EPA-600/4-77-047
October 1977

Environmental Monitoring Series

STATUS AND QUALITY OF RADIATION MEASUREMENTS
Food and Human Urine

Environmental Monitoring and Support Laboratory
Office of Research and Development
U.S. Environmental Protection Agency
Las Vegas, Nevada 89114

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STATUS AND QUALITY OF RADIATION MEASUREMENTS

Food and Human Urine

by

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FOREWORD

Man and his environment must be protected from the adverse effects of pesticides, radiation, noise, and other forms of pollution, and the unwise management of solid waste. Efforts to protect the environment require a focus that recognizes the interplay between the components of our physical environment - air, water, and land. The Environmental Monitoring and Support Laboratory-Las Vegas contributes to this multidisciplinary focus through programs engaged in

- developing and optimizing systems and strategies for monitoring pollutants and their impact on the environment, and
- demonstrating new monitoring systems and technologies by applying them to fulfill special monitoring needs of the Agency's operating programs.

This summary report, "Status and Quality of Radiation Measurements-Food and Human Urine," should be useful in evaluating the quality of environmental radiation data. The data contained in this report should be of value to the EPA, other Federal agencies, State agencies, and private laboratories. For further information on the data contained in this publication contact the Quality Assurance Branch, Environmental Monitoring and Support Laboratory, Las Vegas, Nevada.

George B. Morgan
Director
Environmental Monitoring and Support Laboratory
Las Vegas

ABSTRACT

As part of the radiation quality assurance program conducted by the U.S. Environmental Protection Agency, calibrated radionuclide solutions are distributed to participating laboratories for instrument calibration and yield determinations. Laboratory performance studies involving the analysis of radionuclides in environmental media are also conducted.

A summary is given of the results for the food and human urine cross-check programs for 1972-1975. For tritium, which was the least difficult to analyze, eighty-two percent of the laboratories were within the control limits for accuracy and ninety-nine percent within the control limits for precision over the 3-year period. For strontium-89, the most difficult to analyze, thirty-three percent were within the accuracy control limits and seventy-seven percent within the precision control limits.

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INTRODUCTION

Environmental radiation measurements are made by international, Federal, State, local, and private agencies. The data obtained from these measurements are utilized by the U.S. Environmental Protection Agency (EPA) and other agencies for such purposes as estimating dose and health effects, establishing standards and guides, and conducting regulatory activities. It is therefore imperative that the precision and accuracy of radioassay procedures be assured so that policy decisions concerning environmental quality are based on valid and comparable data.

The radiation quality assurance program of the EPA is designed to encourage the development and implementation of quality control procedures at all levels of sample collection and analysis. As an integral part of the EPA's program, the Quality Assurance Branch of the Environmental Monitoring and Support Laboratory-Las Vegas (EMSL-LV) distributes calibrated radionuclide solutions for instrument calibration and chemical yield determinations, develops and tests analytical procedures for possible use as EPA-approved laboratory methods, and conducts a number of laboratory performance studies involving the analysis of radionuclides in environmental media.

The laboratory intercomparison studies program (performance studies) enables participating laboratories to maintain checks on their analyses and assists them in documenting the validity of their data. In addition, this program enables EPA to obtain an overall estimate of the precision and accuracy of currently implemented environmental radioassay procedures and the precision and accuracy of currently used laboratory procedures for environmental samples.

Performance programs currently in progress involve samples of a variety of environmental material and include milk, air, water, soil, diet, urine, and noble gases. Table 1 is a summary of these programs. Participants include private nuclear facilities, State, Federal, and international laboratories. Because of the large and growing number of participants and the continuing nature of the programs, sufficient data are generated to enable periodic assessment of the quality of environmental data obtained from these laboratories.

Participating laboratories perform analyses on the cross-check samples and return their data to the Quality Assurance Branch for statistical evaluation. Comparisons are made between laboratories and within an individual laboratory for accuracy and precision. A computer report and an updated performance chart are returned to each participant. This enables each laboratory to document the precision and accuracy of its radiation data, to identify instrumental and procedural problems, and to compare their performance with other laboratories.

A preliminary report on the laboratory performance studies conducted for

food and human urine is presented on the following pages.

METHODS AND PROCEDURES

Food and human urine samples containing known amounts of specific radionuclides were prepared and distributed to a number of Federal, State, and private laboratories. These samples were designed to test the ability of participating laboratories to analyze human urine for tritium, and food for strontium-90, strontium-89, iodine-131, cesium-137, barium-140, and potassium. A distribution schedule for the food and urine samples is shown below.

YEAR	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP	OCT	NOV	DEC
1972	-	-	-	-	-	-	-	-	-	-	-	O
1973	O	O	-	O	O	O	O	-	O	-	-	O
1974	-	-	O	-	X	O	-	X	O	-	-	⊠
1975	-	-	O	X	-	O	-	X	O	-	-	⊠

Food: X Urine: O None: - Both: ⊠

The quantity and activity levels of each type of sample are described in the following paragraphs.

FOOD SAMPLES

Three 4-liter simulated food samples containing known amounts of radionuclides were sent to each participant. The samples were formulated to include the dietary intake of the Standard Adult Man.* A chart depicting these various radionuclides and the concentration ranges is shown below.

ISOTOPE	CONCENTRATION RANGE
^{90}Sr	60 - 198 pCi/kg
^{89}Sr	0 - 204 pCi/kg
^{131}I	0 - 216 pCi/kg
^{137}Cs	65 - 205 pCi/kg
^{140}Ba	0 - 207 pCi/kg
K	2216 - 2619 mg/kg

*See "Radiological Health Handbook" compiled and edited by the Bureau of Radiological Health and the Training Institute Environmental Control Administration, U.S. Department of Health, Education and Welfare, January 1970, p. 216.

A 4-liter food sample is composed of the following ingredients.

Soya flour	70.3 g/4 liter
Instant potatoes	563.0 g/4 liter
Milk powder	76.0 g/4 liter
White flour	113.0 g/4 liter
French dressing	225.0 g/4 liter
Formalin	28.2 g/4 liter
Distilled water (aged 30 days)	3208.7 g/4 liter

The first four ingredients are dry-mixed in a 40-quart blender. The dry mixture is then combined with distilled water in the blender using a proportion of two parts water to one part dry mix. The liquid mixture is transferred to a 100-gallon plastic tank, which is resting on a 1000 pound capacity portable platform scale, and stirred with a large commercial electric mixer. After all the dry ingredients have been transferred, the French dressing and the formalin are added directly to the mixture. Additional distilled water is added until final sample weight is reached. The sample is stirred for at least 3 hours and aliquots are removed and counted for background determination.

Upon completion of this initial mixing, the sample has a density of 1.071 grams/milliliter and will yield approximately 4 grams of ash per kilogram of food sample. Accurately measured amounts of the desired radionuclides are added to the food sample, and it is then stirred for approximately 17 hours. Three aliquots are analyzed for radioactivity, providing a check on homogeneity and accuracy before the food is transferred into 4-liter cubitainers for distribution to the participating laboratories.

URINE SAMPLES

A 50-milliliter human urine sample, containing a known amount of tritium, and a 50-milliliter background human urine sample were sent to each participant. Seventeen different samples were distributed between December 1972 and December 1975. The concentrations ranged from 969 (June 1974) to 3432 (July 1973) pCi/liter.

Composite human urine is collected and utilized in the preparation of the tritium in urine samples. The total urine sample is preserved with 7.5 ml of formaldehyde solution per liter of urine and then divided into two parts. One half is used in the preparation of the 50-ml background samples, while a precalculated amount of tritium is added to the other half. The portion containing the tritium is thoroughly mixed and then transferred to 60-milliliter glass bottles for distribution. Before shipping to participants, random samples are analyzed and the batch checked for homogeneity.

ANALYSIS BY PARTICIPANTS

Participating laboratories are instructed to conduct three independent determinations for each radionuclide included in the particular cross-check sample, and report the results to the Quality Assurance Branch. Control limits previously established by the Analytical Quality Control Service*, are used in analyzing the quality of the results obtained by these laboratories. These limits are based on the purpose for which the data are being obtained and on reasonable laboratory ability. Upon receipt of the reports from all participating laboratories, the data are analyzed using a computer. For each radionuclide, this analysis includes determination of the following parameters: the experimental average and standard deviation for each laboratory, the normalized range, the normalized deviation from the known and grand average, and the experimental sigma and the grand average of all laboratories. Examples of sample calculations to illustrate the computations performed by the computer are shown in the Appendix.

A report is generated containing the data reported by all participating laboratories, listed according to identity code, along with the results of the data analysis. Examples are shown in Figure 1. In addition, a control chart is generated for each radionuclide included in the sample (Figure 2). The control charts are updated each time a laboratory participates in a cross-check study, thus giving each laboratory a continuous record of its performance. A copy of the computer printout and a control chart for each radionuclide are mailed to each participant approximately 6 weeks following the report due date.

RESULTS AND DISCUSSION

A laboratory is considered accurate, for our purposes, if its normalized deviation (known) is within ±3. A laboratory is considered precise if its range analysis (R + SR) is less than or equal to 4.

The results of the radionuclides in food intercomparison studies are summarized in Table 2 and Figures 3 to 8.

STRONTIUM-90 (Figure 3)

All six studies contained a strontium-90 spike. Forty-seven percent of the laboratories reported accurate results and ninety-two percent reported precise results during the 2-year period. For April and August of 1975 the known and grand average values were equal or almost equal. No strontium-89 was added to these samples. In three of the other four studies where strontium-89 was added, the grand average was less than the known value. This may

*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, November 1964.

be an indication that strontium-89 activity in the sample may cause the analytical results to be biased low.

STRONTIUM-89 (Figure 4)

Four of the six studies included this isotope. Thirty-three percent of the laboratories reported accurate results and seventy-seven percent met the precision requirements. In all four studies the laboratories' grand average values were lower than the known values, possibly indicating a negative bias for this analysis.

IODINE-131 (Figure 5)

This isotope was present in five of the six samples used in this study. Fifty-nine percent of the laboratories reported results within the accuracy limits, and eighty-seven percent of the laboratories reported replicate results with a precision meeting the requirements of the study. A comparison of the known and grand average values shows no indication of bias in the analysis.

CESIUM-137 (Figure 6)

Cesium-137 was present in all six samples. Seventy-one percent of the laboratories reported data within the acceptable accuracy limits while ninety-seven percent of the laboratories had acceptable precision in their measurements. No bias was evident from the reported results. Some improvement can be seen in the 1975 data over the 1974 data where the grand average values fell closer to the known value.

BARIUM-140 (Figure 7)

Only two of six samples contained this isotope. Fifty-three percent of the laboratories reported results within the control limits, and ninety-one percent met the requirements for precision. More studies containing barium-140 must be conducted before any conclusions can be made about bias or other trends in laboratory performance.

POTASSIUM (Figure 8)

Since the potassium concentration occurs naturally in the food sample, all six studies contained potassium. Sixty-eight percent of the laboratories met the requirements for accuracy, and ninety-nine percent met the standards for precision. In all six studies the laboratories' low grand average would imply that the analyses may be biased.

TRITIUM

The results of the radionuclides in human urine studies are summarized in Table 3 and Figure 9. Eighty-two percent of the laboratories reported results within the 3 sigma control limits while ninety-nine percent reported results with acceptable precision. No bias in the reported results was evident over the 3-year period in which the data were collected.

SUMMARY

Table 4 is a summary of the results for the six radionuclides analyzed in the food cross-check samples and the one isotope analyzed in the human urine cross-check sample. Using the percentage of laboratories reporting data within the 3 sigma control limits as criteria, the radionuclides are listed in the order of the ability of the laboratories to perform the radionuclide analysis. The top chart refers to the ability of the laboratories to maintain the required accuracy, while the bottom chart is a measure of the laboratories' ability to meet the precision requirements.

