NUREG/CR-3809
DOE/NBM 1071
Vol. 1

Performance Testing of Radiobioassay Laboratories: In-Vitro Measurements, Pilot Study Report

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U.S. Nuclear Regulatory Commission

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PNL-5248
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Manuscript Completed: September 1984
Date Published: December 1984

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Prepared for
Division of Radiation Programs and Earth Sciences
Office of Nuclear Regulatory Research
U.S. Nuclear Regulatory Commission
Washington, D.C. 20555
NRC FIN B2417

Prepared for
Radiological Controls Division
Office of Nuclear Safety
U.S. Department of Energy
Under Contract DE-AC06-76RLO 1830

FOREWORD

In recent years, the U.S. Nuclear Regulatory Commission (NRC) and the U.S. Department of Energy (DOE) have jointly sponsored research to improve occupational radiation protection. Of particular concern have been the accuracy and quality control of performance criteria for personnel dosimeters, radiation survey instruments, and bioassay laboratory measurements.

The Health Physics Society Standards Committee was requested by NRC and DOE to provide guidance regarding acceptable criteria for bioassay laboratory measurements. The Standards Committee appointed Working Group 2.5 to prepare, for the American National Standards Institute (ANSI), a draft ANSI Standard N13.30, Performance Criteria for Radiobioassay.

Draft ANSI Standard N13.30 provides quantitative performance criteria for bias, precision, and acceptable minimum detectable amounts in radiobioassay measurements, for a selected list of measurement categories and commonly assayed radionuclides. It also provides standard quality control procedures for the internal quality assurance programs of radiobioassay laboratories. The draft Standard includes guidelines to be used by a future laboratory for testing the conformance of bioassay service laboratories to the performance criteria and standard quality control guidelines that could be used in a program for laboratory accreditation. The completed Standard will provide guidance for future efforts to upgrade radiobioassay measurements. This document describes part of a research program to evaluate portions of the current (September 1983) draft ANSI N13.30. The program is focused on performing test measurements and evaluating actual laboratory performance against criteria proposed in the draft Standard.

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ABSTRACT

This research program at the Pacific Northwest Laboratory entitled "Technical Evaluation of Draft ANSI Standard N13.30, Performance Criteria for Radiobioassay" is jointly sponsored by the Nuclear Regulatory Commission and the Department of Energy. It is a nationwide, two-round bioassay intercomparison study to test the analytical performance of both in-vitro and in-vivo bioassay laboratories and determine their ability to meet the minimum performance criteria specified in the draft American National Standards Institute (ANSI) Standard.

Round One is the Pilot Study involving a small number of voluntarily participating laboratories. Round Two will involve a larger number of laboratories and will expand on the results of Round One. This report, In-Vitro Measurements, is a review of the methodology and results of Round One. For this part of the research program, test samples of artificial urine containing precisely known quantities of certain radionuclides were sent to 19 bioassay laboratories, and 16 of those returned analysis data.

Results show that some of the participating laboratories had difficulty meeting the performance criteria specified in the current draft ANSI Standard N13.30. Based on these results, specific recommendations were made to the working group preparing the draft Standard.

SUMMARY

The Code of Federal Regulations, Title 10, Part 20 (10 CFR 20), and the U.S. Department of Energy Order 5480.1, Chapter XI (DOE 1983), require assessment of occupational radiation exposures. To correctly assess internal exposure to radioactive materials, accurate bioassay measurements are necessary. However, a concern of licensees of the U.S. Nuclear Regulatory Commission (NRC) and of DOE facilities and contractors is that bioassay laboratories may not be providing accurate and consistent results.

To meet this concern, a Health Physics Society Working Group 2.5, formed at the request of NRC and DOE, prepared a draft of an American National Standards Institute (ANSI) standard of performance for radiobioassay laboratories. The draft Standard provides performance criteria in the form of minimum numerical values necessary to meet an acceptable minimum detectable amount (AMDA), provides limits for measurement bias (B_r), and specifies the precision (S_B) required for meeting the performance standards. After the draft Standard was prepared, NRC issued an advance notice of intent to require licensees to obtain services from "accredited" in-house or commercial laboratories (Federal Register 1981). In addition, DOE is reviewing the feasibility of an accreditation program for bioassay laboratories serving its facilities and contractors.

In order to evaluate the appropriateness of the draft Standard, NRC and DOE jointly sponsored a research program at the Pacific Northwest Laboratory (PNL) entitled "Technical Evaluation of Draft ANSI Standard N13.30, Performance Criteria for Radiobioassay." It is a nationwide, two-round bioassay intercomparison study to test the analytical performance of both in-vitro and in-vivo bioassay laboratories and determine their capability to meet the minimum performance criteria specified in the draft Standard. Round One is the Pilot Study involving a small number of voluntarily participating laboratories. Round Two will involve a larger number of laboratories and will be based on the results of Round One. This report is a review of the results of Round One, in-vitro measurements.

Round One of the in-vitro testing involved the preparation and distribution of about 560 samples of artificial urine containing carefully controlled quantities of either ^{3}H, ^{90}Sr, ^{241}Am+^{238}Pu, ^{137}Cs, or natural uranium. Nineteen laboratories volunteered to participate in this round and tests were conducted in the following categories: liquid scintillation counting, gross-beta measurements, alpha spectrometry, gamma spectrometry, and mass determination (for uranium).

Test samples were sent to the 19 laboratories. Sixteen laboratories returned analysis data and of those, eight laboratories omitted data on some of the test samples. In all, data on 345 samples were received and data were not returned for 126 samples. The measurement data were studied according to statistical methods presented in the draft Standard.

Results of the In-Vitro Pilot Study show that some of the participating laboratories had difficulty meeting the performance criteria specified in the current draft ANSI Standard N13.30. Failures to meet the criteria in alpha spectrometry were due to a combination of difficulties in all criteria areas, whereas uranium analysis failures were largely attributable to unacceptable bias (B_r). The major cause of failure in the gamma spectrometry test was failure to report a minimum detectable amount (MDA) less than the AMDA specified in the Standard. Very few failures, however, were observed in the liquid scintillation and beta-counting categories.

Recommendations sent to the working group preparing draft ANSI Standard N13.30 included comments regarding the:

- evaluation of AMDA levels and ranges
- standardization of count time
- use of artificial urine as a sample medium
- development of a test program for fecal sample analysis.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the joint sponsors of this research program: the U.S. Nuclear Regulatory Commission (NRC), Occupational and Radiation Protection Branch, Robert E. Alexander, Branch Chief; and the U.S. Department of Energy (DOE), Health Physics Programs, Edward J. Vallario, Group Leader. We also acknowledge Dr. Allen Brodsky of NRC and Joe Fitzgerald, Jr., of DOE for their technical guidance during the experimental and report phases of the Pilot Study. Dr. J. M. R. (Robin) Hutchinson and Dr. K. G. W. (Ken) Inn of the National Bureau of Standards kindly supplied the standard solutions of radionuclides for this study (under separate contract with NRC) and, in addition, provided many valuable suggestions and comments regarding technical aspects of sample preparation.

Finally, we wish to extend our appreciation to our editor, Ann Marshall, for her assistance in preparing and editing the manuscript; to Mary Eierdam and Ann Rupert in word processing, who wrestled with the many symbols and tables; and to Janet Rhodes, who typed the many hundreds of labels used on sample bottles and shipping cartons.

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INTRODUCTION

In recent years, both the U.S. Department of Energy (DOE) and the U.S. Nuclear Regulatory Commission (NRC) have sponsored research to improve occupational radiation protection. Particular emphasis has been devoted to improving methods for detecting and characterizing radiation sources to which workers may be exposed. Of particular concern have been the accuracy of and performance criteria for personnel dosimeters, radiation survey instruments, and bioassay laboratory measurements. The performance testing of personnel dosimetry services in support of ANSI Standard N13.11 has been the subject of several research projects (Yoder et al. 1979; Plato and Hudson 1980; Plato and Miklos 1983; Roberson and Holbrook 1984). Technical evaluation of the capability of radiation protection survey instrumentation to meet the performance specifications of draft ANSI Standard N42.17 (Selby et al. 1983; Swinth et al. 1983; Kenoyer et al. 1983) was jointly sponsored by DOE and NRC. Bioassay laboratory measurements are addressed here.

BACKGROUND FOR THIS RESEARCH PROGRAM

Radiobioassay is used to estimate the amount of a radionuclide inside the body. In-vitro analysis, one type of bioassay procedure, involves measuring radioactivity in samples of body excreta and subsequently extrapolating to body/organ burden. In-vivo analysis, another type of bioassay procedure, measures the radioactive emissions from the body (usually gamma rays) using external detectors and extrapolates to body content and/or distribution. In order to assess a worker's exposure to radioactivity, accurate bioassay measurements are necessary.

While there are many important differences in the radioanalytic techniques used for bioassay and the varied physical/chemical forms of radionuclides measured, there are also many common aspects of bioassay program management including: quality control, recordkeeping, limits of detection, precision and accuracy. In order to establish standards of bioassay performance upon which a uniform national program of performance testing could be based, NRC asked the Health Physics Society to establish Working Group 2.5 to develop performance criteria for radiobioassay.

In 1979 at the request of the NRC (Robert Alexander), the Health Physics Society Standards Committee, chaired by Edward J. Vallario of DOE, formed Working Group 2.5 under the chairmanship of Kenneth R. Heid to prepare the ANSI Standard N13.30, Performance Criteria for Radiobioassay. The first draft of this standard was completed in 1981.

The primary concern addressed by draft ANSI Standard N13.30 is that bioassay service laboratories, both commercial and private (or institutional), may not be providing accurate results for analyses performed. A number of factors may contribute to analytical inaccuracies:

- Current analytical procedures may not be adequate.
- Laboratories usually have their own approach to analytical procedures, and common methods or performance criteria are lacking.
- There is little motivation to upgrade and improve analytical capabilities.
- Adequate instrumentation is expensive so economics may be an important factor.
- Quality assurance may be deficient (i.e., no written procedures).

Although a formal system for certifying the bias, precision, and quality control of bioassay procedures has not yet been established, NRC recently issued advance notice of proposed rule making that would require NRC licensees to use accredited laboratories after NRC establishment of an accreditation program (Federal Register 1981). In addition, DOE is establishing an accreditation program in dosimetry (DOELAP) for its contractors. Most bioassay laboratories welcomed the concept of accreditation.(a) An accreditation program would be based on recommendations contained in the final version of ANSI Standard N13.30.

