

FIRST RESULTS OF TMI-2 SUMP SAMPLES ANALYSES -  
ENTRY 10

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# FIRST RESULTS OF TMI-2 SUMP SAMPLES ANALYSES - ENTRY 10

## SUMMARY

The first results of analysis of the TMI-2 sump samples obtained during Entry 10 on May 14, 1981, are presented in Table 1. The samples are from the basement water below the covered hatch in the northeast quadrant of elevation 305 in the reactor building. The samples were obtained prior to Bechtel's large-scale decontamination effort that occurred on the same date. Samples 1, 3, 6, and 8 were analyzed. Samples 2, 4, 5, and 7 were archived. The heights above the basement floor at which samples were collected are 84-3/4 in., 47-3/4 in., 5-3/8 in., and 0 in. The samples corresponding to these heights are 1 and 2, 3 and 4, 5 and 6, 7 and 8, respectively. Samples 7 and 8 contained bottom sludge as well as liquid; the others contained no observable insolubles. Both liquid and sludge were analyzed from Sample 8. Nuclide analyses were done for the gamma emitters:  $^{137}\text{Cs}$ ,  $^{134}\text{Cs}$ ; the beta emitter:  $^{90}\text{Sr}$ ; the x-ray emitter:  $^{129}\text{I}$ ; and for fissile material. The presence of  $^{144}\text{Ce}$ ,  $^{125}\text{Sb}$ , and  $^{60}\text{Co}$  was observed also and quantitatively measured where possible. Analyses procedures used are noted in the table and described on the pages following the table.

TABLE 1. TMI-2 REACTOR BUILDING BASEMENT WATER SAMPLE ANALYSES RESULTS<sup>(1)</sup>

Nuclide	Sample					
	1	3	6	8	8	8
	( $\mu\text{Ci/ml}$ )	( $\mu\text{Ci/ml}$ )	( $\mu\text{Ci/ml}$ )	Slurry ( $\mu\text{Ci/ml}$ )	Supernate ( $\mu\text{Ci/ml}$ )	Particulate ( $\mu\text{Ci/g solids}$ )
<sup>54</sup> Mn	ND*	ND	ND	>2 E-04 <sup>a</sup>	NA**	ND
<sup>60</sup> Co	>6 E-04 <sup>a</sup>	>3 E-03 <sup>a</sup>	>2 E-03 <sup>a</sup>	>8 E-04 <sup>a</sup>	NA	1.7 $\pm$ 0.2 E+01 <sup>b</sup>
<sup>90</sup> Sr	5.0 $\pm$ 0.2 E+00 <sup>c</sup>	5.4 $\pm$ 0.2 E+00 <sup>c</sup>	5.2 $\pm$ 0.2 E+00 <sup>c</sup>	NA	NA	8 $\pm$ 2 E+02 <sup>d</sup>
<sup>90</sup> Sr	5.4 $\pm$ 0.5 E+00 <sup>e</sup>	5.2 $\pm$ 0.5 E+00 <sup>e</sup>	5.1 $\pm$ 0.5 E+00 <sup>e</sup>	NA	5.3 $\pm$ 0.5 E+00 <sup>f</sup>	7.8 $\pm$ 0.8 E+02 <sup>g</sup>
<sup>106</sup> Ru	ND	ND	ND	>4 E-04 <sup>a</sup>	NA	ND
<sup>125</sup> Sb	>3 E-02 <sup>a</sup>	>3 E-02 <sup>a</sup>	>3 E-02 <sup>a</sup>	>5 E-02 <sup>a</sup>	NA	4.5 $\pm$ 0.2 E+02 <sup>b</sup>
<sup>129</sup> I	5.5 $\pm$ 0.7 E-06 <sup>h</sup>	5.4 $\pm$ 0.7 E-06 <sup>h</sup>	3.8 $\pm$ 0.5 E-06 <sup>h</sup>	NA	2.5 $\pm$ 0.5 E-06 <sup>i</sup>	NA
<sup>134</sup> Cs	1.85 $\pm$ 0.01 E+01 <sup>j</sup>	1.84 $\pm$ 0.01 E+01 <sup>j</sup>	1.86 $\pm$ 0.01 E+01 <sup>j</sup>	1.87 $\pm$ 0.01 E+01 <sup>j</sup>	NA	1.79 $\pm$ 0.04 E+02 <sup>b</sup>
<sup>137</sup> Cs	1.43 $\pm$ 0.01 E+02 <sup>j</sup>	1.42 $\pm$ 0.01 E+02 <sup>j</sup>	1.43 $\pm$ 0.01 E+02 <sup>j</sup>	1.44 $\pm$ 0.01 E+02 <sup>j</sup>	NA	1.29 $\pm$ 0.01 E+03 <sup>b</sup>
<sup>144</sup> Ce	ND	ND	ND	>8 E-03 <sup>a</sup>	NA	7.6 $\pm$ 0.6 E+01 <sup>b</sup>
	( $\mu\text{g/ml}$ )	( $\mu\text{g/ml}$ )	( $\mu\text{g/ml}$ )	( $\mu\text{g/ml}$ )	( $\mu\text{g/ml}$ )	(mg/g solids)
<sup>235</sup> U & <sup>239</sup> Pu	<1 E-02 <sup>k</sup>	<1 E-02 <sup>k</sup>	<1 E-02 <sup>k</sup>	NA	NA	8.8 $\pm$ 0.9 E-02 <sup>l</sup>
<sup>238</sup> Pu	4 $\pm$ 1 E-08 <sup>m</sup>	NA	NA	5 $\pm$ 1 E-07 <sup>m</sup>	NA	5 $\pm$ 1 E-07 <sup>n</sup>
<sup>239</sup> Pu	2.2 $\pm$ 0.7 E-04 <sup>m</sup>	NA	NA	2.6 $\pm$ 0.5 E-03 <sup>m</sup>	NA	2.9 $\pm$ 0.6 E-03 <sup>n</sup>

(1) Concentrations as of 6-1-81.

