**Environmental Monitoring Series** 

# ENVIRONMENTAL RADIOACTIVITY LABORATORY INTERCOMPARISON STUDIES PROGRAM 1975



NATIONAL ENVIRONMENTAL RESEARCH CENTER
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
LAS VEGAS, NEVADA 89114

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Research reports of the Office of Research and Development, U.S. Environmental Protection Agency, have been grouped into five series. These five broad categories were established to facilitate further development and application of environmental technology. Elimination of traditional grouping was consciously planned to foster technology transfer and a maximum interface in related fields. The five series are:

- 1. Environmental Health Effects Research
- 2. Environmental Protection Technology
- 3. Ecological Research
- 4. Environmental Monitoring
- 5. Socioeconomic Environmental Studies

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# ENVIRONMENTAL RADIOACTIVITY LABORATORY INTERCOMPARISON STUDIES PROGRAM 1975

by

Quality Assurance Branch Technical Support Laboratory National Environmental Research Center Las Vegas, Nevada

> ROAP Number 21BEK Program Element 1HA327

NATIONAL ENVIRONMENTAL RESEARCH CENTER OFFICE OF RESEARCH AND DEVELOPMENT U.S. ENVIRONMENTAL PROTECTION AGENCY LAS VEGAS, NEVADA 89114



#### PREFACE

Quality assurance is an integral part of any viable environmental monitoring activity. The primary goals of the U.S. Environmental Protection Agency's (EPA) quality assurance program are to improve and document the credibility of environmental measurements. To achieve these goals, quality assurance is needed in nearly all segments of monitoring activities and should cover personnel, methods selection, equipment, and data handling procedures.

Five major functions, each essential to an effective quality assurance effort, comprise EPA's quality assurance program.

- Promulgation of standardized methods of measurement
- Distribution of standard reference materials
- Issuance of guidelines and procedures
- Training and technical assistance
- Evaluation and certification of monitoring activities

This manual has been prepared to assist laboratories involved with environmental radiation measurements in developing and maintaining a quality control program and documenting the precision and accuracy of their data. All EPA monitoring programs are requested to make use of this document in planning their own radiation measurements and in assisting the States in carrying out radiation monitoring activities.

Comments concerning the utility of this document, along with any suggestions for possible changes and revisions, are welcomed. Questions on matters related to quality assurance of environmental measurements in various fields should be directed to the following person(s):

# Air Pollution

Mr. Thomas Clark Methods Standardization and Performance Evaluation Branch Quality Assurance and Environmental Monitoring Laboratory National Environmental Research Center Research Triangle Park, North Carolina 27711

#### Water

Mr. John Winter, Chief Quality Assurance and Laboratory Evaluation Branch Methods Development and Quality Assurance Research Laboratory National Environmental Research Center Cincinnati, Ohio 45268

### Pesticides

Mr. Jack Thompson, Chief Chemistry Branch Pesticides and Toxic Substances Effects Laboratory National Environmental Research Center Research Triangle Park, North Carolina 27711

# Radiation

Mr. Arthur N. Jarvis, Chief Quality Assurance Branch Technical Support Laboratory National Environmental Research Center Las Vegas, Nevada 89114

Periodically, manuals and documents will be issued which provide guidelines to be followed in all phases of monitoring activities. Use of these guidelines throughout the Agency will enable a uniform approach to be established within EPA which ultimately can be implemented at the State level. This should permit a significant improvement in the validity and reliability of environmental data collected throughout the Nation.

The implementation of a total and meaningful national environmental quality assurance effort cannot succeed without the full support of all monitoring programs. Your cooperation is appreciated.

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#### THE LABORATORY INTERCOMPARISON STUDIES PROGRAM

Environmental measurements of radiation are made daily by many Federal, State, local, and private agencies. The data from these measurements are used for a wide variety of purposes including assessment of health effects, the establishment of standards and guides, and for enforcement activities. It is therefore imperative that the precision and accuracy of the data be assured in order that policy decisions concerning environmental quality are based on valid and comparable data of known reliability.

In order to attain this goal, an Agency-wide quality assurance program has been implemented within the U.S. Environmental Protection Agency (EPA). In the area of radiation, quality control responsibilities have been assigned to the Quality Assurance Branch at the EPA's National Environmental Research Center-Las Vegas which carries out a program designed to encourage the development and implementation of quality control procedures for sample collection, laboratory analysis, and data handling and reporting.