Draft ANSI Standard N13.30 provides quantitative performance criteria for bias and precision in radiobioassay measurements for a selected list of measurement categories and commonly assayed radionuclides. It also provides

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(a) This is the result of an informal survey by Dr. Allen Brodsky of participants at the 28th Annual Conference on Bioassay, Analytical and Environmental Chemistry, October 13-14, 1982, Natick, Massachusetts. Discussions by the authors with bioassay laboratory participants in this intercomparison further support this statement.

standard quality control procedures for the internal quality assurance programs of radiobioassay laboratories. Draft versions have included guidelines to be used by a future laboratory for testing whether bioassay service laboratories conform to both the quantitative performance criteria for bias and precision and to standard quality control procedures, such as might be required in a test for laboratory accreditation.

PROGRAM PURPOSE

This research program, entitled "Technical Evaluation of Draft ANSI Standard N13.30 Performance Criteria for Radiobioassay," is jointly sponsored by DOE and NRC to evaluate the appropriateness of the draft ANSI Standard by conducting a bioassay performance intercomparison study. The objectives of this program, which was begun at about the time the first draft Standard was completed, are to:

- establish testing procedures for evaluating bioassay laboratories in accordance with the draft Standard
- set up the necessary laboratory equipment and facilities to conduct preliminary testing of bioassay laboratory performance
- conduct two rounds of intercomparison testing
- compile results and compare the performance of bioassay laboratories to the draft Standard performance criteria
- analyze the data to determine sources of error
- recommend any necessary revisions to the draft Standard
- prepare a procedures manual for a future laboratory to follow in conducting an ongoing performance-testing program for bioassay laboratory accreditation.

This research program was scheduled to be conducted during a three-year period which began October 1, 1981. It involves three major phases: 1) developing testing procedures and establishing laboratory facilities for preparation of test samples and in-vivo phantoms; 2) conducting a pilot intercomparison study with a small number of voluntarily participating in-vitro and in-vivo laboratories; and 3) (yet to be completed) conducting a second-round intercomparison study with a larger number of participating laboratories. A

procedures manual and a research program final report are planned as part of the third phase.

Although this research program includes testing involving both in-vitro and in-vivo measurement facilities, only the in-vitro results are presented here. (In-Vivo Measurements will present results of the lung, thyroid, and whole-body counting intercomparison testing.) The remainder of this report will cover a description of Round One of the in-vitro testing, a discussion of the result of that round, and recommendations based on the results for future revisions of draft ANSI Standard N13.30.

METHODS AND PROCEDURES

Round One of the in-vitro testing was conducted by selecting a group of bioassay laboratories from volunteers. PNL then sent to the participating laboratories samples of artificial urine containing radionuclides along with instructions for handling samples and reporting analysis data. In addition, PNL conducted a survey of the laboratories' estimates of minimum detectable amount (MDA) and propagated error.

LABORATORY PARTICIPATION

Invitations to participate in both rounds of testing were mailed to 40 bioassay laboratories. A response form was provided with each invitation along with the following information: participation would be entirely voluntary; all costs pertaining to the measurement of samples would be borne by the participating laboratory; and confidentiality of the laboratory names, their categories of participation, and in the identification of their results would be strictly maintained to allow uninhibited participation.

Of the 40 laboratories invited to participate, 26 (65%) returned response forms. Four laboratories indicated no interest in participating. Twenty-one laboratories indicated a desire to be included in both rounds of testing. The participating laboratories may not constitute a representative sample of all bioassay service laboratories. It is the authors' judgment that the laboratories most concerned with quality assurance and analytical performance were more likely to volunteer.

Five measurement categories were offered for the first round of testing:

- liquid scintillation counting for ^{3}H
- alpha spectrometry for mixed ^{241}Am + ^{238}Pu
- beta measurements for ^{90}Sr
- mass determination for U-nat
- gamma spectrometry for ^{137}Cs.

The radionuclides for these categories were selected from the list of radionuclides in the draft Standard. The selection of test radionuclides was

based on considerations regarding their relative importance for internal dosimetry, frequency of need for bioassay services, and the judgment of project staff members. Ideally, a laboratory would have the opportunity to be tested with each radionuclide of a particular category. In general, responding bioassay laboratories were interested in participating in all the categories in which they normally process samples. The projected expense incurred by each laboratory for a complete study, however, justified the limitations that were adopted.

The first-round intercomparison study was limited to nine participants per category. Participants were matched to categories of interest, and telephone calls were then made to confirm their participation and to indicate the schedule for shipment of samples. Due to the large number of willing participants, the participation of each bioassay laboratory was limited to a maximum of four test categories.

SURVEY OF MINIMUM DETECTABLE AMOUNT (MDA) AND ESTIMATED ANALYTICAL ERROR

The minimum detectable amount (MDA) is an indicator of the detection capability of a laboratory's analytical method. A survey of participating in-vitro laboratories was taken before test samples were prepared (a sample letter is shown in Appendix A). The laboratories were asked to provide an estimate of their MDA, using the formula recommended by the draft Standard and using estimated parameters (or average historical parameters if available) for their estimation of MDA.

The survey of participating laboratories also included a request for an estimate of propagated errors at various analytical levels. The analytical levels chosen were multiples of the acceptable minimum detectable amount (AMDA) levels set forth in the draft Standard for each nuclide.

These estimated MDAs and estimated propagated errors were used to help select appropriate test levels for Round-One testing and then were compared to the actual reported test measurements.

PREPARATION OF IN-VITRO TEST SAMPLES

Under an interagency agreement between the National Bureau of Standards (NBS) of the U.S. Department of Commerce and NRC, the Bureau prepared and provided calibrated, standardized radioactive stock solutions for this project. The radionuclides (listed in Table 1) were obtained from NBS in heat-sealed glass ampules.

Table 2 shows the measurement-testing categories, radionuclides, and testing levels chosen for the Pilot Study. The testing levels shown in Table 2 correspond to the testing ranges recommended in the June 1983 draft Standard. Certification and documentation accompanied each radionuclide preparation supplied by NBS. In addition, NBS radiochemists visited our laboratory, reviewed and audited proposed procedures for diluting the radionuclides into artificial-urine test samples, and provided recommendations for improved accuracy in the preparation of test samples. Their recommendations are incorporated in the procedures of Appendix B. This direct and frequent collaboration between PNL and NBS resulted in an increased level of confidence in the accuracy of radionuclide levels in samples prepared for the intercomparison testing.

TABLE 1. Chemical Form of Radionuclides Supplied by NBS

Nuclide	Chemical Form
^{3}H	Tritated water
^{238}Pu	Plutonium nitrate in 5 M nitric acid
^{241}Am	Americium nitrate in 1 M nitric acid
^{90}Sr	Strontium chloride in 1 M hydrochloric acid
U-nat	Uranium nitrate in 1 M nitric acid
^{137}Cs	Cesium chloride in 1 M hydrochloric acid

TABLE 2. In-Vitro-Testing Categories, Radionuclides, and Testing Levels

Testing Catetgory	Nuclide	Testing Levels
Liquid scintillation	^{3}H	0, 0.03, 0.30, 3.54 µCi/L
Alpha spectrometry	^{238}Pu	0, 0.11, 0.93 pCi/L
	^{241}Am	0, 0.09, 0.91 pCi/L
Beta measurements	^{90}Sr	0, 16.9, 185 pCi/L
Mass determination	U-nat	0, 7.2, 78 µg/L
Gamma spectrometry	^{137}Cs	0, 1.5, 13.8 nCi/L

Test samples consisted of an artificial-urine matrix spiked with precisely controlled amounts of radionuclide. Artificial urine was selected over natural human urine for several reasons. Artificial urine is easily manufactured in the laboratory from commercially available chemicals. It can be mixed in any amount desired. Artificial urine exhibits chemical stability, has a long shelf life, and requires no preservation. In contrast, natural human urine is chemically and biologically active and therefore changes rapidly with time unless it is stabilized. Because artificial urine can be readily prepared as needed, its cost is considerably less than the cost of obtaining and storing natural urine. In addition, natural urine may be highly variable in composition from one donor to another, whereas artificial urine exhibits greater uniformity of composition. Finally, the background radioactivity of artificial urine is more easily controlled than that of natural urine. For testing purposes, artificial urine was the matrix of choice.

The recipe for artificial urine (Table 3) is a composite from several sources (Free and Free 1978; Attman and Dittmer 1968; Long 1961; Doresmus et al. 1978; Kelsay et al. 1979; Burns and Finlayson 1980; Lentner 1981) and includes major urine components in physiological quantities.

The artificial urine was prepared in 50-L batches according to the recipe given in Table 3. Each concentration of radionuclide was prepared as follows: artificial urine was placed in a 50-L polyethylene carboy containing a 6- by

TABLE 3. Artificial-Urine Recipe

Component	g/kg	Component	g/kg
1. Urea	16.0	10. $NaH_2PO_4 \cdot H_2O$	2.73
2. NaCl	2.32	11. $CaCl_2 \cdot 2H_2O$	0.63
3. KCl	3.43	12. Oxalic acid	0.02
4. Creatinine	1.10	13. Lactic acid	0.094
5. $NaSO_4$ (anhyd.)	4.31	14. Glucose	0.48
6. Hippuric acid	0.63	15. $Na_2SiO_3 \cdot 9H_2O$[(a)]	0.071
7. NH_4Cl	1.06	16. Pepsin	0.029
8. Citric acid	0.54	17. Conc. nitric acid (70%)[(b)]	50.0
9. $MgSO_4$ (anhyd.)	0.46	18. Yellow Food Coloring	0.06

(a) 7 mg Si/kg urine

(b) Added to insure spiked radionuclides remained in ionic form.

1-in. magnetic stirring bar. The urine was placed on a magnetic stirrer, stirred thoroughly, and then the correct volume of spike was added. The spiked urine was stirred 30 minutes, and appropriate volumes (usually 1.4 L) were dispensed into preweighed and prelabeled (usually 2-L) plastic bottles. The bottles were then reweighed. Bottles were randomly divided into lots of three each by drawing numbered chits from a container.

For each test category except liquid scintillation, each participating laboratory received nine 1.4-L samples. Three of these were control urine samples that had not been spiked with radioactive materials; three samples contained radionuclide at the lower testing level shown in Table 2; and three samples contained radionuclide at the higher testing level.

Tritium (^{3}H) samples for liquid scintillation counting were supplied in 100- to 150-mL volumes. Nine samples were prepared as above, and three additional samples were sent with tritium levels at the middle spiked testing level indicated in Table 2. The extra three samples were prepared in anticipation that the testing range for tritium might be lowered by the working group.