The conclusions drawn, of necessity, have been very general due to the limited amount of available data. The tritium data show that eighty-two percent of the laboratories were within the control limits for accuracy and ninety-nine percent within the control limits for precision over the 3-year period. Thirty-three percent were within the accuracy control limits and seventy-seven percent within the precision control limits for strontium-89.

With the continuation of these studies, additional data will be collected and compiled. When more data become available, such parameters as control limits, methods of analysis, and instrument calibration must be critically assessed in determining laboratory performance and improving it when necessary.

EMSL-LV TRITIUM IN URINE CROSS-CHECK PROGRAM---SEPTEMBER 1974

09/20/74 SAMPLE - A 3H

KNOWN VALUE = 3273 pCi/1

EXPECTED LABORATORY PRECISION (1S, 1 DETERMINATION) = 357 pCi/1

LAB	RESULT	EXPERIMENTAL SIGMA	RNG ANLY ($\overline{R}$ + SR)	AVERAGE	NORMALIZED DEVIATION (GRAND AVG)	(KNOWN)
AN	NO DATA PROVIDED					
CF	3269					
CF	3522					
CF	3632	186.1	0.60	3474	0.9	1.0
CM	3261					
CM	3373					
CM	3362	61.7	0.19	3332	0.2	0.3
CO	NO DATA PROVIDED					
D	3060					
D	3060					
D	3240	103.9	0.30	3120	-0.8	-0.7
J	3255					
J	3247					
J	3294	25.1	0.08	3265	-0.1	-0.0
P	NO DATA PROVIDED					
Z	3240					
Z	3340					
Z	3190	76.4	0.25	3257	-0.2	-0.1

EXPERIMENTAL SIGMA (ALL LABS) = 149 GRAND AVERAGE = 3290

Figure 1. Computer performance report.

Explanation of terms in Figure 1:

Title:	Program name, sample collection date, sample code letter, analysis type, known concentration of radionuclide, 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.
Column 4:	Normalized range value in "mean range + standard error 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 5:	Average value.
Column 6:	Normalized deviation from the grand average value of all laboratories expressed in σ_M units.
Column 7:	Normalized deviation from the known value expressed in σ_M units.
Bottom of Chart:	1s experimental error of all laboratories, and the grand average of all laboratories.

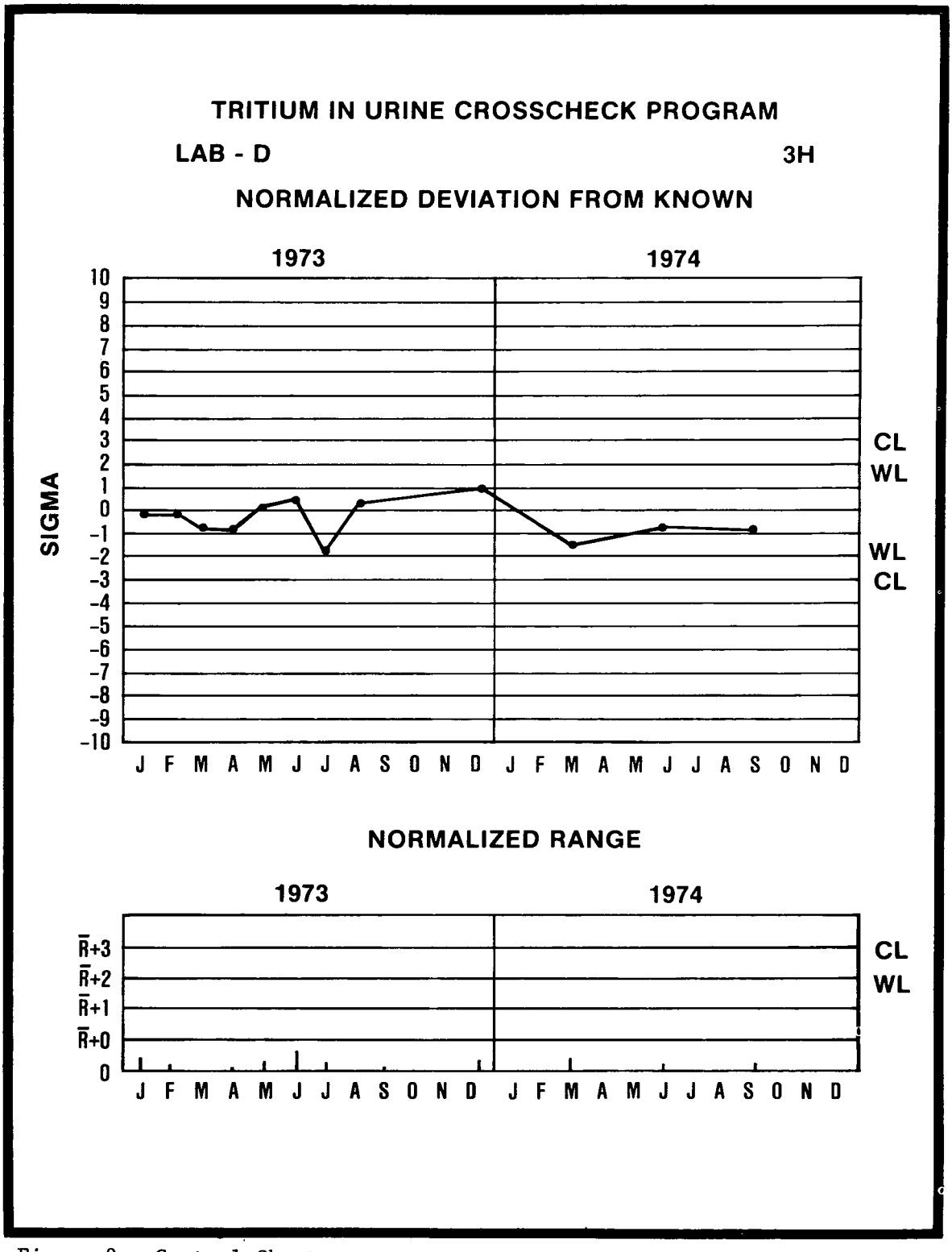

Figure 2. Control Chart.

Explanations 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% ($\mu \pm 2\sigma_M$) and the 99.7% ($\mu \pm 3\sigma_M$) 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 respectively.]

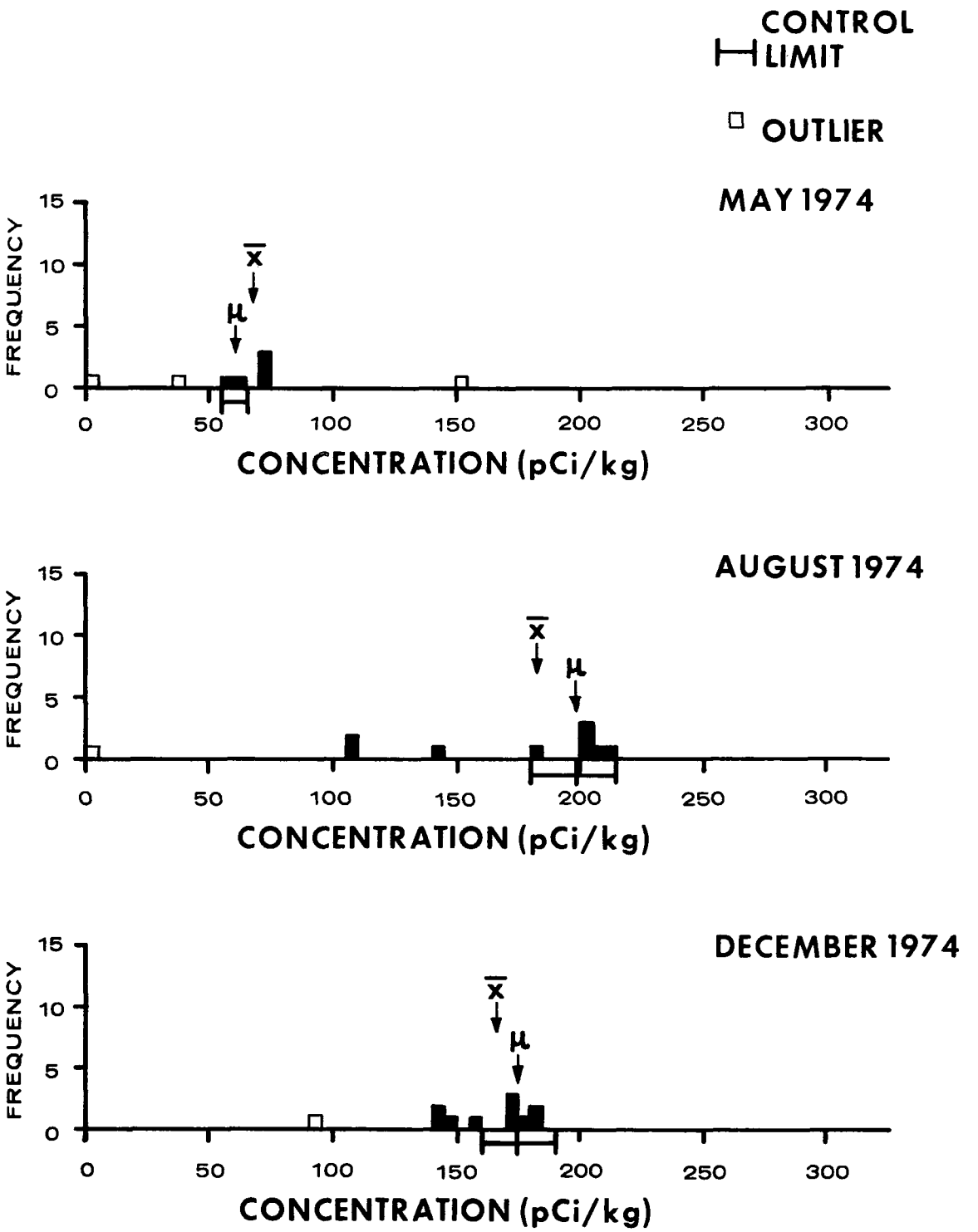

Figure 3. Histogram of laboratory averages reported for strontium-90 in food.

