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INTERCALIBRATION
OF COUNTING LABORATORIES

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INTERCALIBRATION
OF COUNTING LABORATORIES

By

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INTERCALIBRATION
OF COUNTING LABORATORIESBy
J. A. UlsethABSTRACT

Eleven major radioisotope counting laboratories have participated in an intercalibration test to determine the degree of consistency among the laboratories in measuring activations from the $^{54}\text{Fe}(\text{n,p})^{54}\text{Mn}$, $^{46}\text{Ti}(\text{n,p})^{46}\text{Sc}$, $^{58}\text{Ni}(\text{n,p})^{58}\text{Co}$, and $^{59}\text{Co}(\text{n},\gamma)^{60}\text{Co}$ reactions. The counting results of this test compare favorably with a similar test conducted in 1963 with the exception of results for the $^{58}\text{Ni}(\text{n,p})^{58}\text{Co}$ reaction where the standard deviation (1σ) among laboratories has slipped from $\pm 2.6\%$ to $\pm 4.2\%$. The laboratories were also asked to calculate fast ($E > 1 \text{ MeV}$) and thermal fluence values from the activities. In determining fluence from these monitors, the laboratories were consistent only for thermal fluence (standard deviation of 8%). Fast fluence determined from the threshold monitors differ by as much as $\pm 23\%$ for the $^{54}\text{Fe}(\text{n,p})^{54}\text{Mn}$ reaction, to $\sim \pm 15\%$ for the $^{58}\text{Ni}(\text{n,p})^{58}\text{Co}$ and the $^{46}\text{Ti}(\text{n,p})^{46}\text{Sc}$ reactions.

INTERCALIBRATION OF COUNTING LABORATORIES

J. A. Ulseth

INTRODUCTION

The Irradiation Standards and Procedures Committee (ISPC) of the Irradiation Effects on Reactor Materials program conducted an intercalibration in 1963⁽¹⁾ to assure satisfactory consistency in counting of radioisotopes used in neutron dosimetry by the participating laboratories. The present study was undertaken in 1967 to determine whether counting consistency among the laboratories was being maintained.

In addition to the activity in disintegrations/sec/mg (d/s/mg) as in the 1963 study, the laboratories were asked to report activity in d/s/foil to permit any discrepancies in the foil weights to be detected and separated from the activation analysis. Two sets of foils differing in activity level by a factor of 100 were supplied to check calibration at different counting rates. The laboratories were also asked to provide fluence determinations from the activated monitors if feasible. For this purpose, an 18-energy group spectral calculation of the irradiation facility was provided along with the set (if available) of up-to-date differential cross section values for each of the monitors.

The participating counting laboratories were:

Argonne National Laboratory
Atomics International
Battelle Memorial Institute, Columbus, Ohio
Brookhaven National Laboratory
Gulf General Atomic (formerly General Atomic)
General Electric Company, NMPO, Cincinnati, Ohio

(1) H. H. Yoshikawa and W. H. Zimmer. Fast Neutron Dosimetry: Intercalibration of Counting Facilities, HW-81871. Available from Clearinghouse for Federal Scientific and Technical Information, Springfield, Virginia, April 1963.

General Electric Company, APED, San Jose, California
Idaho Nuclear Incorporated
Oak Ridge National Laboratory
Pacific Northwest Laboratory, Richland, Washington
Atlantic Richfield Hanford Company (formerly
Isochem, Inc.)

TEST PROCEDURES

The general test procedure was to select monitor materials from types commonly used for fluence determinations in experimental irradiation facilities. The materials were irradiated to two fluence levels differing roughly by a factor of 100 to test calibration at different counting levels and then weighed and counted by the ARHCO (Richland) Laboratory before distribution to the participating laboratories. Each laboratory was asked to weigh and determine the activity of each sample, calculate the fluence from the activity in feasible cases, and report results to the Pacific Northwest Laboratory for compilation, analysis, and reporting of the test data.