Complete step-by-step sample preparation procedures are provided in Appendix B of this report.

SAMPLE IDENTIFICATION AND SHIPMENT TO PARTICIPATING LABORATORIES

The filled bottles were labeled as shown in Figure 1. An identification code of the form A-Sr-0021-7631-32 was used where

A = laboratory identification code
Sr = strontium, H = tritium, etc.
0021 = sample number from 0001 to 9999
7631 = PNL laboratory book identification number
32 = PNL laboratory book page number.

Each bottle was labeled with PNL's address, date of the spike, the telephone number of a cognizant PNL staff member, and the word "RADIOACTIVE" (see Figure 1). The cap of each sample bottle was sealed with vinyl tape and the bottle was packed in a nest of absorbent material (three bottles to a box).

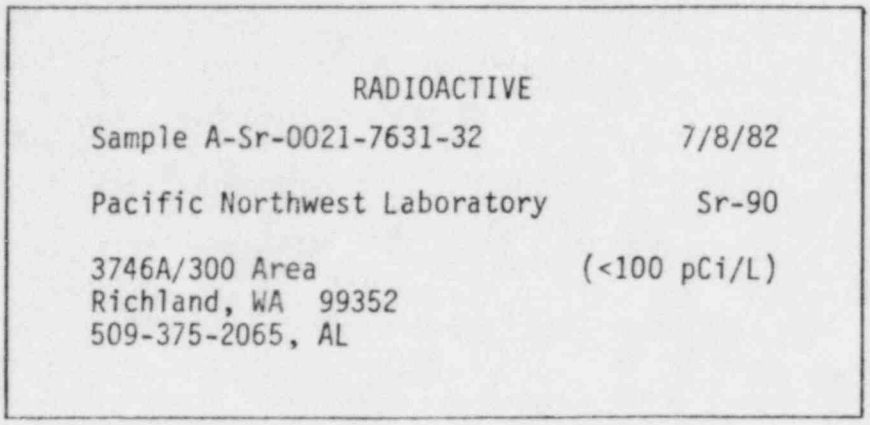

FIGURE 1. Sample Label

Shipping regulations did not require any external radiation labeling of the box. The activity levels qualified under a "limited quantity" designation, and the packing and labeling complied with federal regulations for packaging and shipping nonradioactive materials. All samples were shipped by surface carrier.

A letter was sent to each participant under separate cover advising of the incoming samples. In addition, several enclosures accompanied the package when it was sent to the participating laboratories, including:

1. general instructions and explanations
2. quality assurance guidelines
3. In-Vitro Measurements Report Form.

The general instructions contained procedures for logging in samples and confirming receipt, a request for analytical procedures used, and a data reporting deadline. A sample of the instructions to participating laboratories is included in Appendix C of this report. A copy of the In-Vitro Measurements Report Form is included in Appendix D.

QUALITY ASSURANCE (QA) PLAN

This research project conforms with PNL-MA-65 (1978) and with the draft ANSI Standard N13.30, Performance Criteria for Radiobioassay. As the testing laboratory, PNL is bound by the same QA requirements as the participating laboratories.

At PNL, all equipment and laboratory procedures or evaluations were documented in laboratory notebooks and record books. Standard reference materials were obtained from NBS and were used for all spikes.

QA for Participating Laboratories

Participating laboratories are guided by QA instructions presented in Section 6 of the draft Standard. Pacific Northwest Laboratory specifically addressed Section 6.1.1 (Parts B, C, D and E) in our Instructions to Laboratories (Appendix C) and in the In-Vitro Measurements Report Form (Appendix D).

Third-Party Crosscheck of Samples

The sample preparation involved mixing large batches of artificial urine with small volumes of radionuclide. It was possible, therefore, for problems of absorption, incomplete mixing, precipitation, and cross-contamination to occur, which could have resulted in test samples not receiving the specified radionuclide amounts. Although the sample preparation procedures were designed to prevent these occurrences, some form of crosscheck on the final solution was desirable. Therefore, an aliquot was taken from each batch and submitted to a third-party analytical laboratory for crosscheck analysis.(a) The third-party laboratory was required to be implicitly traceable to NBS. Special handling and nonroutine analysis were also requested of the third-party laboratory to insure accurate measurements.

(a) Performed by EAL Corporation, Richmond, California.

RESULTS

The results of the survey of the estimated propagated error and the estimated MDAs, the test sample analyses, and the third-party crosscheck analyses are presented here.

SURVEY OF MINIMUM DETECTABLE AMOUNT AND ESTIMATED ERROR

A comparison of estimated MDAs from the initial survey, PNL-calculated MDAs from the test results, and the AMDAs defined in the draft Standard are shown in Table 4. The method used by PNL to calculate MDAs from the measurement data is given in Appendix E. This comparison shows that in most cases the PNL-calculated MDA was similar to the laboratory-estimated MDAs provided prior to the beginning of the intercomparison test. We concluded that the draft Standard contains technically achievable AMDAs for the radionuclides tested in the Pilot Study. However, this observation was based on a small number of results. Much of the data requested from participating laboratories

TABLE 4. Comparison of MDAs Estimated by Participating Laboratories (LABs)[(a)] and MDAs Calculated by PNL[(b)] for Test Radionuclides

^{3}H (nCi/L)		^{238}Pu (pCi/L)		^{241}Am (pCi/L)		^{90}Sr (pCi/L)		U-nat (µg/L)		^{137}Cs (pCi/L)	
PNL	LABs	PNL	LABs	PNL	LABs	PNL	LABs	PNL	LABs	PNL	LABs
1.1	1.5	0.21	0.44	0.15	0.44	5.1	1.4	14.2	5.0	13	14
6.8	7.8	0.023	0.084	0.09	0.08	2.3	5.0	0.6	5.0	5.6	60
3.1	1.0	0.023	0.022	0.06	0.03	1.5	2.3	---	4.7	---	170
0.8	7.0	0.003	0.06	0.27	0.06	13.4	9.8	4.7	---	---	100
1.1	---	0.13	1.05	0.18	1.5	---	2.5	---	0.80	---	36
2.2	---	---	0.015	---	0.08	---	0.5	---	1.3	---	35
1.0	---	---	0.07	2.7	---	---	1.9			---	0.048
---	6.0					---	2.4				
---	200										
---	1.0										
---	0.8										
Defined AMDAs											
100		0.06		0.06		10.0		5.0		40	

(a) MDA estimated by the participating laboratory as defined by draft ANSI Standard N13.30.
(b) MDA estimated as 4.65 S_b, where S_b is the standard deviation of the blank urine samples.

was not provided according to the instructions given, and MDAs could not be calculated for each laboratory. It is possible that laboratories having difficulty meeting the AMDAs did not respond.

The survey of participating laboratories also included a request for an estimate of propagated errors at various analytical levels. The analytical levels chosen were multiples of AMDA levels set forth in the draft Standard for each nuclide. These data are summarized in Table 5. The acceptable precision error permitted by the draft Standard is up to 40% ($S_B \leq 0.40$). Comparison of the estimated capability of the various laboratories to the limit set by the draft Standard reveals that none of the laboratories would anticipate likely failure at sample concentrations of 20 to 100 times the AMDA

TABLE 5. Laboratory Estimates of Propagated Error at Multiple AMDA Levels for Nuclides in Round-One Testing

Activity Level[a]	Percent Error (One Standard Deviation)					
	^{3}H[b]	^{238}Pu[c]	^{241}Am	^{90}Sr	U-nat	^{137}Cs
AMDA	5	40	27	30	40	60
20 AMDA	5	10	10	10	8	1
100 AMDA	5	10	8	5	4	1
AMDA	7	---		16		10
20 AMDA	7	16		14		4
100 AMDA	7	13		14		3
AMDA	5			10		10
20 AMDA	5			8		5
100 AMDA	1.5			6		4
AMDA	5	280	233	20	40	50
20 AMDA	3	20	17	5	5	4
100 AMDA	1	8	8	2	4	3
AMDA		24	23			
20 AMDA		6	6			
100 AMDA		4	5			
AMDA	5	20	20	16		20
20 AMDA	3.5	10	10	5		10
100 AMDA	3.5	5	10	5		1
AMDA	22	22	22	22		25
20 AMDA	22	22	22	22		21
100 AMDA	21	22	22	22		25

(a) AMDAs for each nuclide are given in Appendix F.
(b) ^{3}H AMDA was 2.0 µCi/L when this survey was performed.
(c) ^{238}Pu AMDA was 0.05 pCi/L when this survey was performed.

level. Even at the AMDA level, only a few failures are anticipated by the data provided. Thus, based on laboratory estimates of error, we concluded at that time that the S_B criteria set by the standard were readily achievable.

IN-VITRO TEST SAMPLE RESULTS

A summary of the percentage of reported measurement results is given in Table 6. A total of 345 results from 16 in-vitro laboratories were received. In addition, eight laboratories failed to report 126 results. Nonresponding laboratories were contacted at least twice regarding the need for measurement results.

TABLE 6. Percentage of Participating Laboratories That Reported Test Results

Nuclide	Testing Category	Percentage of Results Received
^{3}H	Liquid scintillation	89
^{238}Pu	Alpha spectrometry	56
^{241}Am	Alpha spectrometry	56
^{90}Sr	Beta counting	56
U-nat	Mass determination	78
^{137}Cs	Gamma spectrometry	89

The results of measurements by participating in-vitro bioassay laboratories included raw data such as background count rates and counting times. The reported test data were tabulated and treated by the statistical methods described in the draft Standard.

The measurement results for each test category and laboratory are presented in Appendix F. Also included in Appendix F are the true activity concentration (nuclide added to the artificial urine), the calculated bias and precision estimators, the laboratory MDA, and an indication of whether the laboratory was able to meet the performance criteria of the draft Standard. These criteria were:

1) MDA $\leq$ AMDA
2) $-0.25 \leq B_r \leq 0.50$
3) $S_B \leq 0.40$

In an ongoing testing program, failure to meet any one of the above criteria for a radionuclide in a test category would result in failure for the entire test category. For this Pilot Study, each radionuclide and each concentration group were evaluated separately against the criteria. For example, ^{241}Am and ^{238}Pu were both in the test category "alpha spectrometry." A laboratory that failed to analyze the high-level ^{238}Pu spiked sample did not automatically fail the low-level ^{238}Pu spiked sample or the ^{241}Am samples.