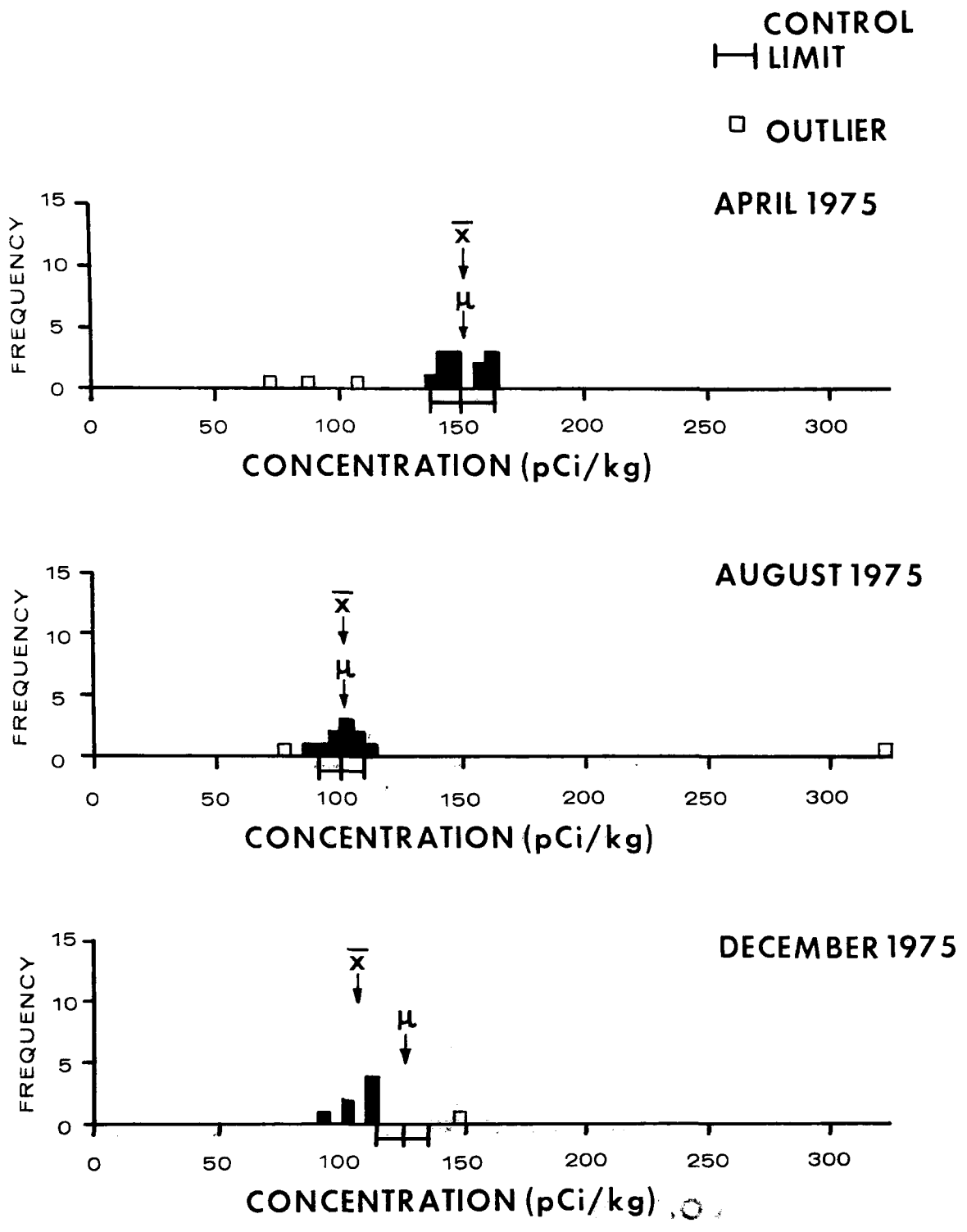

Figure 3 (continued). Histogram of laboratory averages reported for strontium-90 in food.

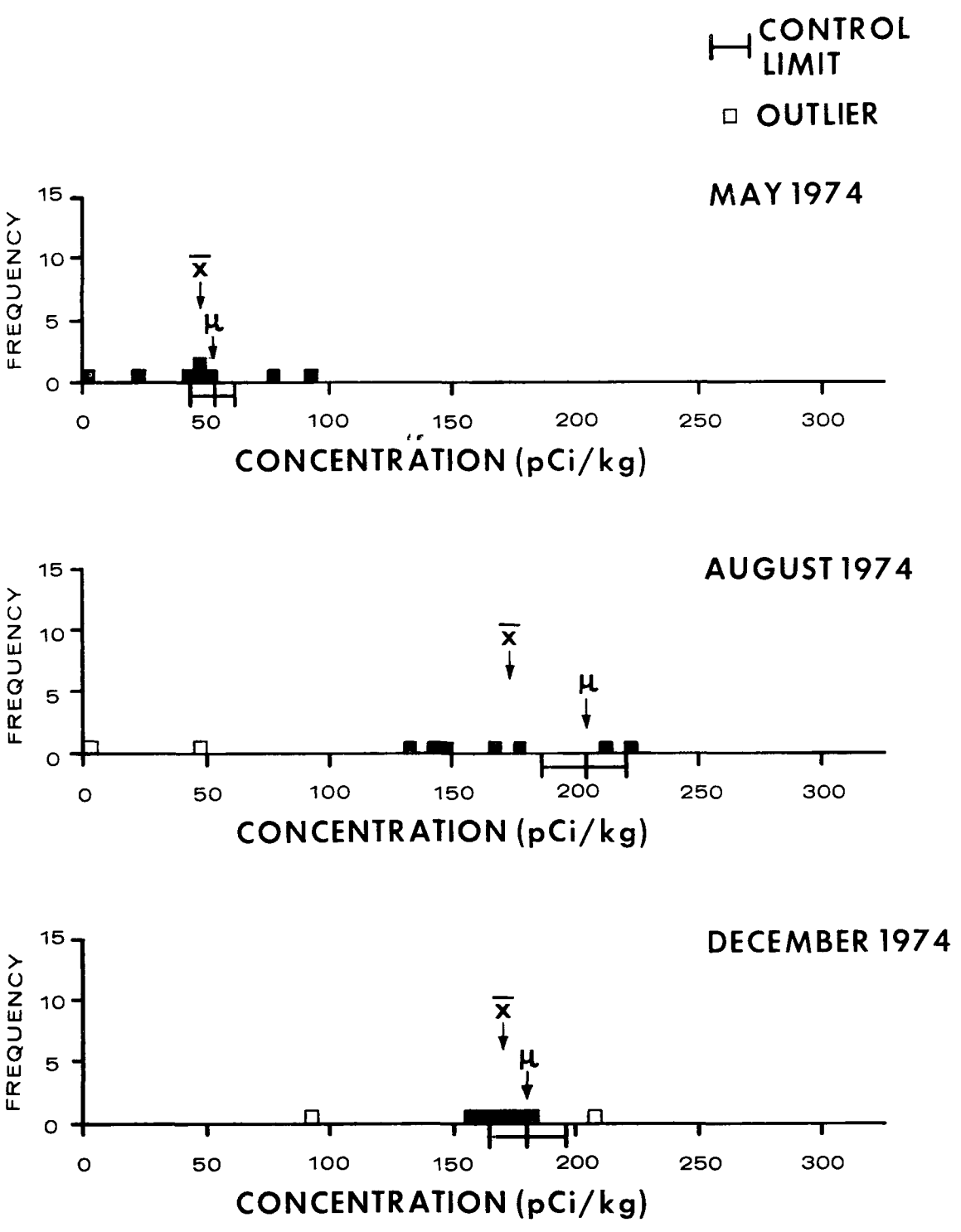

Figure 4. Histogram of laboratory averages reported for strontium-89 in food.

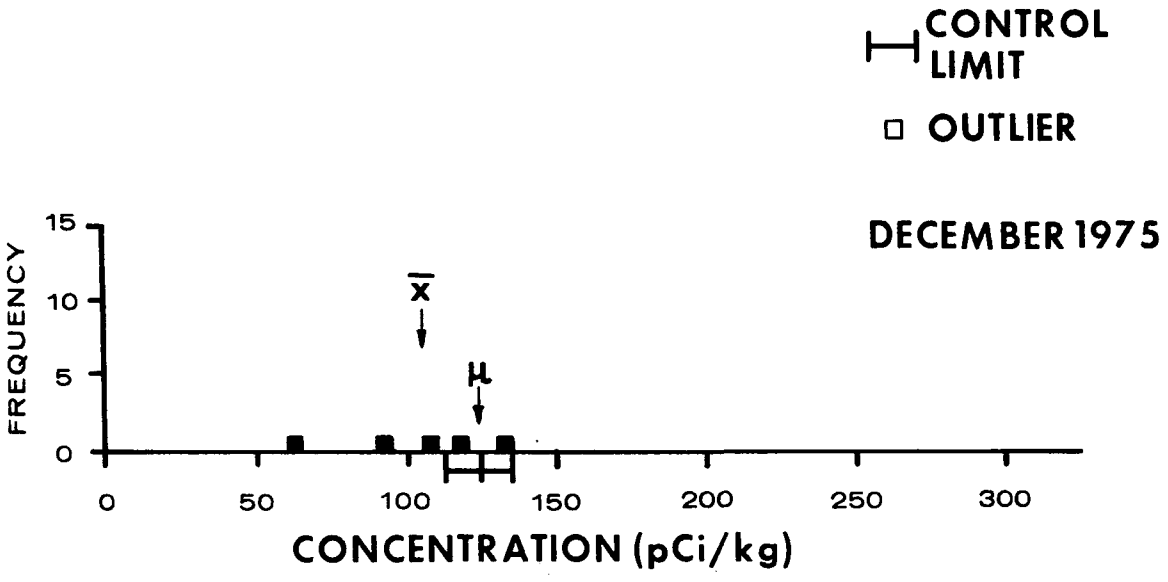

Figure 4 (continued). Histogram of laboratory averages reported for strontium-89 in food.

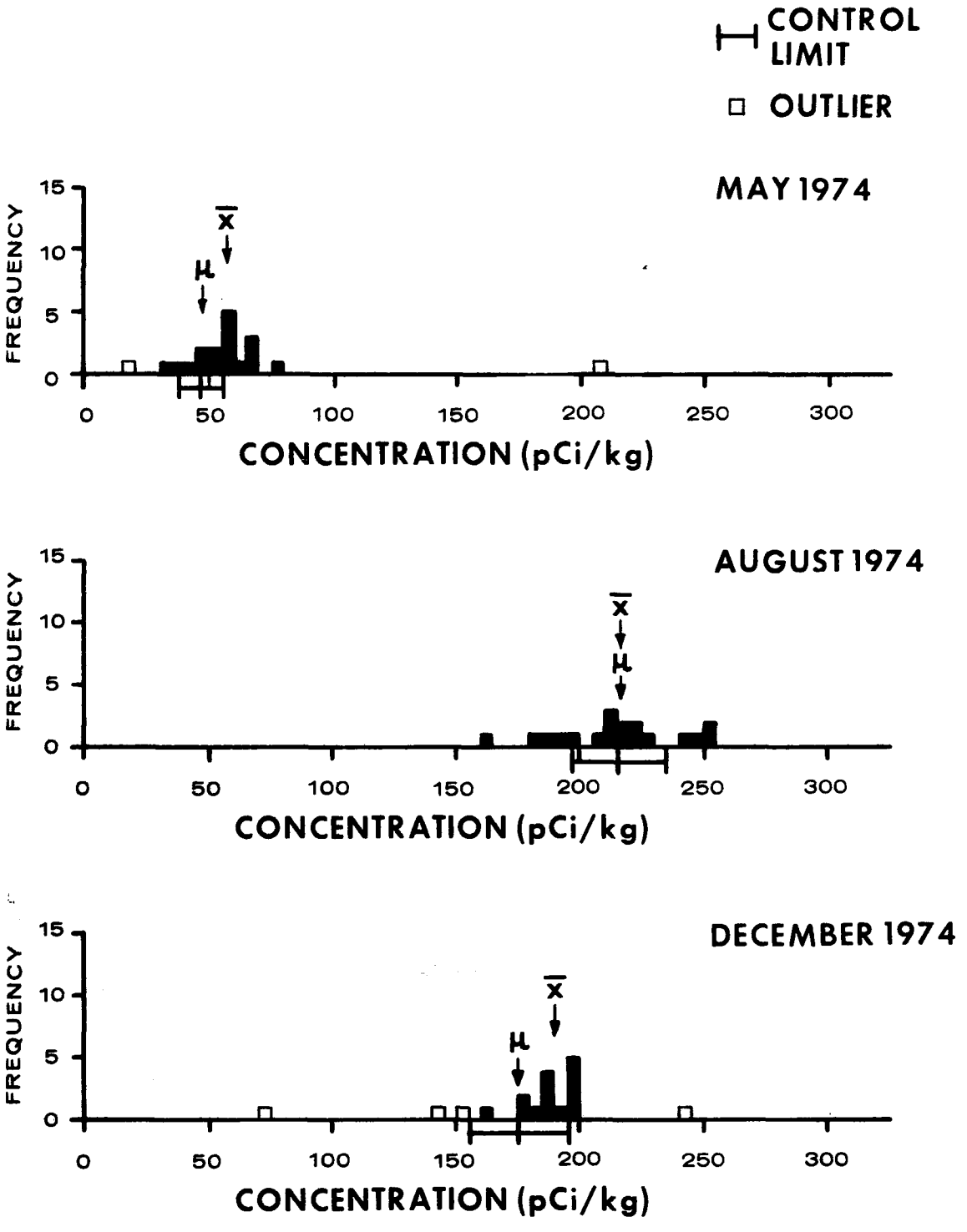

Figure 5. Histogram of laboratory averages reported for iodine-131 in food.