A major objective of this program is to assist laboratories involved in environmental radiation measurements to develop and maintain both an intralaboratory and an interlaboratory quality control program. In part, this is accomplished through an extensive laboratory intercomparison study ("cross-check") program involving environmental media (milk, water, air, food, soil, and gases) and a variety of radionuclides with activities at or near environmental levels.

Simulated environmental samples, containing known amounts of one or more radionuclides, are prepared and routinely distributed to all laboratories upon request. These laboratories perform the required analyses and return their data to the Quality Assurance Branch for statistical analysis and comparison with a known value and analytical

values obtained by other participating laboratories. A report and a control chart are returned to each participant. The program thus enables each laboratory to document the precision and accuracy of its radiation data, identify instrumental and procedural problems, and to compare its performance with that of other laboratories.

Each laboratory making environmental measurements for radiation should have an internal quality control program in operation to insure that all instrumentation is calibrated and functioning, and that analytical procedures are being carried out properly. Such a program includes continual monitoring of instrumentation, the plotting of instrument control charts, frequent analysis of replicate samples to check precision, and the regular measurement of samples to which known amounts of activity have been added to check the accuracy of systems.

Participation in a laboratory intercomparison does not automatically assure the precision and accuracy of a laboratory's data and should not be considered as a substitute for a continuous quality control program within a laboratory. Intercomparison data may be useful for documenting precision and accuracy and helping to indicate instrumental or procedural problems. Participation in intercomparison studies is useful in augmenting a laboratory's quality control program and serves as a check on its internal quality control program.

You are encouraged to have your laboratory participate in the cross-check program of the Quality Assurance Branch of the NERC-LV's Technical Support Laboratory, or to expand its participation if you wish to do so.

#### TYPES OF ENVIRONMENTAL SAMPLES DISTRIBUTED

The current laboratory intercomparison studies program covers the analysis of a variety of media containing various levels of radio-activity. These include:

Milk: Four-liter milk samples containing potassium, strontium-89, strontium-90, iodine-131, cesium-137, and barium-140 are distributed on a bimonthly basis.

Water: Water containing several different mixtures of radioactive materials is included in the cross-check program.

- Four-liter samples for the analysis of gross alpha and gross beta activity are sent to participating laboratories bimonthly.
- Four-liter samples containing chromium-51, zinc-65, cobalt-60, ruthenium-106, cesium-134, and cesium-137 are distributed bimonthly for analysis of gamma emitters.
- Sixty-milliliter samples for tritium analysis are distributed on a bimonthly basis.
- Four-liter water samples containing plutonium-239 are shipped to laboratories during February and August of each year.
- Four-liter samples of well water containing radium-226 are distributed in January, May, July, and November.

Air: Air filters, with optional two-inch or four-inch diameters, are sent out on a quarterly basis for gross alpha, gross beta, plutonium-239, cesium-137, and strontium-90 analysis.

Soil: Hundred-gram soil samples, containing plutonium-239, will be distributed twice each year.

Food: Three 4-liter food slurries containing strontium-89, strontium-90, iodine-131, cesium-137, barium-140, and potassium are sent to participants quarterly.

Urine: Urine samples (60-ml) containing tritium are shipped on a quarterly basis.

Gases: Cylinders of air, containing krypton-85, are distributed twice each year.

Sample size, approximate activity levels, type of analysis, and other pertinent information concerning the cross-check samples are summarized in Table 1. The distribution schedule is outlined in Table 2.

A laboratory may participate in any one or all of the studies described.