Table 7 provides a summary of those laboratories that received samples in each test category and shows laboratory performance as measured against the performance criteria of the draft Standard. Table 8 shows the percentage of in-vitro measurements that did not meet the performance criteria. Again, each radionuclide and each concentration level were treated separately. For example,

TABLE 7. Summary of Round-One In-Vitro Testing Results[a,b]

Lab Code	^{3}H			^{241}Am		^{238}Pu		^{90}Sr		U-nat		^{137}Cs	
	L	M	H	L	H	L	H	L	H	L	H	L	H
A				NR	NR	NR	NR	NR	NR				
B				NR	NR	NR	NR	P	F				
C								NR	NR	F	P	NR	NR
D								NR	NR	NR	NR		
E				F	F	F	F	P	P	F	P		
F								P	P				
G	P	P	P					P	P			P	P
H								NR	NR	F	F		
I	NR	NR	NR	NR	NR	NR	NR	P	P			P	P
J	P	P	P									F	F
K	F	F	F							F	F	F	F
L	P	P	P	NR	NR	NR	NR					P	P
M	P	P	P	P	P	F	P					F	F
N	P	P	P									F	F
O				P	P	P	P			P	P	P	P
P										NR	NR		
R										P	P		
S	P	P	P	F	P	P	P			P	P		
V	P	P	P	F	F	F	F						

(a) L = low-level spike H = high-level spike F = fail
M = medium-level spike P = pass NR = no data returned
(b) Pass/fail decision was based on the comparison of the average analytical result for each concentration and radionuclide and the laboratory supplied MDA estimate to the draft Standard criteria.

TABLE 8. Percentage of In-Vitro Measurements That Did Not Meet Draft ANSI Standard N13.30 Criteria

Nuclide	Testing Category	Analysis Failures[a] (%)	Total Failures[b] (%)
^{3}H	Liquid scintillation	13	22
^{238}Pu	Alpha spectrometry	50	72
^{241}Am	Alpha spectrometry	50	72
^{90}Sr	Beta counting	10	50
U-nat	Mass determination	43	56
^{137}Cs	Gamma spectrometry	38	44

(a) Data indicates MDA > AMDA; or $B_r < -0.25$ or $B_r > +0.50$; or $S_B > 0.40$. Percentage of failures based on data received from laboratories.
(b) Includes laboratories that did not report data. Percentage of failures based on total number of samples sent out.

if five laboratories were sent two concentrations (three aliquots/concentration) of U-nat, this would result in 10 data points, each consisting of the laboratory average of the three replicates. These 10 data points would be compared to the three draft standard criteria and scored. If any two of the data points were not within the criteria, this would result in a 20% failure rate for that nuclide. These data indicate that the failure rate varied markedly from nuclide to nuclide and was quite high for several of the nuclides. Particular difficulty was noted in meeting draft Standard criteria for U-nat, ^{238}Pu, ^{241}Am, and ^{137}Cs. Table 9 shows a summary of the measurement failures by performance

TABLE 9. Summary of Failures by Performance Criteria

Performance Criterion	Failure[a] (%)
MDA	27
B_r	12
S_B	4
MDA or B_r or S_B	33
MDA or B_r or S_B or failure to return data	55

(a) Percentage of failures based on data received from laboratories.

criteria. The highest percentage of analytical failures were due to not meeting the AMDA requirements.

Table 10 shows a summary of the grand average of test sample results, relative bias (B_r), and precision (S_B). The relative bias (B_r) is a measure

TABLE 10. Grand Average of Test Sample Results, B_r, and S_B

Nuclide	Grand Averages: Test Sample Results ± 1σ[a]	B_r	S_B	Sample Number (n)	Units
^{3}H	0.03 ± 0.01	-0.04	0.04	21	µCi/L
	0.29 ± 0.04	-0.04	0.01	22	µCi/L
	3.5 ± 0.5	-0.03	0.02	23	µCi/L
^{238}Pu	0.15 ± 0.08	0.27	0.34	15	pCi/L
	0.86 ± 0.07	-0.08	0.08	15	pCi/L
^{241}Am	0.11 ± 0.04	0.28	0.40	15	pCi/L
	0.85 ± 0.11	-0.05	0.10	15	pCi/L
^{90}Sr	15 ± 2	-0.10	0.19	15	pCi/L
	155 ± 28	-0.16	0.13	15	pCi/L
U-nat	11 ± 4	0.45	0.13	21	µg/L
	82 ± 11	0.01	0.07	21	µg/L
^{137}Cs	1.41 ± 0.11	-0.05	0.03	21	nCi/L
	13.0 ± 0.9	-0.03	0.01	21	nCi/L

(a) σ = one standard deviation of the test results.

of deviation from the "true" value or activity of a sample, and precision (S_B) is a measure of the reproducibility of an analysis. At the higher level of testing (usually around 15 times AMDA), laboratory averages in all test categories met the bias and precision requirements of the draft Standard. However, there were numerous failures to meet performance criteria at the lower testing level--particularly for alpha spectrometry (^{238}Pu and ^{241}Am) and natural uranium determination. Failure to adequately measure ^{238}Pu and ^{241}Am were due to a combination of unacceptable MDAs, S_B, and B_r. Failure to measure U-nat most often involved unacceptably high bias (B_r), and most failures in the ^{137}Cs analysis were attributable to unacceptable MDAs.[a] There were very few failures in the liquid scintillation and beta measurement categories. It should be noted that the AMDA for ^{137}Cs was changed from 1.0 nCi/L to 40 pCi/L during Round-One testing, and many laboratories were unable to meet the lower MDA.

THIRD-PARTY CROSSCHECK ANALYSIS[b]

The results of the third-party laboratory analyses are shown in Table 11, and are compared to the desired radionuclide concentrations. In general, there

(a) A reassessment of MDA calculations and AMDA requirements by Working Group 2.5 is currently in progress.
(b) Third party analysis performed by EAL Corporation, Richmond, California.

TABLE 11. Third-Party Crosscheck of Spiked Artificial-Urine Samples

Nuclide	Third-Party Assay Results ± 1σ[a]	PNL-Calculated Results ± 1σ[b]	Units
^{3}H	0.0000 ± 0.0002[c]	0.00	pCi/L
^{3}H	0.029 ± 0.001	0.0295 ± 0.0002	pCi/L
^{3}H	0.29 ± 0.01	0.301 ± 0.002	pCi/L
^{3}H	3.7 ± 0.2	3.54 ± 0.02	pCi/L
^{238}Pu	0.01 ± 0.01	0.00	pCi/L
^{238}Pu	0.10 ± 0.01	0.107 ± 0.001	pCi/L
^{238}Pu	0.89 ± 0.03	0.93 ± 0.01	pCi/L
^{241}Am	0.00 ± 0.01	0.00	pCi/L
^{241}Am	0.096 ± 0.013	0.089 ± 0.002	pCi/L
^{241}Am	1.00 ± 0.04	0.91 ± 0.02	pCi/L
^{90}Sr	0.05 ± 0.13	0.00	pCi/L
^{90}Sr	14.1 ± 0.9[d]	16.9 ± 0.4	pCi/L
^{90}Sr	179 ± 11	184.5 ± 4.6	pCi/L
U-nat	0.037 ± 0.014	0.00	µg/L
U-nat	7.4 ± 0.4	7.24 ± 0.02	µg/L
U-nat	7.24 ± 0.02[e]	---	µg/L
U-nat	79 ± 3	77.8 ± 0.2	µg/L
^{137}Cs	<0.01	0.00	nCi/L
^{137}Cs	1.51 ± 0.04	1.51 ± 0.02	nCi/L
^{137}Cs	13.7 ± 0.1	13.8 ± 0.2	nCi/L

(a) Results based on analysis of one sample. Includes estimate of propagated and statistical errors.
(b) Two-step gravimetric dilution of NBS certified standards. Error propagated by methods described in Appendix G.
(c) Tritium results given to ±2σ.
(d) Significantly different from PNL-calculated results at the 95% confidence level but not at the 99% confidence level.
(e) Measured at PNL by laser phosphorimetry.

was excellent agreement, indicating that the dilution scheme was followed as planned. In addition, selected urine samples containing uranium were measured onsite using a laser phosphorimetry technique (Bushaw 1982). Again, the agreement between measured uranium levels and intended levels was very close.

Third-party assay results of one of the ^{90}Sr spikes gave a result of 14.1 pCi/L, which was 17% lower than the PNL-calculated result. For the purpose of Round-One testing, this low result was not further investigated. The reasons for this were:

1. Out of 19 samples, it is statistically predictable that one or more results will be outside the 3σ limits.
2. No laboratories were failed in the ^{90}Sr category due to a negative bias on this set of samples.

3. The PNL-calculated results and third-party assay results were not statistically different (at the 95% confidence level) from the grand average of participating laboratories.

Particular attention will be devoted to preparation of Round-Two strontium samples and additional assays of Round-One samples to resolve the issue.

APPROPRIATENESS OF PERFORMANCE CRITERIA

In evaluating the appropriateness of the performance criteria for radiobioassay presented in the draft ANSI Standard, it is important to consider whether the specified values of AMDA are adequate and reasonable and whether the limits that define the acceptable bias and precision of laboratory measurements are also appropriate. Results of the intercomparison testing program were used to evaluate the performance criteria specified in the draft Standard.

MINIMUM DETECTABLE AMOUNT (MDA)[a]

The minimum detectable amount (MDA) is an indicator of the detection capability of a laboratory's analytical method. The draft Standard provides "acceptable" minimum detectable amounts (AMDAs) for each radionuclide listed.

These AMDAs represent activity levels that are important for radiological protection reasons and are considered generally achievable by bioassay service laboratories. Bioassay laboratories should possess the ability to analyze samples containing activities equal to or above the AMDA to certain criteria of bias and precision.

Each bioassay service laboratory should be able to demonstrate that its own MDA is less than or equal to the AMDA. The MDA can be estimated by the bioassay service laboratory using the equation provided in the draft Standard:

$$MDA = \frac{4.65\ S_b}{E\ R\ V\ K}$$

where

4.65 = derived factor to limit Type I and II errors (Currie 1968)

S_b = standard deviation of an appropriate blank

E = counting efficiency

R = recovery

(a) The equation used to calculate MDA is currently under revision by Working Group 2.5.

V = sample volume

K = constant to convert to appropriate units.

This equation utilizes values for sample recovery and counting efficiency that pertain to the measurement of spiked samples. The corrections are based on long-term laboratory averages and result in an idealized estimate of MDA.