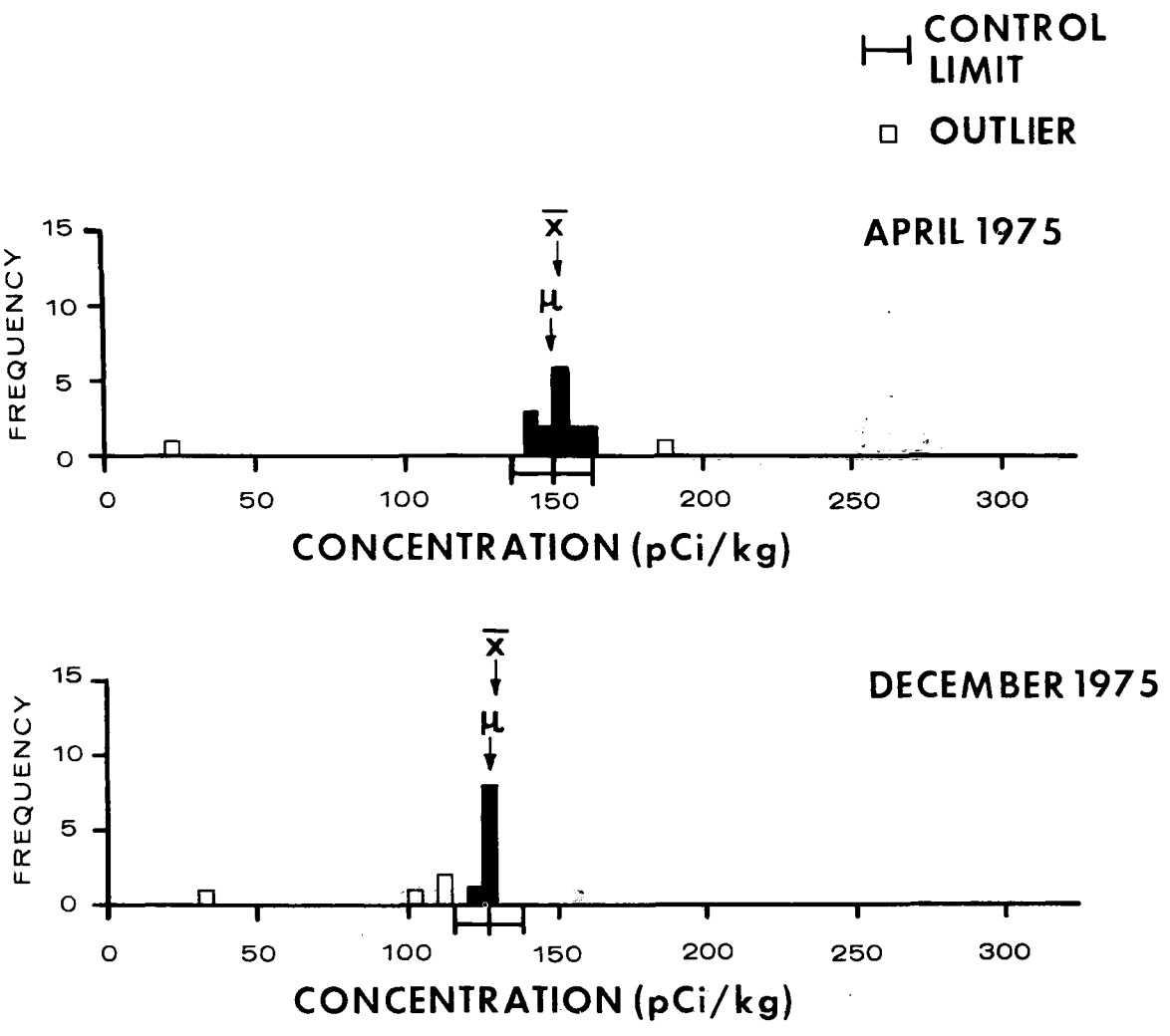

Figure 5 (continued). Histogram of laboratory averages reported for iodine-131 in food.

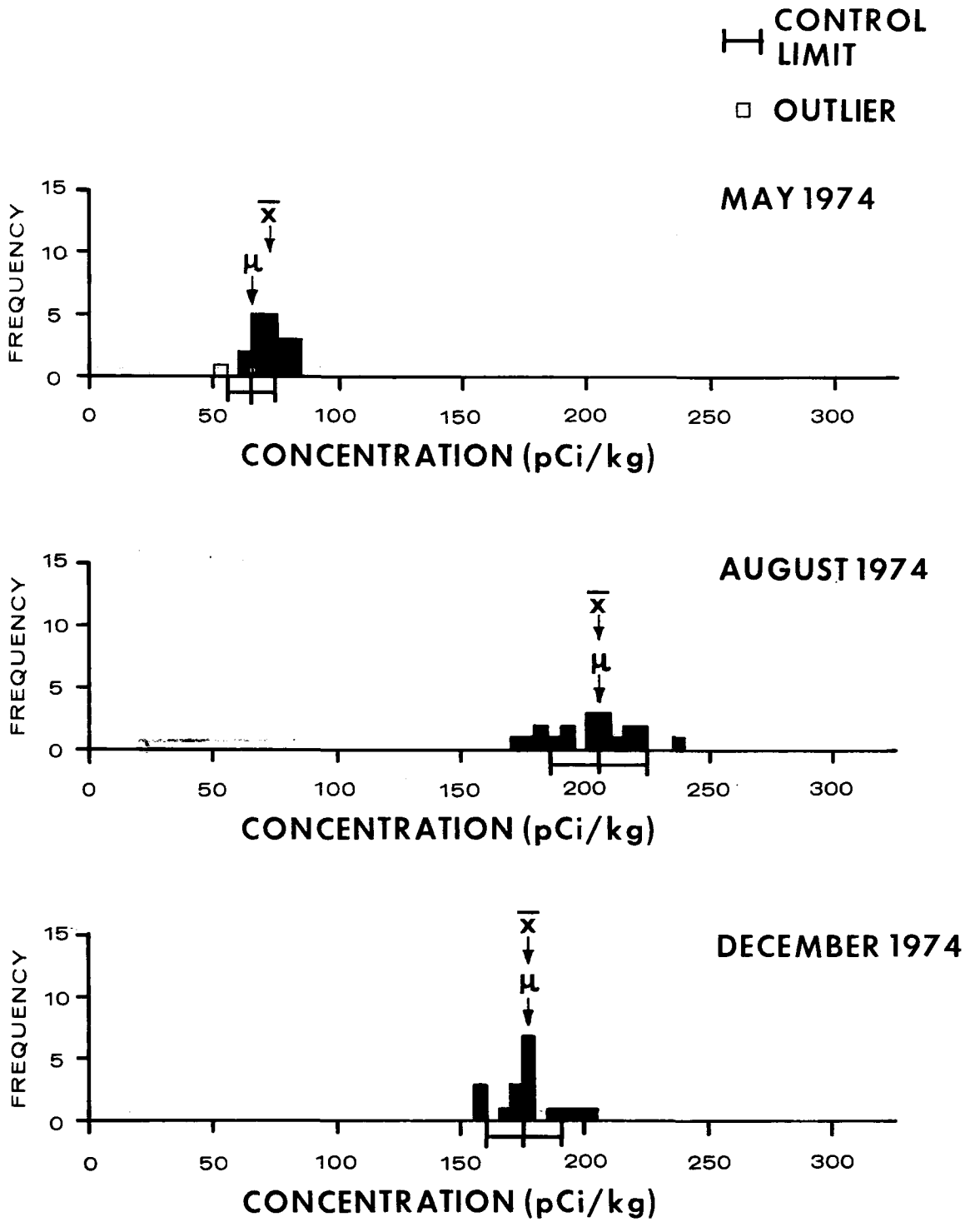

Figure 6. Histogram of laboratory averages reported for cesium-137 in food.

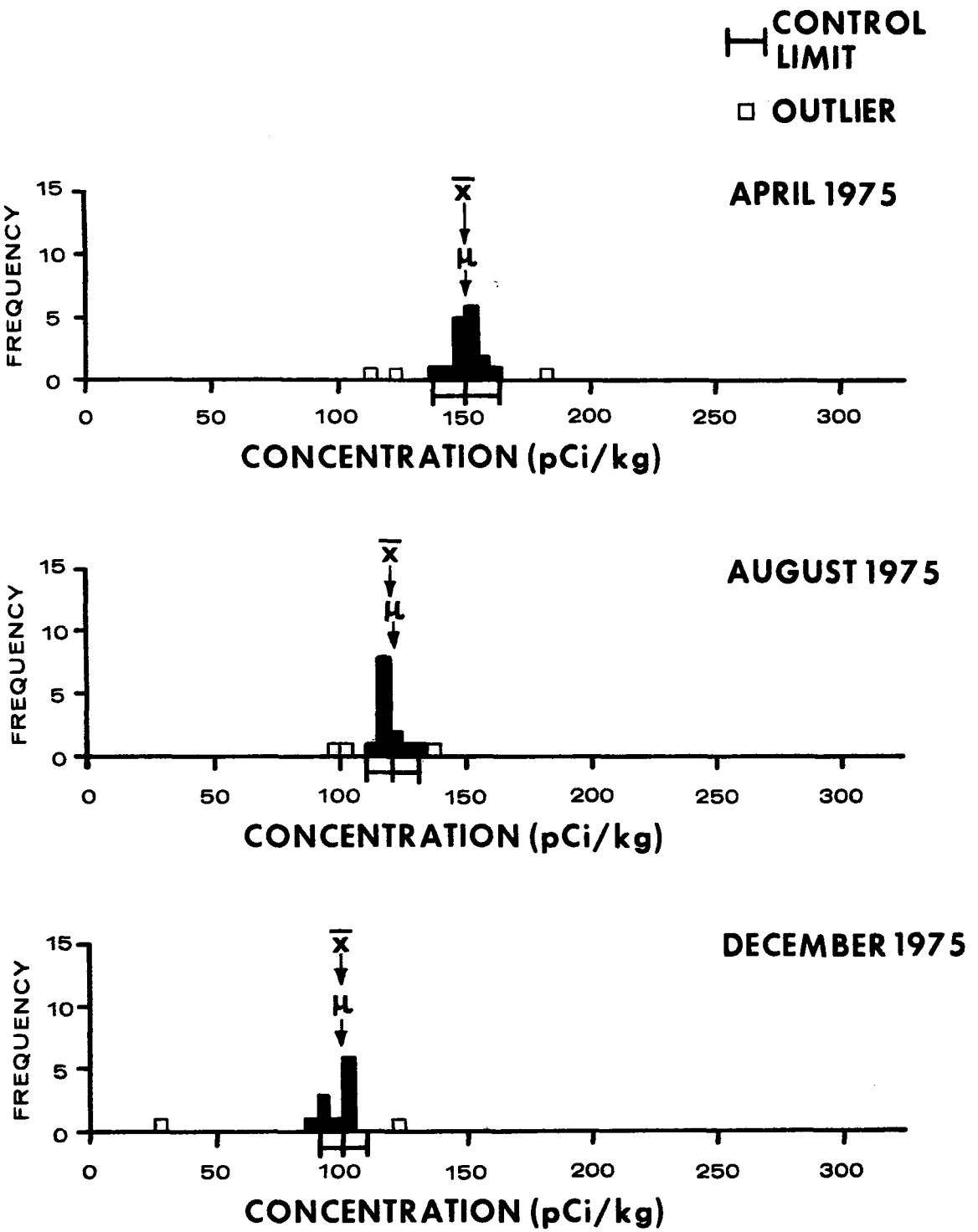

Figure 6 (continued). Histogram of laboratory averages reported for cesium-137 in food.