Table 1. SUMMARY OF CROSS-CHECK PROGRAMS\*

SAMPLE	ANALYSIS	ACTIVITY PER ISOTOPE	QUANTITY SUPPLIED	PRESERVATIVE	DISTRIBUTION	TIME FOR ANALYSIS & REPORT
Milk	89Sr, <sup>30</sup> Sr, <sup>131</sup> I, <sup>137</sup> Cs, <sup>140</sup> Ba, K	< 200 pCi/l	ν 4 liters	Formalin	Bimonthly	6 weeks
Water						
Gross a, B*	055 α,	< 100 pCi/1	$\sim$ 4 liters	0.5 N HNO <sub>3</sub>	Bimonthly	4 weeks
Gamma	60Co, 106Ru, 134Cs, 137Cs, 51Cr, 65Zn	< 500 pCi/l	ν 4 liters	0.5 N HNO <sub>3</sub>	Bimonthly	4 weeks
3Н	Н°	< 3500 pCi/l	√ 60 ml	none	Bimonthly	4 weeks
239 <sub>7,1,4</sub>	239Pu	< 10 pCi/l	ι 4 liters	0.5 N HNO <sub>3</sub>	Semiannually	8 weeks
226Ra	226Ra	< 20 pCi/l	∿ 4 liters	0.5 N HNO <sub>3</sub>	Quarterly	6 weeks
Air						
Grossα, β*	α, β, γ	< 200 pCi/sample	3 - 2" or 4" diam. air filters	none	Quar ter ly	4 weeks
239Pu*	239Pu	< 2 pCi/sample	3 - 2" or 4" diam. air filters	none	Quarterly	6 weeks
Soil*	2°9Pu	< 50 pCi/sample	∿ 100 g	none	Semiannually	8 weeks
Diet	89Sr, 90Sr, 131I, 137Cs, 140Ba, K	< 200 pCi/kg	3 - 4-liter samples	Formalin	Quarterly	8 weeks
Urine	ЭН	< 3500 pCi/l	∿ 60 ml	Formalin	Quarterly	4 weeks
Gas	85Kr	< 20 pCi/ml	10 liters	none	Semiannually	8 weeks

\* Laboratories are required to have the necessary licenses before receiving these samples.

Table 2. CROSS-CHECK SAMPLE DISTRIBUTION SCHEDULE (Numbers indicate week of the month)

Gas	85Kr				1						1		
Urine	Нε			2			2			2			2
Diet	Sr, y				4				4				1
Soil	γ, <sup>239</sup> Pu					2						2	
Air Filter	Gross α,β <sup>239</sup> Pu		4				4			င			4
	226 <b>Ra</b>	3				ĸ		3				3	
	239Pu		-										
Water	3Н		2		2		2		2		2		2
	٨		က		3		က		က		3		е
	Gross a,8	4		4		4		4		4		4	
Milk	Sr, Y			-				г		П			
Month	in 1975	Jan	Feb	Mar	Apr	May	Jun	JuJ	Aug	Sep	0ct	Nov	Dec

#### ANALYSIS OF DATA

Each participating laboratory is expected to carry out three independent determinations for each radionuclide included in a particular cross-check study and to report its results on a form (Figure 1) provided with the sample.

Upon receipt of the reports from all participating laboratories, the data are transferred to punch cards for computer analysis. As indicated in the sample calculations, this analysis includes determination of the laboratory standard deviation, calculation of the normalized range, normalized deviation, sample standard deviation, and the grand average of all laboratories. The analytical precision values, used as a basis for judging laboratory performance for specific nuclides, are summarized in Table 3.

A report is generated containing data reported by participating laboratories, listed according to their identity code, along with the results of the analysis (Table 4). In addition, a control chart is generated and reproduced for each radionuclide included in the sample (Figure 2). The control charts are updated each time a laboratory participates in a particular cross-check study, thus giving each laboratory a continuous record of its performance.

A letter giving the known value for each radionuclide is mailed to participants approximately two weeks after the report due date. This is followed by a complete report which includes a copy of the computer printout and control chart.



# U.S. ENVIRONMENTAL PROTECTION AGENCY NATIONAL ENVIRONMENTAL RESEARCH CENTER LAS VEGAS, NEVADA

# QUALITY ASSURANCE BRANCH

Tritium in Urine Cross-check Program

Contact Person					-
Collection	Date	9-20-74	0400 hours	PST	-
	Analysis	#1		-	
	Analysis	#2		-	
	Analysis	#3		-	
	al activi		than 3500 pC		tracted.
Tot Please send your res U.S.	ults no l	ty is less ater than _	October	i/l <sup>3</sup> H.	
Tot Please send your res U.S. Nati Qual P.O.	ults no l Environm onal Envi	ater than _ ental Prote ronmental R ance Branch 7	October ction Agency	i/l <sup>3</sup> H.	
Tot Please send your res U.S. Nati Qual P.O.	Environmonal Environgity Assur Box 1502 Vegas, NV	ater than _ ental Prote ronmental R ance Branch 7 89114	October  ction Agency esearch Cente (TSQ)	i/l <sup>3</sup> H. 18, 1974 er	to:

Figure 1. Results Reporting Form

Table 3. LABORATORY PRECISION: ONE STANDARD DEVIATION VALUES FOR VARIOUS ANALYSES

Analysis	Level	Standard Deviation Single Determination
Gamma Emitters	5 to 100 pCi/liter or kg >100 pCi/liter or kg	5 pCi/liter 5% of known value
Strontium-89	5 to 100 pCi/liter or kg >100 pCi/liter or kg	5 pCi/liter 5%
Strontium-90	2 to 3 pCi/liter or kg >30 pCi/liter or kg	1.5 pCi/liter 5%
Potassium	≥0.1 g/liter or kg	5%
Gross Alpha	≤20 pCi/liter >20 pCi/liter	5 pCi/liter 25%
Gross Beta	≤100 pCi/liter >100 pCi/liter	5 pCi/liter 5%
Tritium	<4000 pCi/liter ≥4000 pCi/liter	ls (%) = 16985 × (pCi/liter)9067 10%
Radium-226	≥0.1 pCi/liter	15%
Plutonium-239	≥0.1 pCi/liter, gram or sample	10%

Table 4. SAMPLE ANALYSIS AND REPORT OF PARTICIPANT'S DATA

NERC-LV TRITIUM IN URINE CROSSCHECK PROGRAM --- SEPTEMBER 1974

09/20/74 SAMPLE - A 3H

	EXPECTED LABOR	RATORY PRECISION	(1S, 1 DE)	KNOWN-VALUE ERMINATION)		PCI/L PCI/L
LAB	RESULT	EXPERIMENTAL SIGMA	RNG ANLY (R + SR)	AVERAGE	NORMALIZEI (GRAND-AVG	) (KNOWN)
ΔN	NO DATA	PROVIDED				
CF CF	3269 3522 3632	186.1	•60	3474	•9	1.0
CM CM CM	3261 3373 3362	61.7	.19	3332	•2	• 3
СО	NO DATA	PROVIDED				
D D D	3060 3060 3240	103.9	.30	3120	8	7
J J	3255 3247 3294	25.1	.08	3265	1	0
Р	NO DATA	PROVIDED				
Z Z Z	3240 3340 3190	76.4	• 25	3257	-•2	1
EXP	ERIMENTAL SIGMA	A (ALL LABS) =	149	GRAND AVERAG	GE = 329	90

### Explanation of terms in Table 4:

Title: Program name, sample collection date, sample code letter, analysis type, known concentration of radio-nuclide, expected standard deviation of analysis - single determination.

Column 1: Laboratory identification code (A, B, C, etc.).

Column 2: Laboratory results (0-25 results listed down column).

Column 3: 1s (standard deviation) of the experimental results.

Columns 4 Normalized range value in "mean range + standard error and 5: of the range"  $(\bar{R} + \sigma_R)$  units for comparability. (See Statistical Techniques for Quality Control of Environmental Radioassay, AQCS Report Stat-1, November 1964, pages 4-8.) ( $S_R = \sigma_R$  for printing purposes.)

Column 6: Average value.

Column 7: Normalized deviation from the grand average value of all laboratories expressed in  $\sigma_M$  units.

Column 8: Normalized deviation from the known value expressed in  $\sigma_M$  units.

Bottom of 1s experimental error of all laboratories, and the Chart: grand average of all laboratories.