The MDA for each bioassay laboratory may also be estimated by a testing laboratory using the analytical results of the control urine samples[(a)] containing no added nuclide. These estimates contain actual values for recovery and efficiency that vary as would an actual sample. This method is similar to the lower detectable limit (LDL) as defined by Currie (1968). However, these estimates of MDA are determined from a small number of samples and thus may not always be accurate. Both of the above methods can provide a means for checking the stated MDA of a bioassay laboratory. They can also be used to evaluate the appropriateness of AMDA values in the draft Standard. These estimates are not, however, useful for testing and certifying the ability of a bioassay laboratory to analyze samples at or near the AMDA to the bias and precision criteria specified in the draft Standard.

As can be seen from the results presented in Table 4, the MDAs provided by participating bioassay laboratories generally agreed with PNL estimates based on intercomparison test results. There were, however, notable exceptions. To test whether a laboratory's MDA is less than or equal to the AMDA specified in the draft Standard, test samples should be analyzed at levels close to the AMDA value (1 to 10 AMDA). This is also supported by the data obtained in Round-One testing. Inspection of the individual data in Appendix F as well as the summary of B_r and S_B (Table 10) data for the high and low concentrations of the various nuclides showed that at the high testing level (approximately 15 times AMDA), nearly all of the laboratories participating in Round-One testing passed the draft Standard criterion. At the lower testing level (one to 10 times AMDA), however, laboratories often did not meet the draft Standard criteria for ^{241}Am, ^{238}Pu, or U-nat. The actual capability of laboratories to analyze samples at AMDA levels in routine operation would not be adequately tested in the range of 10 to 100 AMDA. Statistical fluctuations

(a) Control samples were not identified.

in the data at MDA levels may necessitate analysis of additional samples to determine if a laboratory's MDA is less than the defined AMDA.

The intercomparison test results provide an empirical estimate of the appropriateness (with regard to technical feasibility) of the suggested AMDA values in the draft Standard. The following discussion of appropriateness of AMDA levels considers only technical feasibility and not radiological protection needs. From Table 4, Table 10, and Appendix F, it can be seen that the AMDA for ^{3}H is approximately 15 to 100 times higher than typical MDAs reported. The AMDA for ^{3}H is thus easily achievable. For ^{90}Sr, the AMDA is slightly higher than both reported and estimated MDAs, and should probably remain unchanged. For ^{137}Cs, the MDAs ranged from 0.001 to 4.0 times the AMDA, indicating that the AMDA should probably be raised if health physics considerations permit. For the alpha emitters ^{238}Pu and ^{241}Am, the laboratory MDAs were highly variable but were usually higher than the AMDA, which indicates that the AMDA may need to be raised to a higher value if possible. It would appear that the AMDA for natural uranium is appropriate, but additional test data from Round-Two testing is needed before definitive conclusions can be made.

Most failures to meet draft Standard criteria were a result of a failure to meet AMDA requirements of the draft Standard (Table 9). This indicates the need to closely examine AMDA levels and re-evaluate radiological needs and analytical capabilities for some of the radionuclides.

RELATIVE BIAS, B_r

The relative bias is a component of the accuracy of the measurement system. It indicates how closely the analysis reports the true activity or amount in analyzed samples. It is defined in the draft Standard as

$$B_r = \text{relative bias}$$

$$= \sum_{i=1}^{N} B_{ri}/N$$

where

N = the number of samples, and

B_{ri} = the bias of a single measurement

$$= (A_i - A_{ai})/A_{ai}$$

where

A_i = the reported concentration, and

A_{ai} = the known concentration of the sample.

The relative bias allows comparison of samples at differing concentrations, and it is a good unbiased estimator. The draft Standard specifies that the relative bias is to be determined by at least three test samples and that bioassay laboratories should achieve a relative bias of $-0.25 \leq B_r \leq +0.50$ in the testing range of 10 to 100 times the AMDA. Approximately 12% of the Round-One measurements failed because relative biases were outside the recommended limits (Table 9).

Table 11 shows that bioassay laboratories had a fairly high average bias for ^{241}Am, ^{238}Pu, and U-nat. The individual data for these nuclides (Appendix F) shows that the analytical results were outside the bias criterion of the Standard (-0.25 to +0.50) 20% of the time for laboratories analyzing ^{238}Pu and ^{241}Am and 29% of the time for laboratories analyzing U-nat. All of these failures were at the lower level of activity (approximately 1.5 AMDA), and most of laboratories that experienced difficulty reported MDA > AMDA. In addition several of the laboratories reported instrumental difficulties during Round One, so the number of laboratories with large bias may be significantly lower following Round-Two testing. The laboratories generally reported bias in the range of 0.05 to 0.10 for nuclides in the upper activity range (10 to 100 AMDA).

The proposed performance criterion for the relative bias appears appropriate at or near the AMDA but may be unnecessarily large when applied to measurements at higher levels (10 to 100 AMDA). Tighter relative bias limits may be more appropriate for testing in the higher activity range. The test data will

be reevaluated at the end of Round-Two testing and a recommendation forwarded to Working Group 2.5.

A consistent negative bias was observed in the ^{90}Sr measurement results. Although only one laboratory failed the bias criterion, this observed trend may indicate deficiencies in the radiochemical procedures used to measure ^{90}Sr. Third-party crosscheck results were also lower than the intended activity levels. This apparent trend will be further investigated in Round-Two testing.

RELATIVE PRECISION, S_B

The relative precision is a measure of the reproducibility of an analysis. It is defined in the draft Standard as the standard deviation of the bias:

$$S_B = \sqrt{\sum_{i=1}^{N} (B_{ri} - B_r)^2 / N - 1}$$

This expression can be rearranged and expressed as simply:

$$S_B = \frac{S}{A_{ai}}$$

where S is the standard deviation of a series of measurements and A_{ai} is the known concentration of nuclide as defined above.

For in-vitro samples with identical activities, S_B is the relative dispersion of measurement values from their mean. According to the draft Standard, the relative precision should be less than or equal to 0.4 in the testing range of 10 to 100 times AMDA. Only 4% of the measurements in Round One failed to meet the relative precision criterion.

Although there were very few failures as a result of failure to meet S_B criterion, those failures occurred in the analysis of ^{238}Pu and ^{241}Am (Table 11 and Appendix F). Again we find that the failures occurred in the lower end of the testing range, usually for laboratories that also report MDA > AMDA. In the upper testing range, all laboratories met the criterion and the experimental S_B values generally ranged from 0.05 to 0.20.

The proposed criteria for relative precision appear to be reasonable at or near the AMDA, although more than 95% of the laboratories tested in Round One could meet a smaller S_B of 0.30 a majority of the time in the 1 to 10 AMDA range. A higher degree of precision may be desirable at testing levels 10 to 100 times AMDA. Results from Round-Two testing will shed further light on this observation.

EVALUATION SUMMARY

One important assumption made in these statistics is that bioassay results are normally distributed (follow a Gaussian distribution). Plots of S_B from Round-One testing on normal-probability and log-probability paper show best fit to normal distributions. Tests of skewness also indicate that the distributions are normal. Each in-vitro assay involves three samples each of a blank or zero concentration, a concentration just above the AMDA, and a concentration 15 to 20 times the AMDA. The analysis data are limited and the assumption of normality will be more fully tested when additional measurement results become available following Round-Two testing.

The data from Round-One testing, although limited, indicate that some revision of the draft Standard criteria for S_B and B_r may be indicated if preliminary results are confirmed by Round-Two testing. We have suggested retaining the present limits of S_B and B_r in the 1 to 10 AMDA testing range and tightening the limits to $B_r = \pm 0.25$ and $S_B \leq 0.25$ in the 10 to 100 AMDA range. It is appropriate to investigate what effect this reduction in error limits would have on the certification (pass/fail) of the laboratories that participated in Round-One testing.

Inspection of the individual laboratory data in Appendix F shows that two additional laboratories would have failed as a result of these changes. Indeed, of the laboratories that failed to meet one or more of the present criteria for the standard (including AMDA criteria), only two laboratories would have failed to meet these proposed criteria for S_B and B_r. In both of these cases, failure could be directly traced to instrumental failures independent of the laboratories' analytical ability.

It is also necessary to consider the impact of more restrictive S_B and B_r criteria on the ability of a laboratory to pass multiple testing categories. Round-One and Round-Two test results will be examined under various sets of criteria to address this concern.

These considerations also illustrate that it is the inability to meet the AMDAs defined in the draft Standard that is the most common cause of failure. Indeed, if those laboratories that could not pass the criteria for AMDA were to be excluded from consideration, there would be no failures due to S_B or B_r. In order to apply a single (or double) set of S_B and B_r criteria to the varied nuclides in the draft Standard, it is important to relate AMDA to MDA in a consistent manner. This could be accomplished initially by defining AMDA as a multiple of the average estimated MDAs listed in Table 4 or in some other manner. An assessment of the appropriateness of the draft Standard criteria for all of the nuclides is very difficult without some method of standardizing the AMDA/MDA ratio.

RECOMMENDATIONS

Recommendations to the committee preparing ANSI Standard N13.30 and proposals for conducting Round Two of the in-vitro bioassay study are presented here.

SUGGESTED REVISIONS TO THE DRAFT ANSI STANDARD N13.30

During the performance of this project there have been frequent discussions between members of this project and members of the Health Physics Society Working Group 2.5 preparing the draft Standard. Project representatives have attended each of the work meetings of the working group to insure that the intercomparison testing corresponded to the recommendations of the draft Standard. Project staff members provided numerous suggestions for improving the draft Standard during these meetings. Many of these recommendations were incorporated or are currently under consideration by the working group. For example, recommendations have involved the following:

- definition of terms
- procedures for in-vivo testing
- procedures for in-vitro testing
- revision of statistical formulas
- selection of categories and radionuclides for both in-vitro and in-vivo testing
- descriptions of phantoms for in-vivo testing (torso, whole-body, and neck phantoms)
- descriptions of quality control procedures.

Listed below for consideration by the committee are suggestions for future revision of the draft Standard.

1. The Standard should contain a discussion of the radiological basis for each of the AMDAs, including literature references.[a] If the AMDA level

(a) Working Group 2.5 is presently preparing an appendix to address this point.

obtained from relevant sources is not analytically feasible at this time, then it may need to be revised upward but would be regarded as a temporary value that could change as analytical capabilities improve. As discussed in the previous section, a high percentage of failures were the result of failure to report an MDA less than or equal to the defined AMDA of the draft Standard. This would indicate a need to closely examine the defined AMDA values for individual nuclides and adjust them if necessary.