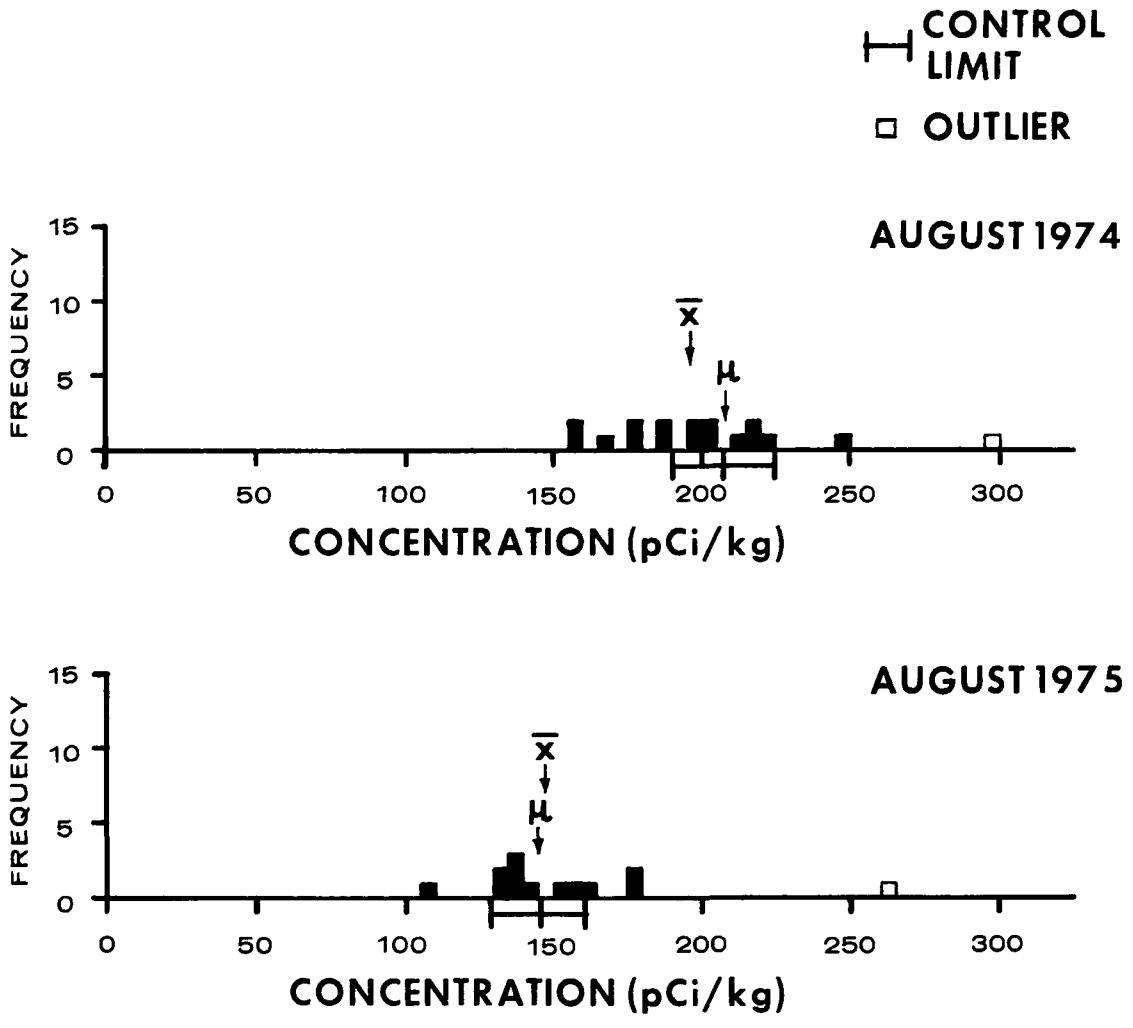

Figure 7. Histogram of laboratory averages reported for barium-140 in food.

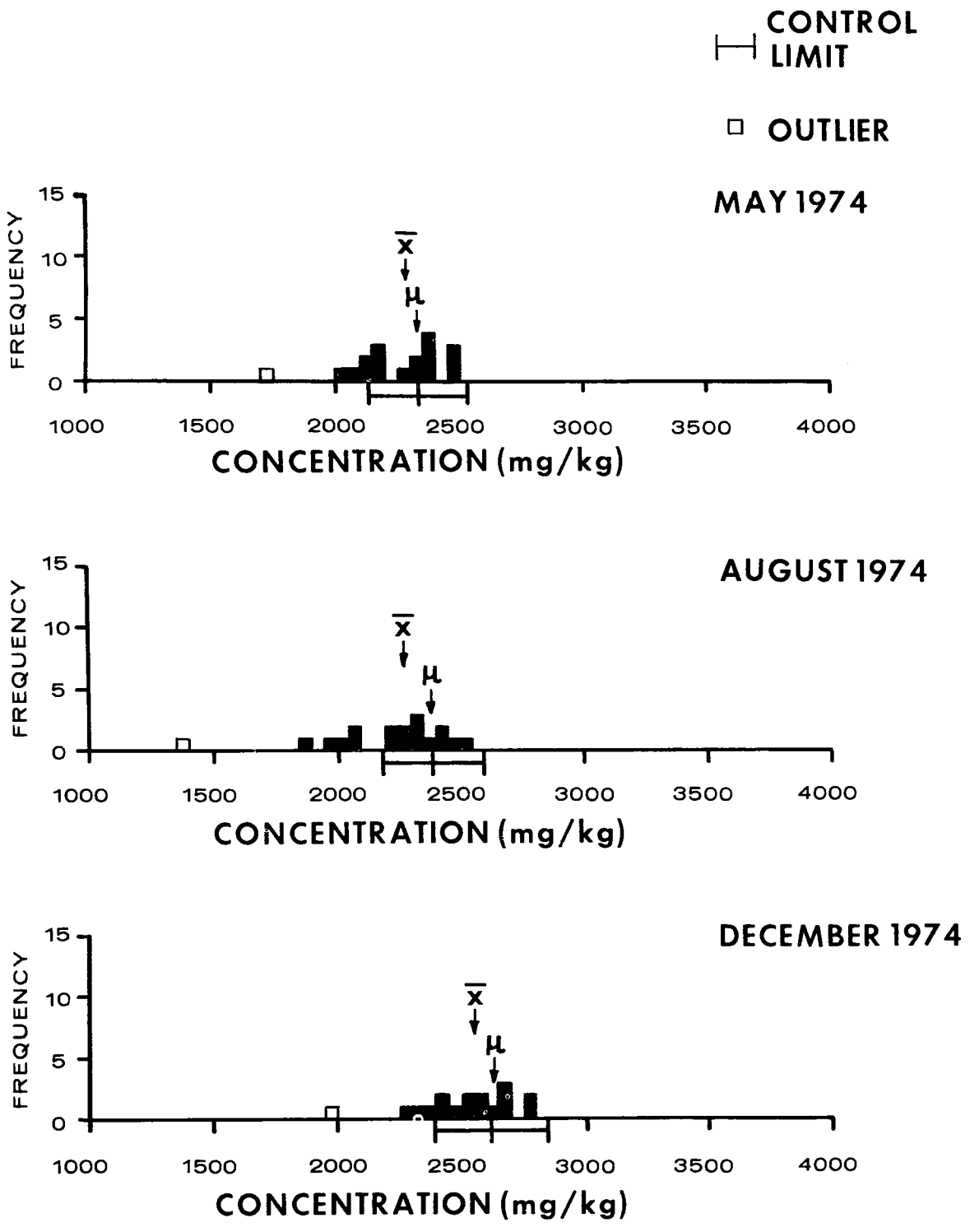

Figure 8. Histogram of laboratory averages reported for potassium in food.

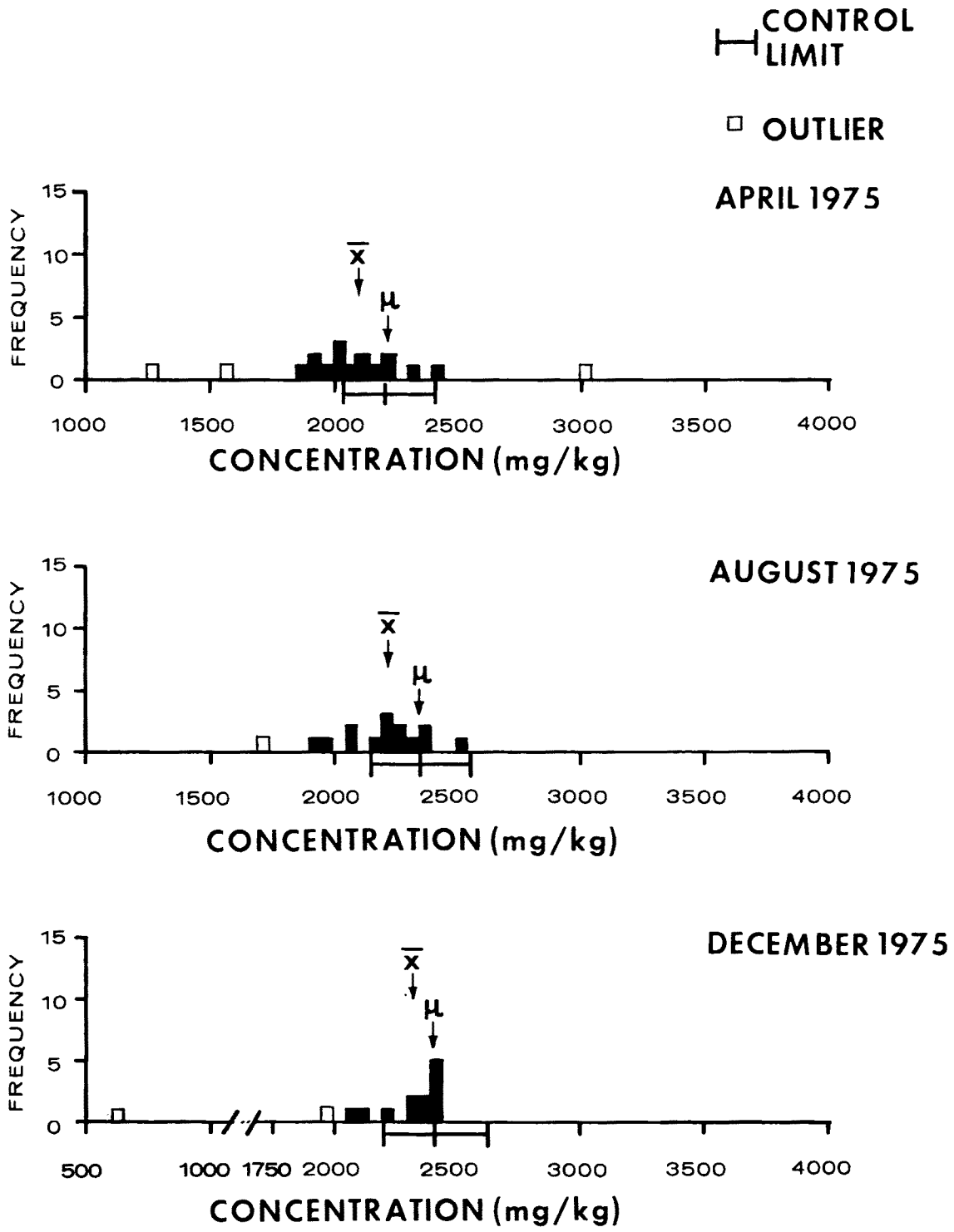

Figure 8 (continued). Histogram of laboratory averages reported for potassium in food.