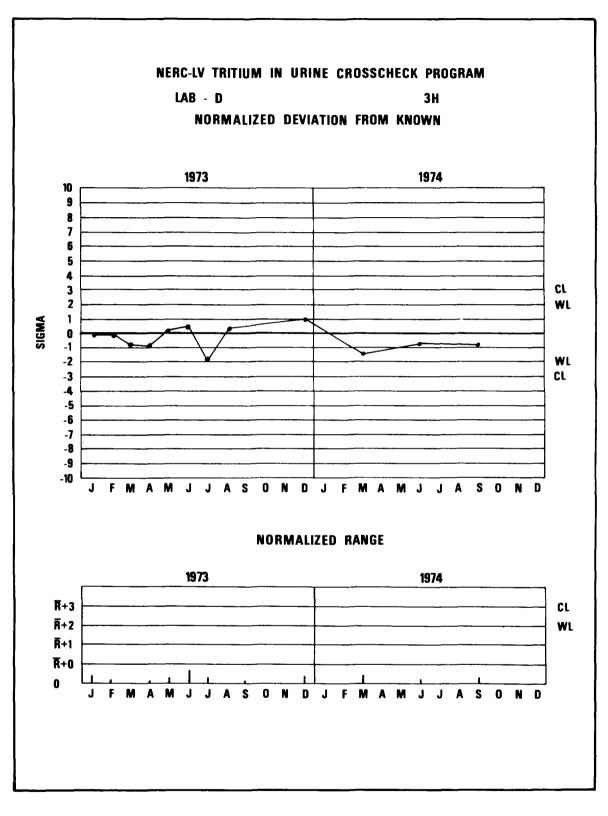


Figure 2. Control Chart

Explanation of terms used in the control chart (Figure 2).

Title: Name of program, laboratory code letter, and type

of analysis.

Upper Graph: "Normalized deviation from known versus the month

of analysis." [The 95.0% ( $\mu$  ± 2 $\sigma_M$ ) and the 99.7% ( $\mu$  ± 3 $\sigma_M$ ) confidence levels were chosen as the

warning levels and control limits respectively.]

Lower Graph: "Normalized range values ( $\bar{R}$  +  $\sigma_R$ ) versus the

month of analysis." [The 97.5% ( $\bar{R}$  + 2 $\sigma_R$ ) and  $\sim 100\%$  ( $\bar{R}$  + 3 $\sigma_R$ ) confidence levels were chosen as

the warning levels and control limits respec-

tively.]

#### PARTICIPATION IN THE LABORATORY INTERCOMPARISON STUDIES PROGRAM

Any laboratory involved in, or concerned with, environmental radiation monitoring and surveillance, is eligible to participate in any one or all of the cross-checks described. Moreover, dependent upon personnel available and their workload, a laboratory may elect to receive samples on a less frequent basis than indicated on the distribution schedule (Table 2).

To become a participant in the laboratory intercomparison studies program, complete one of the forms included at the end of this publication and return to:

U.S. Environmental Protection Agency National Environmental Research Center Quality Assurance Branch (TSQ) P.O. Box 15027 Las Vegas, NV 89114

Should the laboratory require additional types of cross-check samples at some later date, a second form indicating the samples desired should be submitted.

#### STATISTICAL CALCULATIONS

To illustrate the computations performed by the computer, example calculations are given using data for three actual samples analyzed at one laboratory (Laboratory D, see Table 4).

The experimental data are listed and the mean, range, and the experimental sigma are computed. These statistics provide measures of the central tendency and dispersion of the data.

The normalized range is computed by first finding the mean range,  $\bar{R}$ , the control limit, CL, and the standard error of the range,  $\sigma_R$ . The normalized range measures the dispersion of the data (precision) in such a form that control charts may be used. Control charts allow one to readily compare past analytical performance with present performance. In the example, the normalized range equals 0.3  $\bar{R}$  which falls inside the upper warning level,  $\bar{R} + 2\sigma_R$ . The precision of the results is acceptable.

The normalized deviation is calculated by computing the deviation and the standard error of the mean,  $\sigma_m$ . The normalized deviation allows one to readily measure central tendency (accuracy) through the use of control charts. Trends in analytical accuracy can be determined in this manner. For this example, the normalized deviation is -0.7 which falls within the upper and lower warning levels. The accuracy of the data is acceptable.

Finally, the experimental error of all laboratories, the grand average, and the normalized deviation from the grand average are calculated in order to ascertain the performance of all the laboratories as a group. Any bias in methodology or instrumentation may be found from these results.