2. The draft Standard should provide guidance to assure that standard blank and sample counting times are used to calculate the MDA. MDA estimates should be calculated with average count times used for routine samples. This would prevent laboratories from meeting AMDA levels by counting test samples for unrealistic times or by using very long background counts to calculate MDA. In several cases where the estimated MDA (supplied by the bioassay laboratory) was much different from a PNL-calculated MDA (calculated from sample results), this was the apparent source of the discrepancy.

3. When conducting a laboratory certification program, a testing laboratory should test S_B and B_r at or near the AMDA levels (1 to 10 AMDA) in addition to higher levels of activity (10 to 100 AMDA). The results of Round-One testing show that most of the laboratories are able to meet the analytical performance criteria (S_B and B_r) of the draft Standard at the current AMDA levels. In routine bioassay, most samples analyzed contain very low or nondetectable activity. We do not feel that the laboratory testing for certification should rely solely on estimates of MDA provided by the laboratories to test the criterion of MDA $\leq$ AMDA. If Round-One test results are confirmed by Round-Two testing, the S_B and B_r levels established by the standard appear unnecessarily broad in the 10 to 100 AMDA testing range. An S_B of 0.40 in the range of 1 to 10 AMDA and 0.25 in the range of 10 to 100 AMDA would increase confidence in the bioassay measurements. A bias of B_r of -0.25 to + 0.50 likewise may be acceptable at or near the AMDA, but, considering analytical capabilities, seems unnecessarily large when applied to assay results at the higher levels. Based on Round-One data, a B_r of ± 0.25 in the 10 to 100 AMDA range would be reasonable and would increase statistical confidence in data received

from bioassay laboratories. Final recommendation of test criteria will await analysis of Round-Two data and investigation into the effects of testing multiple categories.

4. The chemical and physical forms of various nuclides in natural urine and feces are often unknown and are not necessarily stable (as would be required for a standard) when they are known. The use of artificial urine offers significant savings in terms of cost and reproducibility in the administration of an ongoing testing program. It is recommended that artificial urine be further tested to show that the measurement results are equally applicable to those that might be obtained using a natural urine matrix. Following further successful testing, artificial urine should be specified in the draft Standard as the test matrix of choice.

5. A testing program should be developed to evaluate the appropriateness of draft Standard performance criteria for the bioassay analysis of fecal samples.

PROPOSED ROUND-TWO TESTING

A second round of in-vitro testing is planned. It will be similar to the first round. The purpose of Round Two is: 1) to determine whether laboratories have improved their capabilities, 2) to obtain a larger sample measurement data base, and 3) to test revisions in the draft Standard that were incorporated during Round-One testing. Table 12 shows the radionuclides and testing ranges chosen for Round Two.

TABLE 12. Proposed In-Vitro Testing Categories and Ranges for Round-Two Urinalysis

Category	Nuclide	Testing Range
Liquid scintillation	^{3}H	0.1 - 2.0 µCi/L
Beta counting	^{89}Sr + ^{90}Sr	10 - 200 pCi/L
Alpha spectrometry	^{238}Pu + ^{241}Am	0.06 - 1.2 pCi/L
Mass determination	U-nat	5 - 100 µg/L
Gamma spectrometry	^{137}Cs +	60 - 1,200 pCi/L
	^{60}Co	50 - 1,000 pCi/L

The first-round testing provided valuable information for developing the second-round plan. During the first-round intercomparison, a number of

laboratories failed to report results for the measurement of natural uranium, ^{241}Am, and ^{238}Pu. It will therefore be important to gather additional results for these categories. We also observed consistently high values of B_r and S_B for ^{90}Sr over the entire test range. A second round of testing may indicate whether this trend will continue.

One problem that became evident during the first round of testing was the failure of some laboratories to analyze the samples and to return the results within 30 days as requested. These laboratories were contacted by telephone at least twice for their results. Nonetheless, a number of participants failed to report test results (Table 7). Another problem was that some laboratories did not follow the format required for reporting of test results and some essential information was omitted. Action will be taken during Round Two to reduce these problems. We plan to communicate more frequently with participants and to stress the importance of complete results. The time permitted for sample analysis will be changed from 30 to 60 days.

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APPENDIX A

SAMPLE LETTER

APPENDIX A

SAMPLE LETTER

REQUEST FOR ESTIMATED MDA AND ERROR

February 23, 1982

Dear

RE: Technical Evaluation of Draft ANSI Standard N13.30.

Thank you for responding to our recent invitation to participate in a bioassay intercomparison study to evaluate the performance criteria contained in draft ANSI Standard 13.30. The draft standard is currently being revised and there may be some additional changes in the choice of test radionuclides and testing ranges. For example, we are currently anticipating the nuclides and ranges shown in Table I. You may notice that we have deleted Ra-226 and the category of gross alpha measurements. In addition, we plan to mix Pu-238 with Am-241, and Cs-137 with Sr-90 in the samples sent out for assay.

The purpose of this program is to evaluate the appropriateness of performance criteria in draft ANSI Standard 13.30, Performance Criteria of Radiobioassay, and measure the performance of existing bioassay laboratories against the criteria specified in that standard. The end result of the program will be a manual detailing procedures and criteria by which laboratories providing bioassay services will be "accredited".

We would appreciate your estimate of a minimum detectable activity (MDA) and its associated standard deviation for each nuclide you wish to qualify for. Please use the attached form. We would also request an estimation of uncertainty for the other three levels of activity listed on the attached sheet. The cumulative data will be used to guide in selection of final

testing levels, numbers of samples, and for further evaluation of acceptable minimum detectable activity (AMDA) in the draft ANSI standard. Each response will be held in strictest confidence.

We are planning to use artificial urine in this project. The artificial urine will contain inorganic and biological constituents. If you have comments regarding this option, we would greatly appreciate them. Please try to be specific in your criticism or support.

We feel that your cooperation is essential to the development of a good accreditation procedure and the best possible standard.

Very truly yours,

TABLE 1. In-Vitro Testing Categories, Radionuclides, and Testing Range

Procedure	Radionuclide	Range	
Liquid scintillation	^{3}H	2.0-200	µCi/L
Alpha spectrometry	^{241}Am	0.06-6.0	pCi/L
	^{238}Pu	0.05-5.0	pCi/L
Beta measurements	^{90}Sr	0.01-1.0	nCi/L
Fluorescence measurements	U-nat	5.0-500	µg/L
Gamma spectrometry	^{137}Cs	0.04-4.0	nCi/L

Return to:

Pacific Northwest Laboratory
Health Physics Technology
Group V Building
Richland, WA 99352

Nuclide _____

AMDA suggested by Draft ANSI N13.30 = _____

Estimated MDA ± 1σ = _____ Count time _____

MDA = minimum detectable activity

$$= \frac{S_b \times 4.65}{E \times R \times V \times K}$$

S_b = standard deviation of appropriate blank = _____

E = counting efficiency expressed as a decimal = _____

R = recovery expressed as a decimal = _____

V = sample size = _____

K = conversion factor to convert dpm to appropriate units = _____

1. AMDA ± 1σ = _____ Count time _____

2. 20 AMDA ± 1σ = _____ Count time _____

3. 100 AMDA ± 1σ = _____ Count time _____

Comments: artificial urine, proposed AMDA, other.

APPENDIX B

SAMPLE PREPARATION PROCEDURE

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SAMPLE PREPARATION PROCEDURE

I. BOTTLE PREPARATION, FILLING AND SHIPPING

1. All bottles and caps (50-L carboys, 2-L and 250-ml bottles) are filled with a 4% solution of Radiacwash® and allowed to soak for 24 hours or more.
2. The bottles and caps are rinsed exhaustively with tap water, followed by 2 rinses with deionized water. The deionized water is prepared by passing tap water through a purification system consisting of a 20-micron prefilter cartridge followed by a mixed bed ion exchange cartridge, a charcoal cartridge and finally a 5-micron scrubbing cartridge.
3. The bottles and caps are then air dried. To guard against dust particles falling into the bottles they are either laid on their sides or covered with paper towels during drying.
4. After the bottles have dried the caps are screwed on the bottles and the labels prepared and affixed as below.
5. The labels for the bottles are prepared and taped on the bottles. The labels are standard adhesive backed paper. The labels as covered with a strip of clear 2½-inch tape to further protect the information on them.
6. The capped, labeled bottles are then weighed to ±0.015-g accuracy on a top loading balance.
7. Aliquots of spiked artificial urine are then delivered to each bottle, as described below (steps 8-16). Procedures for preparing the spiked artificial urine are discussed in Section II.
8. The 50-L carboy of spiked artificial urine is positioned near a sample dispensing apparatus consisting of a peristaltic pump, speed control

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® Atomic Products Corp., Center Moriches, New York.

and a handheld remote switch. The supply tubing is placed in the 50-L carboy and 1300-1400 ml (or appropriate volume) of sample are delivered to each bottle.

9. Each bottle is immediately capped and reweighed to the nearest ±0.15 g on a top-loading electronic balance. After weighing, all caps are taped to the neck of the bottle with stretch vinyl tape.

10. All weights are recorded in the laboratory notebook or the balance printout tape is affixed to the notebook.

11. The bottles and laboratories are randomly selected by drawing numbered chits from a container.

12. The assigned sample bottles are then placed in 9½ in. by 9½ in. by 12 in. DOT approved boxes (type 12B), 3 bottles to a box. Vermiculite is poured around each bottle until the box is full. The bottles are lifted slightly to assure there is a layer of vermiculite beneath them.

13. The following sheets are inserted in each box:
    a. Instructions to Laboratories
    b. QA/QC Guidelines
    c. Data Report Sheets.

14. The box is taped securely shut and shipped after the following labels are affixed:
    a. Address Label
    b. Corrosive Liquids Label (NOS 1760)
    c. Corrosive Liquids Label (diamond shape)
    d. "This Side Up" Label.

15. One sample from each radionuclide batch (50-L carboy) is sent to an analytical laboratory for confirmation of calculated dilution.

16. The remaining samples (7-8 from each level) are stored in the laboratory.

II. PREPARATION OF SPIKED ARTIFICIAL URINE

1. Verify the presence of a valid calibration label on all balances to be used.

2. Place a one-inch-by-six-inch magnetic stirring bar in the carboy and tare weigh the carboy, bar, and caps. Insure that there are 13 to 16 inches of nonmagnetic material between the magnetic stirring bar in the bottom of the carboy and top of the balance. This is necessary to alleviate disturbances in the accuracy of the balance due to the magnetic field from the stirring bar.

3. Place about 35 kg of water in a 50-L carboy. Record the weight of the added water in the laboratory notebook (or use the printer tape).