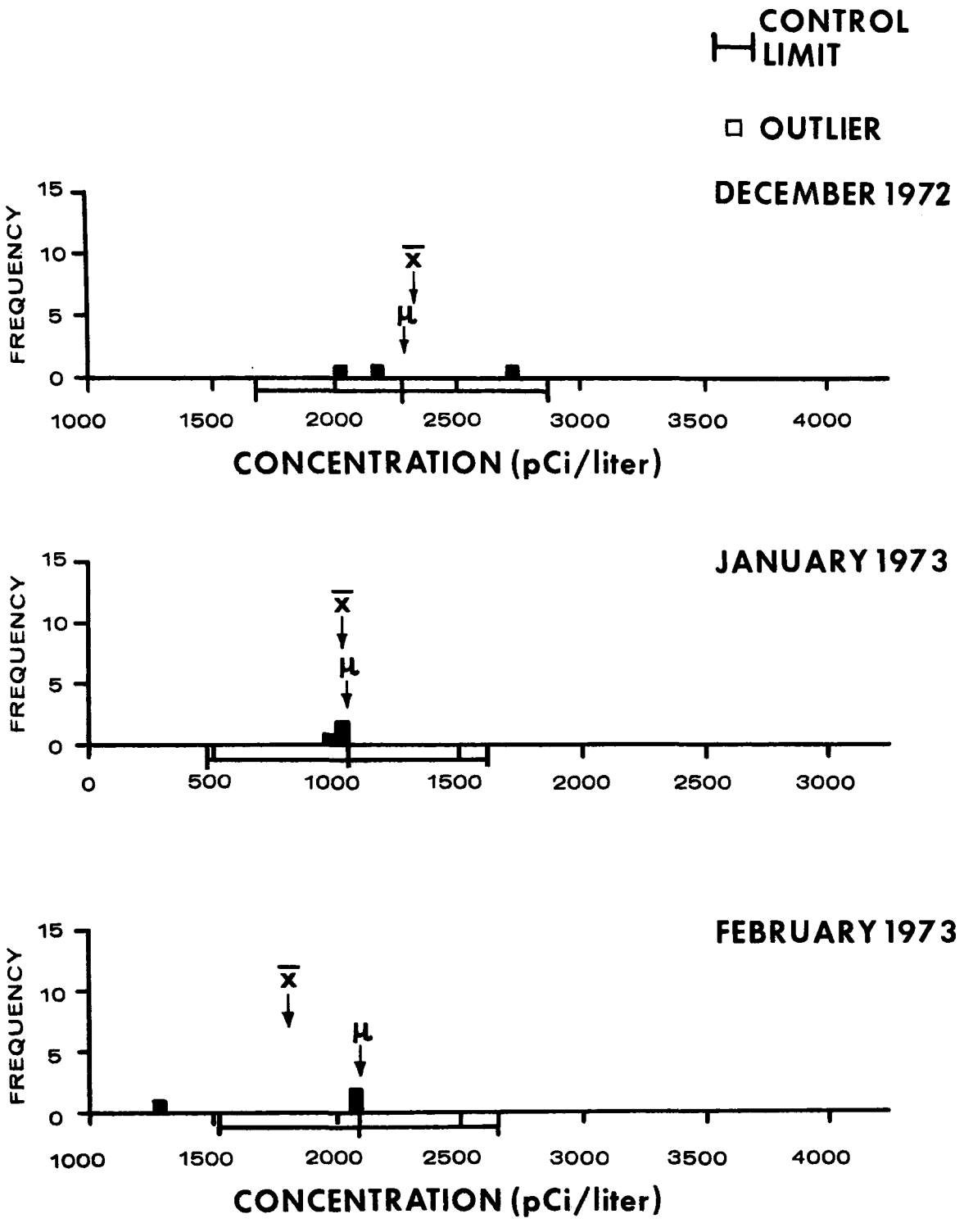

Figure 9. Histogram of laboratory averages reported for tritium in urine.

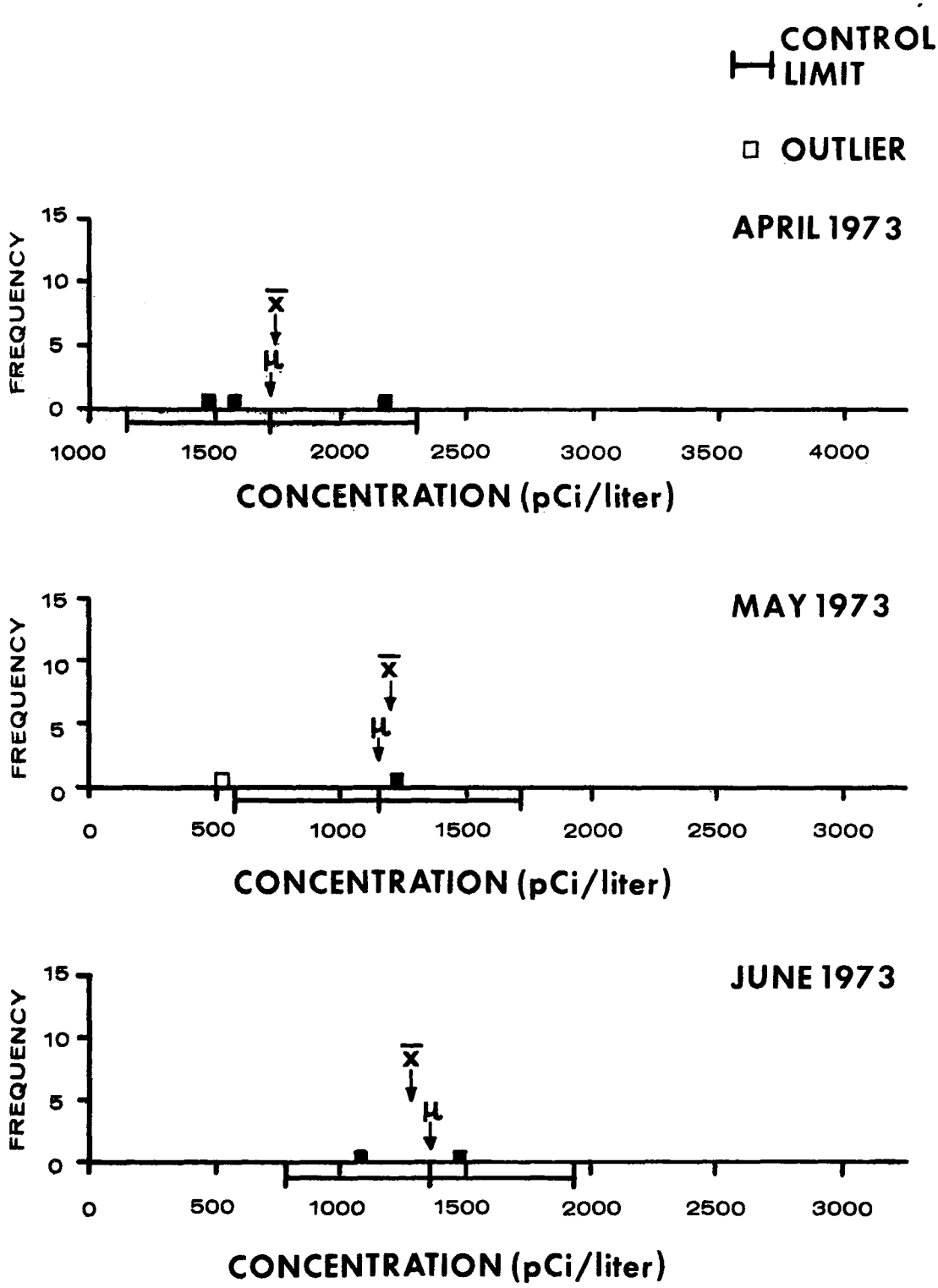

Figure 9 (continued). Histogram of laboratory averages reported for tritium in urine.

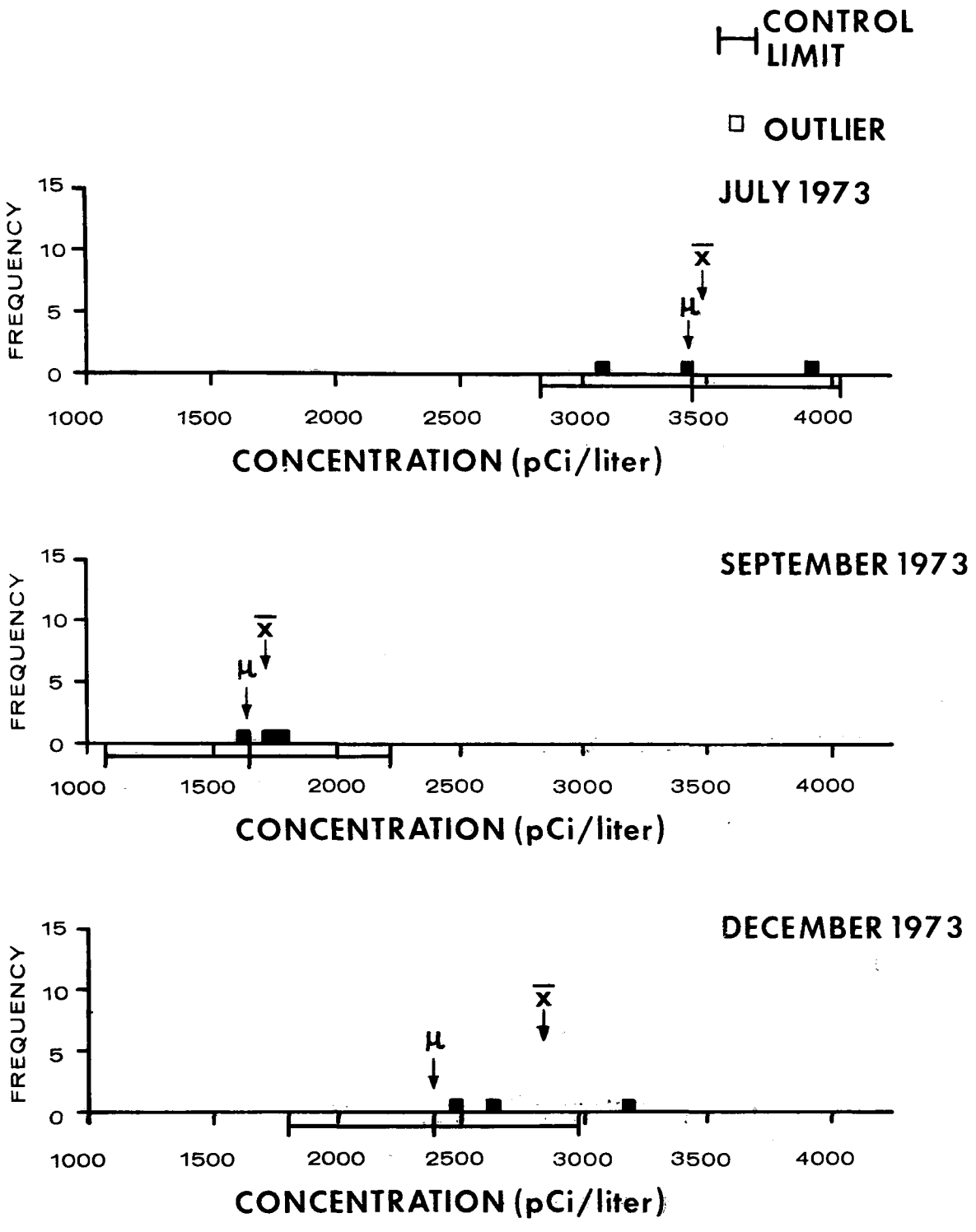

Figure 9 (continued). Histogram of laboratory averages reported for tritium in urine.