EXAMPLE CALCULATIONS (Laboratory D Data, see Table 4)

# Experimental data:

Known value =  $\mu$  = 3273 pCi <sup>3</sup>H/liter urine on September 24, 1974 Expected laboratory precision =  $\sigma$  = 357 pCi/liter

Laboratory	Sample	Result
D	x <sub>1</sub>	3060 pCi/liter
D	x <sub>2</sub>	3060 pCi/liter
D	Хз	3240 pCi/liter

Mean =  $\bar{x}$ 

$$\bar{x} = \frac{\sum_{i=1}^{N} x_i}{N} = \frac{9360}{3} = 3120 \text{ pCi/liter}$$

where N = number of results

Range = r

r = |maximum result - minimum result|= |3240 - 3060| = 180 pCi/liter Experimental sigma = s

$$s = \sqrt{\frac{\sum_{i=1}^{N} (x_i)^2 - \frac{\left(\sum_{i=1}^{N} x_i\right)^2}{N}}{N-1}}$$

$$= \sqrt{\frac{(3060)^2 + (3060)^2 + (3240)^2 - \frac{(3060 + 3060 + 3240)^2}{3}}{2}}$$

$$= 103.9 \text{ pCi/liter}$$

Normalized range =  $w\bar{R} + x\sigma_{R}$ 

Mean range =  $\bar{R}$ 

$$\bar{R}$$
 =  $d_2\sigma$  where  $d_2$  = 1.693 for N = 3\*  
= (1.693)(357)  
= 604.4 pCi/liter

Control limit = CL

CL = 
$$\bar{R} + 3\sigma_R$$
  
=  $D_4\bar{R}$  where  $D_4$  = 2.575 for N = 3\*  
= (2.575)(604.4)  
= 1556 pCi/liter

Standard error of the range =  $\sigma_R$ 

$$\sigma_{R}$$
 = 1/3 ( $\bar{R}$  + 3 $\sigma_{R}$  -  $\bar{R}$ )  
 = 1/3 (D<sub>4</sub> $\bar{R}$  -  $\bar{R}$ )  
 = 1/3 (1556 - 604.4)  
 = 317.2 pCi/liter

<sup>\*</sup> Rosenstein, M., and A. S. Goldin, Statistical Techniques for Quality Control of Environmental Radioassay, AQCS Report Stat-1, U.S. Department of Health, Education and Welfare, PHS, Nov 1964

Normalized deviation of the mean from the known value = ND

Deviation of mean from the known value = D

D = 
$$\bar{x} - \mu$$
  
= 3120 - 3273  
= - 153 pCi/liter

Standard error of the mean =  $\sigma_m$ 

$$\sigma_{\text{m}} = \frac{\sigma}{\sqrt{N}}$$

$$= \frac{357}{\sqrt{3}}$$

$$= 206.1 \text{ pCi/liter}$$

ND = 
$$\frac{D}{\sigma_{\text{m}}}$$
  
=  $\frac{-153}{206.1}$   
= -0.7

Experimental sigma (all laboratories) =  $s_{+}$ 

$$s_{t} = \sqrt{\frac{\sum_{i=1}^{N} (x_{i})^{2} - \frac{\left(\sum_{i=1}^{N} x_{i}\right)^{2}}{N}}{N - 1}}$$

$$= \sqrt{\frac{162639133 - \frac{(49345)^{2}}{15}}{14}}$$

$$= 149 \text{ pCi/liter}$$

Grand average = GA

$$= \frac{\sum_{i=1}^{N} x_{i}}{N}$$

$$= \frac{49345}{15}$$

$$= 3290 \text{ pCi/liter}$$

Normalized deviation from the grand average = ND'

Deviation of the mean from the grand average = D'