4. Start the magnetic stirrer and add the various components of the artificial urine while stirring. Record all sample and tare weights in the laboratory notebook (or use printer tape).

5. Calculate the quantity of 50% concentrated nitric acid to be added so that the final calculated concentration of acid will be equivalent to 50 g of concentrated (70%) HNO_3 per liter of solution, or approximately 0.55 M following addition of radionuclide standard.

6. Add the appropriate diluted NBS-supplied standard spike (see Section III for details) and calculate the weight of water and acid still required as shown below for a 50-kg batch:

$$50\ kg - [W_u + W_w + W_s + W_{aa}] = W_{wa}$$

where: $2.5\ kg - [0.5\ W_a + 0.5\ W_s] = W_{aa}$

W_u = weight of artificial urine constituents (kg)
W_w = weight of water previously added (kg)
W_s = weight of standard spike (kg)
W_a = weight of acid added in step 5 (kg)
W_{wa} = weight of water to be added (kg)
W_{aa} = weight of acid to be added after spike

7. Add W_{aa} and approximately 95% of the water required W_{wa} and stir for 30 minutes.

8. Use the floor crane to lift the carboy from the magnetic stirrer to the 60-kg top-loading balance and add water until the final weight (W_f) of the carboy contents reaches 50 kg (± 1.5 g).

9. Remove the carboy from the balance, and place it on the magnetic stirrer. Stir vigorously for at least 30 minutes before dispensing samples.

III. PREPARATION OF NBS STANDARD SPIKE FOR ADDITION TO ARTIFICIAL URINE

1. Dilutions are performed in 2-L polyethylene bottles which have been washed and dried as previously discussed (Section I).

2. Calculate the amount of NBS standard to be added to the dilution bottle as below:

$$W_{sa} = \frac{(C_{FD})(W_{FD})}{(C_s)}$$

where

W_{sa} = Appropriate weight (g) of the NBS standard to be added to the dilution bottle.

W_{FD} = Final desired weight (usually 50 kg) of total spiked artificial urine batch.

C_{FD} = Final desired nuclide concentration in artificial urine. (activity or mass/kg)

C_s = Concentration of nuclide in NBS supplied standard. (activity or mass/g)

3. Add a 50% solution of concentrated nitric acid (35% actual HNO_3 concentration) to the polyethylene dilution bottle according to the following equation:

$$W_a = 1000g - W_{sa}$$

where

W_a = weight of acid solution to add (g).

W_{sa} = Calculated weight of NBS Standard to be added to artificial urine (g).

4. Open enough NBS standard vials to provide sufficient nuclide and aspirate the standard solution into a disposable Reservial.®

5. Weigh the vial and standard and dispense the calculated weight (W_{sa}) of standard in the polyethylene bottle.

6. Cap the bottle and mix thoroughly.

7. Weigh the bottle and pour the contents into a batch of artificial urine. Reweigh the bottle to calculate the exact amount of standard delivered (W_{sd}).

8. Calculate the final actual concentration of nuclide added to the carboy as below:

$$C_{FC} = \frac{(W_{sd})(C_s)}{(W_f)}$$

C_{FC} = Final calculated nuclide concentration in artificial urine (activity or mass per kg).

W_{sd} = Weight of NBS standard delivered to the artificial urine (g).

W_f = Final weight of carboy contents from Step 8 Section II.

C_s = Concentration of nuclide in NBS supplied standard (activity or mass per g).

® Disposable plastic ampule, Perfector Scientific, Atascadero, California.

APPENDIX C

INSTRUCTIONS TO PARTICIPATING IN-VITRO LABORATORIES

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INSTRUCTIONS TO PARTICIPATING IN-VITRO LABORATORIES

1. Log in the receipt of samples and send a list of samples received to PNL. Samples will be labelled with an identification number in the format shown below:

   A-Sr-0001-7356-32 where

   A = Laboratory code (A, B, C, D, etc.)
   Sr = Strontium or other nuclide
   0001 = Sample identification number 0001-9999
   7356-32 = PNL identification number.

2. The date on the label of each bottle is the date that the nuclide was added to the urine and all data should be decay corrected to that date, if necessary.

3. Use the identification number in all subsequent bookkeeping and correspondence.

4. Send complete analytical procedures, including QA, wet chemistry, counting and data reduction, to PNL for review.

5. Report measurement results within 30 days after receipt of samples and use the report sheets provided.

6. PNL will send participants summary sheets containing the results of all participating laboratories following receipt of data.

7. The identity of participating laboratories and the content of any procedures sent to PNL will not be revealed to any person or agency of the government or private sector without the prior consent of the participant.

APPENDIX D

IN-VITRO MEASUREMENTS REPORT FORM

IN-VITRO MEASUREMENTS REPORT FORM

Nuclide_____ Sample Preparation Date_____

Name of Laboratory and/or Code Letter_____

Contact Person_____ Phone ( )_____

Date of Receipt_____ Date of Analysis_____

Method of Storage:

Sample Manipulations:

Analytic Method:

Apparatus/instrumentation Used:

Sample No.	Total Counts	Count Time	Background Count Rate	Counting Efficiency	Recovery	Sample Assay	Est. Error	Units

S_b = _____ E = _____ R = _____ MDA = _____

Please return this form by _____ to: Al Robinson
Pacific Northwest Laboratory
ESB, Room 9
Richland, WA 99352
(509) 375-2065

INSTRUCTIONS: IN-VITRO MEASUREMENTS REPORT FORM

1. Method of Storage - room temperature, frozen, etc.
2. Sample Manipulations - addition of acid, division of sample, etc.
3. Analytical Method - brief description, or reference the procedure submitted previously to PNL. Indicate differences from routine procedure, if any.
4. Data - do not round off.
5. Count time - minutes.
6. Estimated Error - total error due to counting statistics, systematic errors, and error propagated during calculation of efficiency, recovery, etc.
7. Units - μCi/ℓ or pCi/ℓ for radionuclides; μg/ℓ for natural uranium.
8. MDA (Minimum Detectable Activity) - defined in the Draft ANSI Standard N13.30 as

$$MDA = \frac{S_b \times 4.65}{E \times R \times V \times K}$$

where

S_b = standard deviation of appropriate blank
E = counting efficiency expressed as a decimal fraction
R = recovery, expressed as a decimal fraction
V = sample size
K = conversion factor to convert to appropriate units
4.65 = derived factor to limit type I and II errors.

Note: The MDA is not to be determined by these sample analyses, but rather is to be derived from previous laboratory experience.

APPENDIX E

DETERMINATION OF MDAs

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DETERMINATION OF MDAs

The standard method of Currie (1968) was used to predict the minimum detectable amount based solely on the standard deviation of the control sample.

We assumed that the type 1 and type 2 errors (α and β) were equal and that random errors were normally distributed. We also assumed that the standard deviation of the signal at the detection point was equal to the standard deviation of the control, such that

if $\alpha = \beta = 67\%$, then MDA = $2.32\ \sigma$
if $\alpha = \beta = 95\%$, then MDA = $4.65\ \sigma$
if $\alpha = \beta = 99\%$, then MDA = $6.98\ \sigma$

In addition we assumed for this estimate that blanks and samples are counted for the same period of time. To generate the data in Table 4 (page 12) we chose $\alpha = \beta = 95\%$ so

$$MDA = 4.65\ \sigma_c$$

where

σ_c = one standard deviation of the control sample test results.

APPENDIX F

IN-VITRO BIOASSAY RESULTS

APPENDIX F

IN-VITRO BIOASSAY RESULTS

DEFINITION OF TERMS

a. MDA = "Minimum Detectable Amount," estimate supplied by bioassay laboratories.

b. AMDA = "Acceptable Minimum Detectable Amount," as defined by Draft ANSI Standard N13.30.

c. B_r = Relative Bias, as defined by Draft ANSI Standard N13.30.

d. S_B = Relative Precision, as defined by Draft ANSI Standard N13.30.

e. NR = no data returned.

f. Standard Criteria

$$-0.25 \leq B_r \leq 0.50$$

$$S_B \leq 0.40$$

$$MDA \leq AMDA$$

TABLE F.1. ^{3}H In-Vitro Bioassay Results. AMDA = 0.1 µCi/L

Laboratory	Nuclide Added (µCi/L)	Assay Results (µCi/L)	Calculated B_r	Calculated S_B	Laboratory MDA (µCi/L)	Standard Criteria Met Yes	Standard Criteria Met No
G	0.00	0.00	---	---	0.0015	---	---
	0.0295	0.0290	-0.02	0.03	0.0015	X	
	0.301	0.294	-0.02	0.01	0.0015	X	
	3.54	3.443	-0.03	0.02	0.0015	X	
J	0.00	0.0014	---	---	0.0078	---	---
	0.0295	0.038	0.28	0.10	0.0078	X	
	0.301	0.36	0.18	0.02	0.0078	X	
	3.54	4.24	0.20	0.01	0.0078	X	
K	0.00	0.0016	---	---	NR	---	---
	0.0295	0.0230	-0.22	0.02	NR		X
	0.301	0.2271	-0.24	0.01	NR		X
	3.54	2.688	-0.24	0.00	NR		X
L	0.00	-0.0006	---	---	0.00096	---	---
	0.0295	0.027	-0.08	0.00	0.00096	X	
	0.301	0.289	-0.03	0.01	0.00096	X	
	3.54	3.347	-0.05	0.08	0.00096	X	
M	0.00	0.00077	---	---	0.003	---	---
	0.029	0.0301	0.02	0.03	0.003	X	
	0.301	0.2932	-0.03	0.00	0.003	X	
	3.54	3.494	-0.01	0.00	0.003	X	
N	0.00	0.000	---	---	0.006	---	---
	0.0295	0.0217	-0.16	0.09	0.006	X	
	0.301	0.294	-0.02	0.01	0.006	X	
	3.54	3.543	-0.01	0.01	0.006	X	
S	0.00	0.000	---	---	0.007	---	
	0.0295	0.0284	-0.04	0.02	0.007	X	
	0.301	0.282	-0.06	0.01	0.007	X	
	3.54	3.36	-0.05	0.00	0.007	X	
V	0.00	0.00041	---	---	0.00096	---	
	0.0295	0.0279	-0.06	0.02	0.00096	X	
	0.301	0.276	-0.08	0.01	0.00096	X	
	3.54	3.30	-0.07	0.01	0.00096	X	