\*ND = not detected

\*\*NA = not analyzed

Superscripts a through n refer to analyses procedures which are described on the following pages.

## SUMP SAMPLES ANALYSES PROCEDURES

- a. 1 ml aliquot of sample withdrawn. 5 mg Fe and 5 mg Cs added. 1 ml  $\text{HNO}_3$  added. Diluted to 30 ml.  $\text{NH}_4\text{OH}$  added to precipitate  $\text{Fe}(\text{OH})_3$ . Centrifuged and supernate discarded. Dissolved  $\text{Fe}(\text{OH})_3$  in 1 ml  $\text{HNO}_3$ . Diluted to 30 ml. Repeated  $\text{Fe}(\text{OH})_3$  precipitation and discarded supernate three times. Dissolved  $\text{Fe}(\text{OH})_3$  in 1 ml  $\text{HNO}_3$  and diluted to 5.0 ml. 100  $\lambda$  withdrawn and dried for counting as point source on Ge(Li) spectrometer. Concentrations are reported as > ("greater than") because of unknown losses in the discarded supernates.
- b. 5 ml of sample filtered through 0.45 $\mu$  filter. Dissolved filter in  $\text{HNO}_3$ , HCl, HF. Diluted to 5 ml. 0.5 ml aliquot dried and counted as point source with Ge(Li) spectrometer.
- c. Precipitated  $\text{Sr}_3(\text{PO}_4)_2$ . Dissolved in  $\text{HNO}_3$ . Precipitated  $\text{Sr}(\text{NO}_3)_2$  with fuming  $\text{HNO}_3$ . Dissolved in 5 ml  $\text{H}_2\text{O}$ . Repeated precip w/fuming three times. 5 mg Fe added.  $\text{NH}_4\text{OH}$  added to precipitate  $\text{Fe}(\text{OH})_3$ . Filtered solution and discarded  $\text{Fe}(\text{OH})_3$  precipitate. Added  $\text{Na}_2\text{CO}_3$  and precipitated  $\text{SrCO}_3$ . Counted on end-window gas flow proportional counter.
- d. 5 ml of sample filtered through 0.45 $\mu$  filter. Filter dried and weighed to determine mass of solids. Filter dissolved in  $\text{HNO}_3$  + HCl + HF +  $\text{HClO}_4$ . HF, HCl, and most  $\text{HClO}_4$  fumed off. 2 ml  $\text{HNO}_3$  heated to boiling. Diluted to 10 ml. 50  $\lambda$  and 100  $\lambda$  withdrawn for analyses. Analyses performed as described in paragraph c.
- e. Extracted Sr from pH 3.5 sample solution using 4, 4'(5')-di-tert-butylbenzo-24-crown-8 complexing agent. Stripped Sr from extractant with an equal volume of 0.5 M  $\text{HNO}_3$ . Added 10 mg Sr carrier. Precipitated  $\text{SrCO}_3$  with  $\text{Na}_2\text{CO}_3$ . Washed twice with 0.2 M  $\text{Na}_2\text{CO}_3$ . Dissolved in 0.5 M  $\text{HNO}_3$  and diluted to 5 ml. Added 10 ml of "Instagel" scintillation cocktail and counted on beta scintillation spectrometer.

- f. An aliquot of sample supernate was withdrawn and the analysis procedure of paragraph e was followed.
- g. 5 ml of slurry was filtered. Filter dried and weighed to determine mass of solids. Filter dissolved in 10 ml  $\text{HNO}_3 + \text{HClO}_4 + \text{HF}$ . 0.5 ml aliquot withdrawn and the analysis procedure of paragraph e was followed.
- h. Sample aliquot was analyzed via  $^{129}\text{I}(n,\gamma)^{130}\text{I}$  reaction with an  $^{129}\text{I}$  standard activated concurrently.
- i. An aliquot of sample supernate was withdrawn and passed through anion exchange column. Iodine was then washed from column.  $^{125}\text{I}$  tracer used for process yield. Effluent analyzed via  $^{129}\text{I}(n,\gamma)^{130}\text{I}$  reaction with standard activated concurrently.
- j. 100  $\lambda$  of sample evaporated to a point source and counted on Ge(Li) spectrometer.
- k. Aliquot of sample analyzed via delayed fission neutron technique. Neutron counter calibrated only with  $^{235}\text{U}$  and therefore mass concentration reported as  $^{235}\text{U}$  equivalent. Technique detection limit = 10 ng/ml  $^{235}\text{U}$ .
- l. Aliquot of a well-shaken slurry was analyzed via delayed fission neutron technique. Sample aliquot filtered through 0.45 $\mu$  filter, dried, and weighed to determine mass of solids.
- m. Pu separated from 1 ml aliquot of sample. Separated Pu counted on alpha spectrometer. Pu-239 concentration calculated using ORIGEN generated Pu-239 to Pu-240 ratio and spectral count data.
- n. Analyzed as described in paragraph m. Concentration on mass basis calculated using average measured solids mass concentration of slurry, which was 0.9 mg solid per ml slurry.