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CRNL report Jun 7, 1990

Alpha Spectrometry Section Report by Gwen Milton

During the month of May a new suite of background analyses have been performed, following the revised chemical procedures. In order to train a summer student in these procedures, two more bars of acrylic were spiked with tracer solution prior to ignition. To date only the U analyses are complete: recoveries were 74.3 and 69.4 %. The much improved yields are probably due to repeated refluxing with HNO3 - H2O2 to facilitate the removal of ammonium nitrate prior to plating.

During the week of June 4 the separations of Th and Ra in these samples will be completed. As soon as possible thereafter another 15 kg portion of Stores acrylic will be analyzed. If the results are in good agreement with those obtained in March we shall then be in a position to analyze only those samples giving the lowest values in mass spectrometric analyses.

Neutron Activation Analysis by T. Clifford

1. Several pieces of acrylic were cut using the laser at N.R.C. to remove thin slabs from the surface of each piece. Three of these samples have now been irradiated at CRNL and counted at Queen's. On two samples, the surface pieces (some of which were saw cut at CRNL) were found to have a much higher level of thorium than the inside piece i.e. 78 & 22 ppt vs 3 & 4 ppt. Results from the third set will be available shortly.

2. The two most recent water samples have come back from the reactor with the containers empty and obviously damaged. Records of NRU reactor show that there have been changes to the fuel and absorbers in the relevent section of the reactor and that the temperature has been 20 deg. higher than previously. The cooling is to be adjusted and more samples will be submitted.

3. The effort on analysing monomer has failed to yield results,

so far. The monomer acts differently to acrylic in the chemical separation process in that it destroys the apparatus. This has happened twice but at very different times in the oxidizing process. The process is being reviewed and exceptional safety precautions are required. Contact CRNL for more details.

4. The Ge gamma-ray detector has arrived at CRNL. It is very microphonic, but under quiet conditions its resolution exceeeds the manufacturer's spec's. Further commissioning tests are under way. A shielding castle is being built. With the partial castle and with the NaI anti-Compton shield operating, the background has been measured at 8 counts/keV/hour, and this should improve.

Vaporization & Mass Spectrometry Results by D Earle

April 25, 1990

Five pieces of Rohm material from the one sheet purchased in March were checked for Th and U by TIMS. In estimating the ppt we assume that the TIMS has a 0.3 ng U systematic background which must be subtracted from the measured value and that the rinse gets all of the U but not all the Th. The measured Th background should be added to the acrylic rinse value.

Date	Sample	Th (ng)	U (ng)	ppt
ap 16	Bdg	0.08	0.22	
	0.898 kg	3.6	4.1	4.4/4.2
	Bdg	0.36	0.30	
ap 17	Bdg	0.10	0.29	
	0.905 kg	7.3	4.4	8.5/4.5
	Bdg	0.40	0.48	
ap 18	Bdg	0.09	0.21	
	0.883 kg	3.8	4.6	4.6/4.7
	Bdg	0.24	0.31	
ap 19	Bdg	0.24	0.33	
	0.905	3.5	5.0	4.4/5.2

	Bdg	0.47	0.44	
ap 20	Bdg	0.4	?	
	0.558 slow burn	2.9	3.0	5.6/4.8
	Bdg	0.2	0.4	

May 28, 1990

Four pieces of Rohm acrylic were vaporized and the rinses split into two lots for TIMS and ICPMS. Tube 10 was used for all samples.

		Fraction		Th in ng		U in ng	
Date	Sample	TI	ICP	TI	ICP	TI	ICP
May 8	Bdg	0.51	0.49	0.03	0.046	0.07	0.034
	0.823 kg R5F	0.46	0.54	0.99	1.49	1.51	2.0
	Bdg	0.51	0.49	0.05	0.028	0.08	0.043
May 9	Bdg	0.47	0.53	0.02	0.02	0.05	0.022
	0.803 kg R5G	0.49	0.51	1.71	1.68	2.17	3.17
	Bdg	0.51	0.49	0.06	0.045	0.1	0.089
May 10	Bdg	0.52	0.48	0.04	0.024	0.08	0.059
	1.03 kg R5H	0.45	0.55	3.54	4.69	4.86	7.25
	Bdg	0.48	0.52	0.31	0.285	0.20	0.18
May 11	Bdg	0.49	0.51	0.29	0.047	0.08	0.040
	0.93 kg R5I	0.48	0.52	1.84	1.91	1.42	1.91
	Bdg	0.51	0.49	0.17	0.071	0.10	0.085
	Bdg	0.45	0.55	0.45	0.36	0.22	0.168
	Acid blank	0	1.0		0.15		0.038

Sample	Th in ppt		U in ppt	
	TIMS	ICPMS	TIMS	ICPMS
R5F	2.62	3.35	3.99	4.50
R5G	4.35	4.10	5.52	7.74
R5H	7.64	8.28	10.49	12.80
R5I	4.12	3.95	3.18	3.95

Conclusions:
The comparisons between TIMS and ICPMS are satisfactory. The statistical errors on both methods are around 0.1 ng or less and are negilible in comparison to the systematic errors which, based on the values above, could be as large as either 1 ng or 25% for acrylic runs and 0.2 ng for background runs. The ICPMS background values tend to be a little less than the TIMS background runs.It would appear that we can determine the Th and U in the rinse to better than 50%. It still has to be proven that the rinse contains all of the Th and U that is in the acrylic.

May 8, 1990

Three pieces of acrylic each 50 g were spiked with a 6 M nitric acid solution containing Th and U. The acrylic was vaporized, the suprasil tubes rinsed and TIMS done on the residue. The spiking, rinsing and mass spec. were all done independently to avoid any preconceived results.

Sm.#	U content (ng)		Th content (ng)		% recovery
	spike	mass sp.	spike	mass. sp.	
1	27.3	26.2	36.7	34.6	96/94
2	48.7	44.7	65.3	59.8	92/92
3	0	1.2	0	.62	
4	76.3	74.8	102.4	100.0	98/98

Both the Th and U recovery is about 95% and suprisingly with the same efficiency for both. The background rinses after each acrylic

rinse had quantities of Th/U of the order of 1% of the acrylic rinse.