MONITOR MATERIALS

The monitor materials used for the experiment were 0.020-in. diam sample of iron, nickel, titanium, and aluminum-cobalt (0.1% Co) supplied by PNL from a stock kept especially for participating laboratories. The monitor reactions to be analyzed were $^{54}\text{Fe}(n,p)^{54}\text{Mn}$, $^{58}\text{Ni}(n,p)^{58}\text{Co}$, $^{46}\text{Ti}(n,p)^{46}\text{Sc}$, and the $^{58}\text{Co}(n,\gamma)^{59}\text{Co}$.

A monitor set included one 1/4-in. long piece of each of these materials. Each laboratory received two complete sets, one of which was irradiated for 2 hr and the other for 196 hr ($\sim$ factor of 100) in the MTR rabbit facility, VH2. The iron material was irradiated in a 0.040-in. thick cadmium cover for both irradiation levels in order to reduce the interference of the $^{58}\text{Fe}(n,\gamma)^{59}\text{Fe}$ reaction.

IRRADIATION FACILITY

The Materials Test Reactor (MTR) is a light water-moderated and cooled test reactor located at the National Reactor Testing Station in Idaho. The VH2 rabbit facility is located vertically about 3 in. outside the reactor core in the beryllium reflector.

RESULTS

INTERLABORATORY COMPARISON OF MEASURED ACTIVITIES

The results of the total activity comparisons are compiled in Table 1, where each entry is the ratio of the total activity (disintegrations/sec/foil), determined by the participating laboratory, to the total activity determined for that specific monitor by the reference laboratory (formally Isochem, now Atlantic Richfield). The average ratio for each monitor at both levels of irradiation is listed along with the standard deviation of the data in each column. The ratios in Table 1 marked by an asterisk reflect some large undetermined error and were not included in the average ratios or standard deviation calculations. The results listed in Table 1 indicate the 10 participating laboratories to be consistent within approximately 4% for the nickel, iron, and titanium threshold reactions, and to less than 3% for the thermal reaction.

A comparison between the present study and the 1963 intercalibration study is shown in Table 2. For the Ti, Co, and Fe reactions, the laboratories were consistent to the same degree, but the standard deviation increased from 2.6 to 4.2% for the nickel reaction.

MONITOR WEIGHTS

The difference between the average total activity ratio and the average specific activity ratio of only 0.5% indicates that monitor weights were consistently measured by the participating laboratories.

TABLE 1. 1967 Intercalibration of Counting Laboratories

(Total Activity Ratio - Other Labs/Isochem)

Laboratory	Low Level 2-hr Irradiation				High Level 196-hr Irradiation			
	⁶⁰ Co	⁵⁴ Mn	⁵⁸ Co	⁴⁶ Sc	⁶⁰ Co	⁵⁴ Mn	⁵⁸ Co	⁴⁶ Sc
1	0.930	0.970	0.932	0.975	0.959	0.971	0.963	1.021
2	0.999	0.969	0.993	1.018	1.049	0.979	1.006	1.057
3	1.201*	0.933	0.919	0.962	1.000	0.944	0.799*	0.999
4	0.954	1.021	0.984	1.051	0.949	1.017	1.013	1.101
5	0.965	0.981	0.983	1.017	0.987	1.017	1.000	1.031
6	0.993	0.993	0.896	1.055	1.002	0.994	0.917	1.026
7	0.972	0.951	0.990	2.35*	0.963	1.008	0.989	0.993
8	0.965	Lost	0.968	**	0.979	1.090	0.961	1.045
9	0.970	1.037	0.991	1.086	1.012	Lost	1.067	1.031
10	0.995	1.009	1.009	1.063	0.979	0.996	0.996	1.060
Average	0.971	0.984	0.966	1.028	0.988	1.002	0.990	1.036
% σ	2.3	3.3	3.8	4.2	3.0	4.1	4.2	3.0

* Ratios not included in statistical analysis.