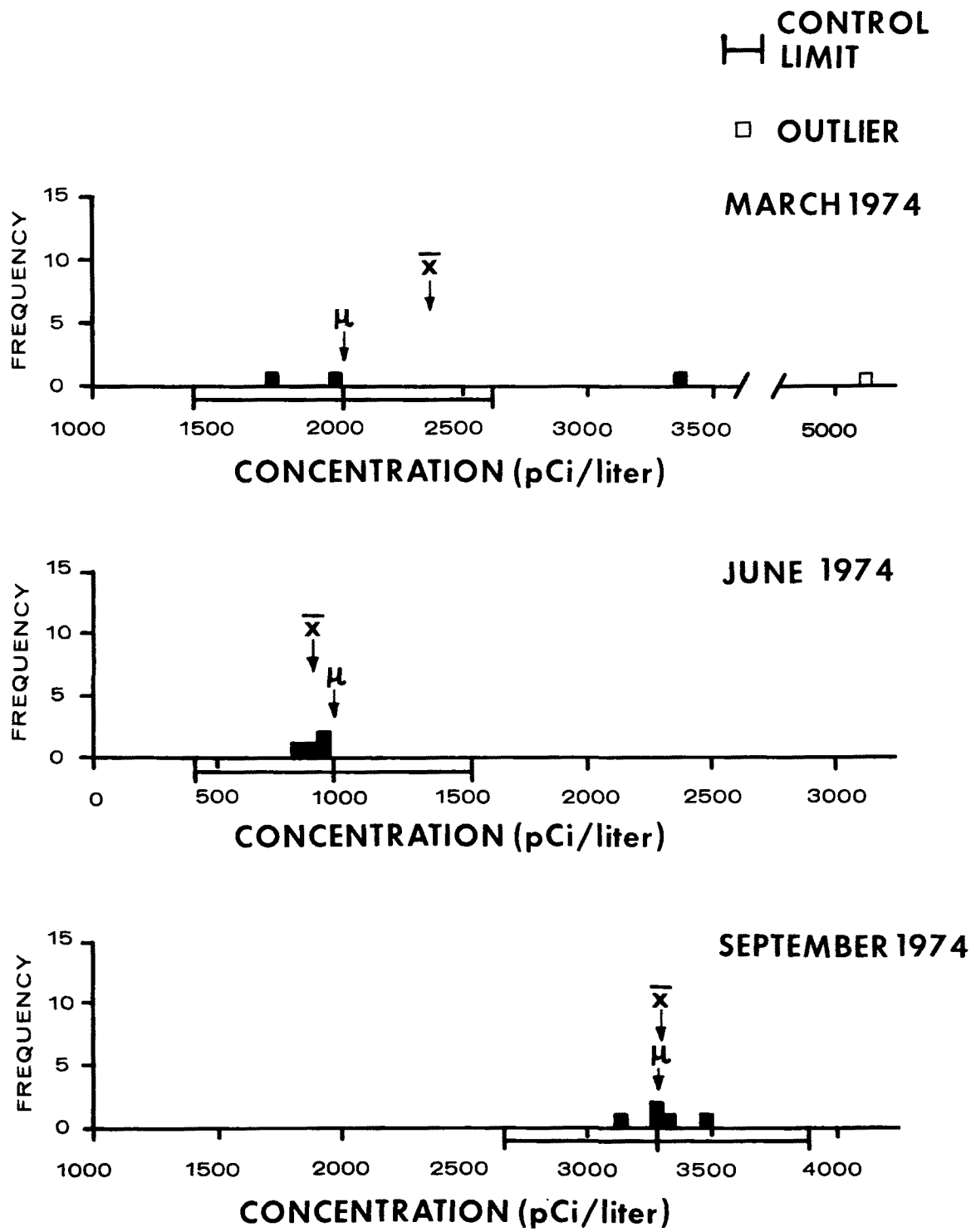

Figure 9 (continued). Histogram of laboratory averages reported for tritium in urine.

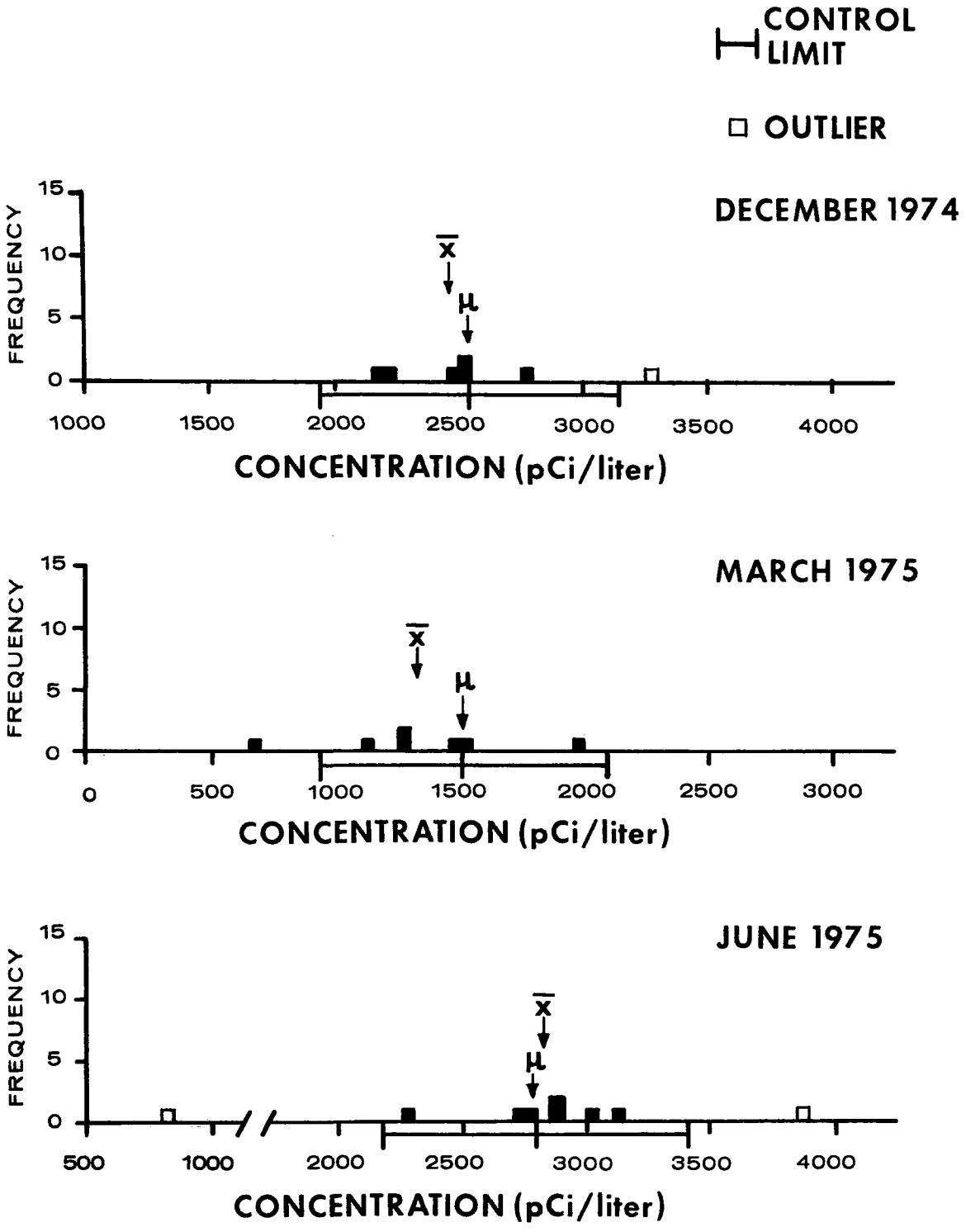

Figure 9 (continued). Histogram of laboratory averages reported for tritium in urine.

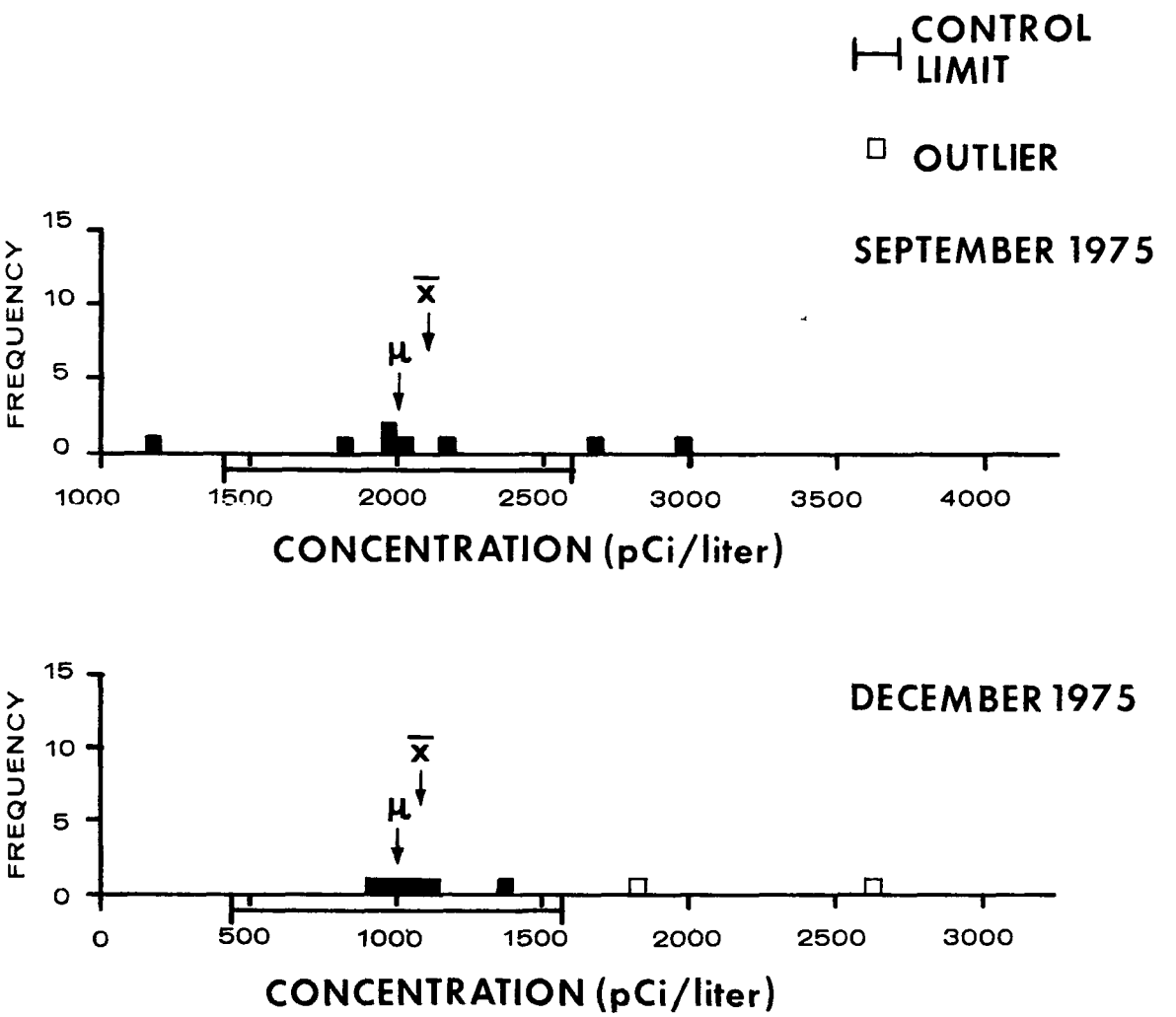

Figure 9 (continued). Histogram of laboratory averages reported for tritium in urine.