$$D' = \bar{x} - GA$$
  
= 3120 - 3290  
= - 170 pCi/liter

$$ND' = \frac{D'}{\sigma_{m}}$$

$$= \frac{-170}{206.1}$$

$$= -0.8$$



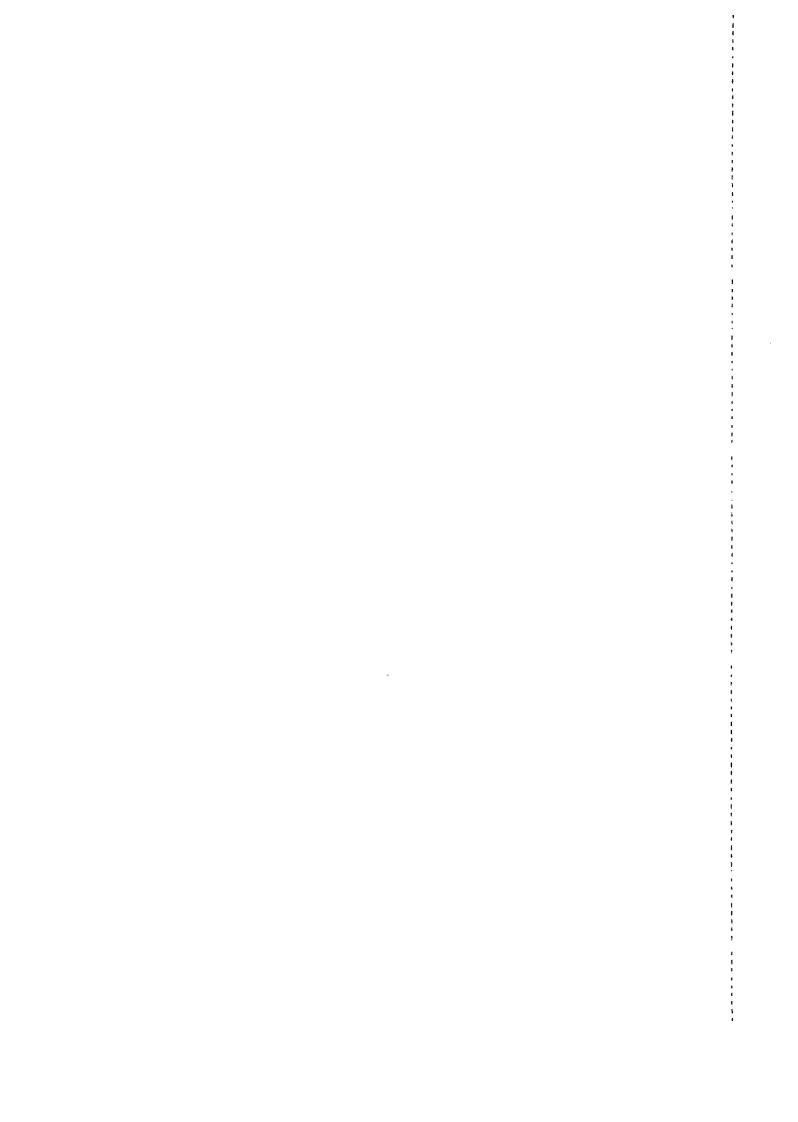
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SUBJECT: Participation in Intercomparison Studies Program

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		rs participation indicate months		
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SAMPLE TYPES SAMPLE TYPES	QUARTERLY SEMIANNUALLY ANNUALLY	SAMPLE TYPES	QUARTERLY SEMIANNUALLY ANNUALLY	
Milk: Sr, γ Water: Gross α, β γ 3H 239Pu		Air Filter: Gross α, β 239Pu Soil: <sup>239</sup> Pu Diet: Sr, γ Urine: <sup>3</sup> H		2" dia. [] or 4" dia. []
<sup>226</sup> Ra  I certify this lab	oratory is au	Gases: <sup>85</sup> Kr   thorized to rece	eive the samp	oles
requested.		Signature	·	
Date		Signature		
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 ${\tt SUBJECT:} \quad {\tt Participation} \quad {\tt in} \quad {\tt Intercomparison} \quad {\tt Studies} \quad {\tt Program} \quad$ 



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Studies Program, 1975		
	8. PERFORMING ORGANIZATION REPORT NO.	
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#### 15. SUPPLEMENTARY NOTES

#### 16. ABSTRACT

The U.S. Environmental Protection Agency's intercomparison studies program for laboratories involved in environmental radiation measurements is described. The types of environmental samples distributed, the analysis required for each sample, the distribution schedule, and the statistical analysis and reporting of results are discussed. Instructions and application forms are included for laboratories desiring to participate in the program.

This document is not a research report. It is designed for use by laboratories participating or desiring to participate in this quality assurance program.

7.	KEY WO	RDS AND DOCUMENT ANALYSIS			
1.	DESCRIPTORS	b.IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group		
quality of quantitate radioact	tive analysis		07 05/14 04		
	non statement unlimited , NTIS)	19 SECURITY CLASS (This Report) Unclassified 20. SECURITY CLASS (This page) Unclassified	21. NO. OF PAGES 24 22. PRICE		