** Activity too low for laboratory to measure.

TABLE 2. Comparison of the 1963 Intercalibration Study to the Present

Monitor Reaction	Average Ratio*			Standard Deviation of Ratios		
	Present			Present		
	1963	2 hr	8 day	1963	2 hr	8 day
⁵⁹ Co(n,γ) ⁶⁰ Co	0.996	0.971	0.988	0.032	0.022	0.029
⁵⁸ Ni(n,p) ⁵⁸ Co	0.990	0.966	0.990	0.026	0.038	0.042
⁴⁶ Ti(n,p) ⁴⁶ Sc	1.040	1.028	1.036	0.046	0.043	0.032
⁵⁴ Fe(n,p) ⁵⁴ MN	0.990	0.884	1.002	0.041	0.032	0.041

* Average ratio of monitor activity measured counting laboratories to monitor activity measured by Isochem.

FLUENCE DETERMINATIONS

Five of the participating laboratories reported the fluences determined from their sets of monitors. Each laboratory received

a calculated 18 energy group neutron spectrum of the MTR VH-2 rabbit facility and were asked to determine fluence using spectral averaged cross sections if feasible, or fission averaged cross sections in reporting fluence from the monitors. Each laboratory was asked to report the value of the average cross sections used and whether they were based on the calculated or the fission spectrum. The cross sections used by the reporting laboratories and the reference laboratory are listed in Table 3. These values were used to determine fluences for the respective sets of monitors.

TABLE 3. *Cross Section Values Used by the Counting Laboratories to Reduce Activation Data to Fluence*

Laboratory	$^{54}\text{Mn}(n,p)^{54}\text{Mn}$	$^{58}\text{Ni}(n,p)^{60}\text{Co}$	$^{46}\text{Ti}(n,p)^{46}\text{Sc}$	$^{59}\text{Co}(n,\gamma)^{60}\text{Co}$
A	93.5 ⁽¹⁾	100 ⁽¹⁾	10.3 ⁽¹⁾	3.7×10^4 ^(2,3)
B	85 ⁽¹⁾	91 ⁽¹⁾	9.1 ⁽²⁾	3.7×10^4 ^(2,3)
C	68 ⁽²⁾	95 ⁽²⁾	9.5 ⁽²⁾	3.8×10^4 ^(2,3)
D	68 ⁽²⁾	95 ⁽²⁾	9.5 ⁽²⁾	3.6×10^4 ^(2,3)
Reference Lab	63.8 ⁽¹⁾	92.4 ⁽¹⁾	10.2 ⁽¹⁾	3.74×10^4 ^(2,3)

-
- (1) *Spectral averaged* $\sigma(E > 1.0 \text{ MeV})$
 (2) *Fission averaged* $\sigma(E > 1.0 \text{ MeV})$
 (3) *Thermal averaged* $\sigma(E > 0.4 \text{ MeV})$

The fluences determined by four participating laboratories and the reference laboratory are listed for the short term irradiation in Table 4 and for the long term irradiation in Table 5. In both tables, fluences reported by the reference laboratory are an average value determined from 15 monitors of each type for both the 2 hr and 196 hr irradiations.

TABLE 4. *Fluences Reported for the 2-hr Irradiation in MTR Rabbit Facility*

Laboratory	$\phi, \text{ n/cm}^2$			
	$\phi, E > 1.0 \text{ MeV}$			$\phi \text{ Thermal}$
	$^{54}\text{Fe}(n,p)^{54}\text{Mn}$	$^{58}\text{Ni}(n,p)^{58}\text{Co}$	$^{46}\text{Ti}(n,p)^{46}\text{Sc}$	$^{59}\text{Co}(n,\gamma)^{60}\text{Co}$
Reference ⁽¹⁾	1.64×10^{17}	1.70×10^{17}	1.70×10^{17}	1.29×10^{18}
A	9.8×10^{16}	1.59×10^{17}	$3.96 \times 10^{17(2)}$	1.29×10^{18}
B	1.22×10^{17}	1.70×10^{17}	1.41×10^{17}	1.10×10^{18}
C	1.01×10^{17}	1.18×10^{17}	1.30×10^{17}	1.11×10^{18}
D	1.46×10^{17}	1.46×10^{17}	1.80×10^{17}	1.26×10^{18}
Ave ϕ	1.26×10^{17}	1.53×10^{17}	1.55×10^{17}	1.21×10^{18}
1 σ	2.86×10^{16}	2.17×10^{16}	2.36×10^{16}	9.70×10^{16}
Deviation	$\pm 23\%$	$\pm 14\%$	$\pm 15\%$	$\pm 8.0\%$

(1) 4 Values listed for the reference laboratory are the average of 15 monitors of each type.