TABLE F.2. ^{241}Am In-Vitro Bioassay Results. AMDA = 0.06 pCi/L

Laboratory	Nuclide Added (pCi/L)	Assay Results (pCi/L)	Calculated B_r	Calculated S_B	Laboratory MDA (pCi/L)	Standard Criteria Met Yes	Standard Criteria Met No
E	0.000	0.14	---	---	0.44	---	---
	0.089	0.11	0.54	0.36	0.44		X
	0.910	0.72	-0.21	0.07	0.44		X
M	0.000	-0.027	---	---	0.06	---	---
	0.089	0.071	-0.21	0.20	0.06	X	
	0.910	0.790	-0.03	0.16	0.06	X	
O	0.000	0.0211	---	---	0.025	---	---
	0.089	0.109	0.26	0.24	0.025	X	
	0.910	0.853	-0.03	0.05	0.025	X	
S	0.000	0.008	---	---	0.06	---	---
	0.089	0.167	0.89	0.93	0.06		X
	0.910	1.014	0.11	0.12	0.06	X	
V	0.000	0.011	---	---	1.527	---	---
	0.089	0.081	-0.09	0.25	1.527		X
	0.910	0.849	-0.07	0.08	1.527		X

TABLE F.3. ^{238}Pu In-Vitro Bioassay Results. AMDA = 0.06 pCi/L

Laboratory	Nuclide Added (pCi/L)	Assay Results (pCi/L)	Calculated B_r	Calculated S_B	Laboratory MDA (pCi/L)	Standard Criteria Met Yes	Standard Criteria Met No
E	0.000	0.20	---	---	0.44	---	---
	0.107	0.29	0.90	0.43	0.44		X
	0.928	0.85	-0.08	0.02	0.44		X
M	0.000	0.022	---	---	0.026	---	---
	0.107	0.167	0.56	0.66	0.026		X
	0.928	0.822	-0.11	0.22	0.026	X	
O	0.000	0.0011	---	---	0.022	---	---
	0.107	0.0860	-0.20	0.11	0.022	X	
	0.928	0.771	-0.17	0.06	0.022	X	
S	0.000	0.0005	---	---	0.06	---	---
	0.107	0.113	0.05	0.13	0.06	X	
	0.928	0.959	0.03	0.06	0.06	X	
V	0.000	-0.011	---	---	1.05	---	---
	0.107	0.113	0.05	0.35	1.05		X
	0.928	0.882	-0.05	0.05	1.05		X

TABLE F.4. ^{90}Sr In-Vitro Bioassay Results. AMDA = 10.0 pCi/L

Laboratory	Nuclide Added (pCi/L)	Assay Results (pCi/L)	Calculated B_r	Calculated S_B	Laboratory MDA (pCi/L)	Standard Criteria Met Yes	Standard Criteria Met No
B	0.00	-1.8	---	---	1.4	---	--
	16.9	12.9	-0.23	0.22	1.4	X	
	185	124	-0.33	0.05	1.4		X
E	0.00	4.64	---	---	5.0	---	---
	16.9	17.4	0.03	0.21	5.0	X	
	185	194	0.05	0.35	5.0	X	
F	0.00	2.46	---	---	2.46	---	---
	16.9	15.7	-0.07	0.21	2.46	X	
	185	143	-0.23	0.18	2.46	X	
G	0.00	0.11	---	---	2.0	---	---
	16.9	13.7	-0.19	0.02	2.0	X	
	185	172	-0.07	0.01	2.0	X	
I	0.00	4.9	---	---	9.8	---	---
	16.9	16.4	-0.03	0.28	9.8	X	
	185	141	-0.23	0.06	9.8	X	

TABLE F.5. U-nat In-Vitro Bioassay Results. AMDA = 5.0 µg/L

Laboratory	Nuclide Added (µg/L)	Assay Results (µg/L)	Calculated		Laboratory MDA (µg/L)	Standard Criteria Met	
			B_r	S_B		Yes	No
C	0.00	6.0	---	---	5.0	---	---
	7.24	14.0	0.93	0.24	5.0		X
	77.8	103	0.33	0.13	5.0	X	
E	0.00	9.34	---	---	5.0	---	---
	7.24	15.6	1.14	0.26	5.0		X
	77.8	77.3	-0.01	0.09	5.0	X	
H	0.00	1.0	---	---	NR	---	---
	7.24	5.0	-0.31	0.14	NR		X
	77.8	54.0	-0.30	0.02	NR		X
K	0.00	4.7	---	---	NR	---	---
	7.24	14.7	1.03	0.08	NR		X
	77.8	76	-0.02	0.13	NR		X
O	0.00	0.00	---	---	1.3	---	---
	7.24	7.05	-0.01	0.07	1.3	X	
	77.8	71.27	-0.08	0.02	1.3	X	
R	0.00	1.53	---	---	5.0	---	---
	7.24	9.73	0.34	0.06	5.0	X	
	77.8	84.83	0.09	0.09	5.0	X	
S	0.00	0.00	---	---	0.6	---	---
	7.24	7.47	0.03	0.08	0.6	X	
	77.8	80.0	0.03	0.04	0.6	X	

TABLE F.6. ^{137}Cs In-Vitro Bioassay Results. AMDA = 0.04 nCi/L

Laboratory	Nuclide Added (nCi/L)	Assay Results (nCi/L)	Calculated B_r	Calculated S_B	Laboratory MDA (nCi/L)	Standard Criteria Met Yes	Standard Criteria Met No
G	0.00	-0.006	---	---	0.019	--	---
	1.51	1.56	0.03	0.03	0.019	X	
	13.8	13.6	-0.01	0.04	0.019	X	
I	0.00	0.026	---	---	0.0157	---	---
	1.51	1.50	0.01	0.00	0.0157	X	
	13.8	14.0	0.02	0.00	0.0157	X	
J	0.00	0.050	---	---	0.07	---	---
	1.51	1.37	-0.09	0.06	0.07		X
	13.8	12.9	-0.06	0.00	0.07		X
K	0.00	0.02	---	---	NR	---	---
	1.51	1.34	-0.09	0.06	NR		X
	13.8	13.47	-0.02	0.00	NR		X
L	0.00	0.0022	---	---	0.034	---	---
	1.51	1.49	-0.015	0.008	0.034	X	
	13.8	13.73	-0.005	0.008	0.034	X	
M	0.00	-0.0056	---	---	0.06	---	---
	1.51	1.504	0.00	0.01	0.06		X
	13.8	13.64	-0.01	0.01	0.06		X
N	0.00	0.100	---	---	0.1	---	---
	1.51	1.37	-0.09	0.04	0.1		X
	13.8	11.80	-0.14	0.01	0.1		X
O	0.00	0.00	---	---	0.036	---	---
	1.51	1.254	-0.17	0.01	0.036	X	
	13.8	11.74	-0.15	0.01	0.036	X	

APPENDIX G

PROPAGATION OF ERROR IN SPIKED ARTIFICIAL-URINE SAMPLES

APPENDIX G

PROPAGATION OF ERROR IN SPIKED ARTIFICIAL-URINE SAMPLES

The methods used to estimate the total error in the in-vitro test samples were the same as those discussed by Kanipe (1977). Briefly we assumed that the individual components of the total error were independent, normally distributed variables and that propagation of error for the manipulation of various functions could be expressed as below.

Function	Error Formula
$Q = X \pm Y$	$\sigma Q = (\sigma_x^2 + \sigma_y^2)^{\frac{1}{2}}$
$Q = aX \pm bY$	$\sigma Q = (a^2 \sigma_x^2 + b^2 \sigma_y^2)^{\frac{1}{2}}$
$Q = XY$	$\sigma Q = XY (\sigma_x^2/X^2 + \sigma_y^2/Y^2)^{\frac{1}{2}}$
$Q = X/Y$	$\sigma Q = X/Y (\sigma_x^2/X^2 + \sigma_y^2/Y^2)^{\frac{1}{2}}$

Using the error formulas above, the equations detailed in Appendix B, and the error estimates quoted in the NBS certificates supplied for each nuclide, the total error in the prepared samples was estimated.

NUREG/CR-3809
DOE/NBM 1071
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UC-41

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DOE/NBM 1071
PNL-5248

2 TITLE AND SUBTITLE
Performance Testing of Radiobioassay Laboratories: In-Vitro Measurements, Pilot Study Report

3 LEAVE BLANK

4 DATE REPORT COMPLETED
MONTH: September YEAR: 1984

5 AUTHOR(S)
A.V. Robinson, D.R. Fisher, R.T. Handley, PNL

6 DATE REPORT ISSUED
MONTH: December YEAR: 1984

7 PERFORMING ORGANIZATION NAME AND MAILING ADDRESS (Include Zip Code)
Pacific Northwest Laboratory
P.O. Box 999
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8 PROJECT/TASK/WORK UNIT NUMBER

9 FIN OR GRANT NUMBER
NRC FIN B2417
DOE Contract DE-AC06-76RLO 1830

10 SPONSORING ORGANIZATION NAME AND MAILING ADDRESS (Include Zip Code)
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11a TYPE OF REPORT
Technical

b PERIOD COVERED (Inclusive dates)

12 SUPPLEMENTARY NOTES
Also sponsored by: Division of Operational and Environmental Safety
Health Physics Programs
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13 ABSTRACT (200 words or less)

The research program at the Pacific Northwest Laboratory entitled "Technical Evaluation of Draft ANSI Standard N13.30, Performance Criteria for Radiobioassay" is jointly sponsored by the Nuclear Regulatory Commission and the Department of Energy. It is a nationwide, two-round bioassay intercomparison study to test the analytical performance of both in-vitro and in-vivo bioassay laboratories and determine their ability to meet the minimum performance criteria specified in the draft ANSI Standard.

Round One is a Pilot Study involving a small number of voluntarily participating laboratories. Round Two will involve a larger number of laboratories and will expand on the results of Round One. This report, In-Vitro Measurements, is a review of the methodology and results of Round One. For this part of the research, test samples of artificial urine containing precisely known quantities of certain radionuclides were sent to 19 bioassay laboratories. Results showed that some of the participating laboratories had difficulty meeting the performance criteria specified in the current draft ANSI Standard N13.30. Based on these results, specific recommendations were made to the working group preparing the draft Standard.

14 DOCUMENT ANALYSIS - a KEYWORDS/DESCRIPTORS
radiobioassay
bioassay laboratories
in-vitro measurements
ANSI Standard N13.30
performance criteria
artificial urine
bioassay

b IDENTIFIERS/OPEN-ENDED TERMS

15 AVAILABILITY STATEMENT
Unlimited

16 SECURITY CLASSIFICATION
(This page) Unclassified
(This report) Unclassified

17 NUMBER OF PAGES

18 PRICE