will try mass spectroscopy. (There is a Re filament mass spectrometer at Carleton — would this be of any use to us? They don't have very clean handling procedures.) They can do delayed neutron spectroscopy. They plan to look for correlations between Th/U and other contaminants in the acrylic.

Green: Is familiar with the Los Alamos mass spectrometry people and he will be visiting there in the near future. He will make contact.

Earle: Suggested program plan:—

- : neutron activation: we should do as many samples as we can. The chemical separation take a lot of time so the following procedure can save us wasting time on dirty samples. A preliminary  $\gamma$ -count can be done by Edwards after which the unseparated sample can be sent to Queen's to be counted on the Compton suppressed spectrometer. If this is not sensitive enough then the sample will be returned to CRNL for chemical separation. Clifford will do the  $\gamma$ -ray spectrum analysis.
- : mass spectrometry: continue to clean up procedure with multiple washes *etc.*
- : neutron activation of monomer samples
- :  $\alpha$ -counting: 5 reproducible 15 kg samples to be run. Radium recovery efficiencies to be confirmed.
- : Queen's Compton-suppressed  $\gamma$ -facility to be duplicated at CRNL with either a large planar or a well-type Germanium crystal. There was a discussion about which option is best and also consideration of the merits of NaI as an alternative.
- : A 30g sample of acrylic will be neutron irradiated and then vapourized in Edwards' fume hood. The rinse will be done in L. Green's clean air fume hood which needs to have its exhaust repaired and also needs to have some suspect components replaced. Earle will cover reasonable costs for repair.

Jagam: Showed separate spectra of distillate and residue of irradiated acrylic.

Deal: Suggested we should use adjacent pieces of acrylic for neutron irradiation and mass spectrometry.

Earle: Agreed but pointed out that we will need a dirty sample of neutron activated acrylic in order to get enough signal for the mass spect.

Edwards: Suggested that we might use *several* 35 g pieces for mass spect. to get the signal up.  $\gamma$ -Counting would allow us to select the most promising pieces for analysis.

Earle: We need to set up a system for identifying the origin of each piece of acrylic that is analysed.

-: R. Deal is cleaning acrylic samples in  $\text{HNO}_3$  before vapourization and this is causing fires again.

Green: Suggested HCl instead.

Edwards: Cl may cause a problem after irradiation. Washing time should be reduced to about half an hour; ultra-sound might help.

Hurteau: Suggested washing surface away with chloroform.

Edwards/Green/Cooper: This may give a sticky mess; spraying chloroform and then air drying might help this.

Jagam: Showed results of neutron irradiation and  $\gamma$ -ray counting for various samples from the same sheet of acrylic. Elemental fluctuations by a factor of 10 were observed

Green: For multi-sample loading capability in the mass spectrometer, it would be better to get a new machine than to modify the present one.

## 2 Late Morning

Clifford: The wide scatter in previous results could be due to problems in the sample preparation or due to heterogeneity of Th. Is there evidence or are there some definitive experiments we could do to give proof positive that the real problem is heterogeneity?

Edwards: Jagam's results show heterogeneity for several elements; it should not be surprising if Th follows the same pattern.

Jagam: Made presentation of neutron irradiation results carried out at the McMaster reactor. Various sources were used, rods, sheets *etc.*, and the method of handling the samples was varied. Al foil was wrapped around the samples for standardizing. The levels of several elements were followed: Cr, Fe, Co, Zn, and Sb.

-: By studying the production process from raw materials to finished product, we should find out how the Th gets into the acrylic.

-: We should look for inclusions optically and try to correlate them to contaminants. There was a discussion; the concensus was that this idea should be persued vigorously.

Edwards/Taylor: We should look for correlations in the scatter of other elements with Th/U.

### 3 Afternoon *—% efficiency with a low background*

Cooper: His branch has a <sup>33</sup>cryostat: the background is 2-3 counts/keV/h. They have had a Ge(Li) go bad recently so the amount of free counting time is limited.

Earle/others: There was a discussion about counting sites including the low background building, the  $\gamma$ -cave, and a second castle of Edwards. Also, detector types were discussed again. The consensus is that a well type of detector is the best all-round choice. A low background cryostat is desirable. Clifford will consider the question of optimum size of the crystal and report back in time to order the detector early next week; also the physical compatibility of the cryostat and the Compton suppressor will be considered.

Edwards: Recommends using an inner sleeve of supricil quartz to hold the monomer for neutron irradiation.

Jagam: What about the evolution of gases?

Taylor: Suggested using a large volume vessel and then putting it under vacuum to allow room for gases.

Earle: Wants a few more water samples to be run through neutron irradiation.

Edwards: There will be results soon; if the rates are low the sample will be sent to Queen's for counting.

Earle: Procedure for R. Deal: Use cycle of a) background, b) 0.6 kg acrylic (aq. reg. + HF, c)/d) 2 washes (HNO<sub>3</sub> + HF). Washes to be done hot (85-100 deg.) and to last 1 hour.

-: more P2 samples to be sent to Edwards; also monomer.

Edwards: Can R. Deal have a balance? (Taylor will oblige.)

Milton: Can be ready for another sample in about a week.

Taylor: 'stil may need replacing if there are further component failures. The present one is made of pyrex but maybe we should consider quartz if a new one is bought.

#### ACTIONS

Miller/Green: Get fume hood fixed up.

Green: Contact Los Alamos group.

Edwards: Procedure/container *etc.* for neutron activation of monomer.

Deal: Change acrylic cleaning procedure: use half hour wash in  $\text{HNO}_3$ .

Earle: Contact Fowler about cleaning agent for acrylic.

Earle: Send N. Elliot minutes of meeting with Polycast.

Clifford: Set up  $\gamma$ -ray spectrum analysis procedures.

Jagam: Investigate optical scanning as a technique for locating inclusions.

Clifford: Determine optimum size for well-type Ge counter.

Deal/Milton: 5 15 kg samples of acrylic to be run