(2) Not used in average or standard deviation determinations.

TABLE 5. *Fluences Reported for the 196-hr Irradiation in the MTR Rabbit Facility*

Laboratory	$\phi, \text{ n/cm}^2$			ϕ_{Thermal}
	$\phi(E > 1.0 \text{ MeV})$			
	$^{54}\text{Fe}(n,p)^{54}\text{Mn}$	$^{58}\text{Ni}(n,p)^{58}\text{Co}$	$^{46}\text{Ti}(n,p)^{46}\text{Sc}$	$^{59}\text{Co}(n,\gamma)^{60}\text{Co}$
Reference ⁽¹⁾	1.86×10^{19}	1.72×10^{19}	1.55×10^{19}	1.09×10^{20}
A	1.14×10^{19}	1.27×10^{19}	1.48×10^{19}	1.04×10^{20}
B	1.40×10^{19}	1.88×10^{19}	1.37×10^{19}	1.05×10^{20}
C	1.20×10^{19}	1.26×10^{19}	1.21×10^{19}	1.14×10^{20}
D	1.63×10^{19}	1.46×10^{19}	1.70×10^{19}	1.03×10^{20}
Ave ϕ	1.45×10^{19}	1.51×10^{19}	1.46×10^{19}	1.07×10^{20}
1 σ	3.01×10^{18}	2.75×10^{18}	1.85×10^{18}	4.52×10^{18}
Deviation	$\pm 21\%$	$\pm 18\%$	$\pm 13\%$	$\pm 4.2\%$

(1) ϕ Values are the average of 15 monitors of each type listed for the reference laboratory

The percent standard deviation among the five reporting laboratories varies from $\pm 23\%$ for fluence ($\phi E > 1.0 \text{ MeV}$) determined for the $^{54}\text{Fe}(n,p)^{54}\text{Mn}$ reaction, to $\pm 4.0\%$ for thermal fluence. Since the cross sections listed in Table 3 for the iron reaction vary by $\pm 19\%$, this variation in fluence values is largely originated from cross sections used in the fluence calculations. Some standardization in cross section values is required to achieve complete consistency in fluence determinations.

CONCLUSIONS AND RECOMMENDATIONS

Activation rates from the $^{54}\text{Fe}(n,p)^{54}\text{Mn}$, $^{46}\text{Ti}(n,p)^{46}\text{Sc}$, and the $^{59}\text{Co}(n,\gamma)^{60}\text{Co}$ reactions determined during the present intercalibration test are consistent among the 11 laboratories to the same degree as the 1963 test except for the $^{58}\text{Ni}(n,p)^{58}\text{Co}$ reaction where the consistency among the laboratories have slipped from a 1 σ value of $\pm 2.6\%$ (1963) to $\pm 4.2\%$ (present).

In determining fluence from the monitors, the laboratories display satisfactory consistency only for thermal fluence (standard deviation of 8%). Fast fluence [$\phi(E > 1 \text{ MeV})$] determined from the threshold monitors differ by as much as $\pm 23\%$ for the $^{54}\text{Fe}(n,p)^{54}\text{Mn}$ reaction, to $\sim \pm 15\%$ for the $^{58}\text{Ni}(n,p)^{58}\text{Co}$ and $^{46}\text{Ti}(n,p)^{46}\text{Sc}$ reactions. Standardization of present differential cross section data for the common threshold reactions such as $^{54}\text{Fe}(n,p)^{54}\text{Mn}$ and $^{58}\text{Ni}(n,p)^{58}\text{Co}$ appears necessary to achieve satisfactory agreement in fluence values.

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