TABLE 1. SUMMARY OF CROSS-CHECK PROGRAMS*

SAMPLE	ANALYSIS	ACTIVITY PER ISOTOPE	QUANTITY SUPPLIED	PRESERVATIVE	DISTRIBUTION	TIME FOR ANALYSIS & REPORT
Milk	^{89}Sr, ^{90}Sr, ^{131}I, ^{137}Cs, ^{140}Ba, K	< 200 pCi/liter	∿ 4 liters	Formalin	Bimonthly	6 weeks
Water						
Gross α, β*	Gross α, β	< 100 pCi/liter	∿ 4 liters	0.5N HNO_3	Bimonthly	4 weeks
Gamma	^{60}Co, ^{106}Ru, ^{134}Cs, ^{137}Cs, ^{51}Cr, ^{65}Zn	< 500 pCi/liter	∿ 4 liters	0.5N HNO_3	Bimonthly	4 weeks
^{3}H	^{3}H	< 3500 pCi/liter	∿ 50 ml	none	Bimonthly	4 weeks
^{239}Pu*	^{239}Pu	< 10 pCi/liter	∿ 4 liters	0.5N HNO_3	Semiannually	8 weeks
Radium	^{226}Ra, ^{228}Ra	< 20 pCi/liter	∿ 4 liters	0.5N HNO_3	Quarterly	6 weeks
Air						
Gross α, β*	α, β, ^{90}Sr, ^{137}Cs	< 200 pCi/sample	3 - 2" or 4" diam. air filters	none	Quarterly	6 weeks
^{239}Pu*	^{239}Pu	< 2 pCi/sample	3 - 2" or 4" diam. air filters	none	Quarterly	6 weeks
Soil*	^{238}Pu, ^{239}Pu ^{228}Th, ^{230}Th, ^{232}Th	< 50 pCi/sample	∿ 35 grams	none	Semiannually	8 weeks
Diet	^{89}Sr, ^{90}Sr, ^{131}I, ^{137}Cs, ^{140}Ba, K	< 200 pCi/kg	3 - 4-liter samples	Formalin	Quarterly	8 weeks
Urine	^{3}H	< 3500 pCi/liter	∿ 50 ml	Formalin	Quarterly	4 weeks
Gas	^{85}Kr	< 20 pCi/ml	10 liters	none	Quarterly	6 weeks

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

TABLE 2. SUMMARY OF FOOD ANALYSIS DATA

May 1974						
	^{90}Sr	^{89}Sr	^{131}I	^{137}Cs	^{140}Ba	K
N	5	8	17	18	-	17
μ (pCi/kg)	60	53	47	65	0	2330 mg/kg
$\bar{x}$ (pCi/kg)	67	48	56	72	-	2291 mg/kg
σ (pCi/kg)	3	5	5	5	-	117 mg/kg
s (pCi/kg)	8	28	17	7	-	151 mg/kg
(σ/μ) 100 (%)	5	9	11	8	-	5
(s/μ) 100 (%)	13	53	36	11	-	6

August 1974						
	^{90}Sr	^{89}Sr	^{131}I	^{137}Cs	^{140}Ba	K
N	9	7	18	19	16	17
μ (pCi/kg)	198	204	216	205	207	2389 mg/kg
$\bar{x}$ (pCi/kg)	183	174	216	205	196	2255 mg/kg
σ (pCi/kg)	9.9	10.2	10.8	10.3	10.3	119 mg/kg
s (pCi/kg)	46	35	27	20	26	207 mg/kg
(σ/μ) 100 (%)	5	5	5	5	5	5
(s/μ) 100 (%)	23	17	13	10	13	9

December 1974						
	^{90}Sr	^{89}Sr	^{131}I	^{137}Cs	^{140}Ba	K
N	10	6	14	18	-	16
μ (pCi/kg)	175	180	175	176	0	2619 mg/kg
$\bar{x}$ (pCi/kg)	166	170	189	177	-	2549 mg/kg
σ (pCi/kg)	8.8	9	8.8	8.8	-	131 mg/kg
s (pCi/kg)	17	18	18	14	-	176 mg/kg
(σ/μ) 100 (%)	5	5	5	5	-	5
(s/μ) 100 (%)	10	10	10	8	-	7

N, number of laboratories; μ, known value; $\bar{x}$, grand average; σ, expected precision; s, standard deviation.

TABLE 2 (Continued).

April 1975						
	^{90}Sr	^{89}Sr	^{131}I	^{137}Cs	^{140}Ba	K
N	12	-	15	16	-	14
μ (pCi/kg)	150	0	149	150	0	2216 mg/kg
$\bar{x}$ (pCi/kg)	151	-	151	151	-	2093 mg/kg
σ (pCi/kg)	7.5	-	7.5	7.5	-	111 mg/kg
s (pCi/kg)	11	-	8	7	-	170 mg/kg
$(\sigma/\mu) \times 100$ (%)	5	-	5	5	-	5
$(s/\mu) \times 100$ (%)	7	-	5	5	-	8

August 1975						
	^{90}Sr	^{89}Sr	^{131}I	^{137}Cs	^{140}Ba	K
N	10	-	-	13	12	14
μ (pCi/kg)	101	0	0	121	145	2352 mg/kg
$\bar{x}$ (pCi/kg)	101	-	-	120	147	2227 mg/kg
σ (pCi/kg)	5.1	-	-	6.1	7.3	118 mg/kg
s (pCi/kg)	8	-	-	6	21	179 mg/kg
$(\sigma/\mu) \times 100$ (%)	5	-	-	5	5	5
$(s/\mu) \times 100$ (%)	8	-	-	5	14	8

December 1975						
	^{90}Sr	^{89}Sr	^{131}I	^{137}Cs	^{140}Ba	K
N	7	5	10	11	-	12
μ (pCi/kg)	125	124	127	101	0	2414 mg/kg
$\bar{x}$ (pCi/kg)	107	104	129	100	-	2330 mg/kg
σ (pCi/kg)	6.3	6.2	6.4	5.1	-	121 mg/kg
s (pCi/kg)	12	25	5	6	-	129 mg/kg
$(\sigma/\mu) \times 100$ (%)	5	5	5	5	-	5
$(s/\mu) \times 100$ (%)	10	20	4	6	-	5

TABLE 3. SUMMARY OF TRITIUM IN HUMAN URINE ANALYSIS DATA

	December 1972	January 1973	February 1973	April 1973	May 1973	June 1973
N	3	3	3	3	1	2
μ (pCi/liter)	2270	1048	2080	1724	1148	1357
$\bar{x}$ (pCi/liter)	2308	1024	1796	1749	1200	1283
σ (pCi/liter)	341	325	333	336	329	332
s (pCi/liter)	333	99	439	422	100	306
(σ/μ) 100 (%)	15	31	16	19	29	24
(s/μ) 100 (%)	15	9	21	24	9	23

	July 1973	September 1973	December 1973	March 1974	June 1974	September 1974
N	3	3	3	3	4	5
μ (pCi/liter)	3432	1641	2391	2012	969	3273
$\bar{x}$ (pCi/liter)	3485	1704	2833	2356	885	3290
σ (pCi/liter)	347	333	340	350	324	357
s (pCi/liter)	379	97	374	785	81	149
(σ/μ) 100 (%)	10	20	14	17	33	11
(s/μ) 100 (%)	11	6	16	39	8	5

	December 1974	March 1975	June 1975	September 1975	December 1975
N	6	7	7	8	6
μ (pCi/liter)	2546	1504	2793	2004	1001
$\bar{x}$ (pCi/liter)	2455	1327	2829	2105	1077
σ (pCi/liter)	349	331	356	345	324
s (pCi/liter)	349	414	330	536	224
(σ/μ) 100 (%)	14	22	13	17	32
(s/μ) 100 (%)	14	28	12	27	22

TABLE 4. SUMMARY OF LABORATORY PERFORMANCE
1972-1975 INTERLABORATORY COMPARISON STUDIES - FOOD AND HUMAN URINE

Radionuclide Analysis	% of Laboratories within ± 3σ (99.7% Control Limits)			
	1972-73	1974	1975	1972-75
Tritium (urine)	88	85	75	82
Cesium-137	-	63	82	71
Potassium	-	75	60	68
Iodine-131	-	47	81	59
Strontium-90	-	41	51	47
Barium-140	-	47	62	53
Strontium-89	-	32	40	33

Radionuclide Analysis	% of Laboratories within $\bar{R} + 3\sigma_R$ (100 % Control Limits)			
	1972-73	1974	1975	1972-75
Tritium (urine)	100	100	97	99
Potassium	-	100	98	99
Cesium-137	-	96	98	97
Strontium-90	-	90	94	92
Barium-140	-	95	85	91
Iodine-131	-	83	94	87
Strontium-89	-	73	100	77

APPENDIX. STATISTICAL CALCULATIONS

To illustrate the computations performed by the computer, an example of range analysis calculations are given using data for only one laboratory (Laboratory D, see Figure 1).

The experimental data are listed and the mean, experimental sigma and range 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, σ_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 which is less than 3, which is the upper warning level. The precision of the results is acceptable.

The normalized deviation is calculated by computing the deviation and the standard error of the mean, σ_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 between +2 and -2, which are 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 Figure 1)

Experimental data:

Known value = μ = 3273 pCi ^{3}H/liter urine on September 24, 1974

Expected laboratory precision = σ = 357 pCi/liter

Laboratory	Sample	Result
D	x_1	3060 pCi/liter
D	x_2	3060 pCi/liter
D	x_3	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 = 3

Experimental sigma = s

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

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

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

Range = r

$$r = |\text{maximum result} - \text{minimum result}|$$

$$= |3240 - 3060| = 180 \text{ pCi/liter}$$

Range Analysis (RNG ANLY)*

Mean range = $\bar{R}$

$$\bar{R} = d_2\sigma \qquad \text{where } d_2^* = 1.693 \text{ for N = 3}$$

$$= (1.693)(357)$$

$$= 604.4 \text{ pCi/liter}$$

* 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.

Control limit = CL

$$CL = \bar{R} + 3\sigma_R$$

$$= D_4\bar{R} \qquad \text{where } D_4{}^* = 2.575 \text{ for } N = 3$$

$$= (2.575)(604.4)$$

$$= 1556 \text{ pCi/liter}$$

Standard error of the range = σ_R

$$\sigma_R = (\bar{R} + 3\sigma_R - \bar{R}) \div 3$$

$$= (D_4\bar{R} - \bar{R}) \div 3$$

$$= (1556 - 604.4) \div 3$$

$$= 317.2 \text{ pCi/liter}$$

Let range $= r = w\bar{R} + x\sigma_R = 180$ pCi/liter

Define normalized range $= w + x$

for $r > \bar{R}$, $w = 1$

then $r = w\bar{R} + x\sigma_R = \bar{R} + x\sigma_R$

or $x = \dfrac{r - \bar{R}}{\sigma_R}$

therefore $w + x = 1 + x = 1 + \dfrac{r - \bar{R}}{\sigma_R}$

for $r \leq \bar{R}$, $x = 0$

then $r = w\bar{R} + x\sigma_R = w\bar{R}$

or $w = \dfrac{r}{\bar{R}}$

therefore $w + x = w + 0 = \dfrac{r}{\bar{R}}$

since $r < \bar{R}$ $(180 < 604.4)$

$$w + x = \frac{180}{604.4}$$

$$= 0.30$$

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* 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

$$D = \bar{x} - \mu$$

$$= 3120 - 3273$$

$$= -153 \text{ pCi/liter}$$

Standard error of the mean $= \sigma_m$

$$\sigma_m = \frac{\sigma}{\sqrt{N}}$$

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

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

$$ND = \frac{D}{\sigma_m}$$

$$= \frac{-153}{206.1}$$

$$= -0.7$$

Experimental sigma (all laboratories) = s_t (See Figure 2)

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

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

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

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'

$$D' = \bar{x} - GA$$

$$= 3120 - 3290$$

$$= -170 \text{ pCi/liter}$$

$$ND' = \frac{D'}{\sigma_m}$$

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

$$= -0.8$$

*U. S. GOVERNMENT PRINTING OFFICE: 1977—785-181

TECHNICAL REPORT DATA
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1. REPORT NO.	2.	3. RECIPIENT'S ACCESSION NO.
EPA-600/4-77-047		

4. TITLE AND SUBTITLE	5. REPORT DATE
STATUS AND QUALITY OF RADIATION MEASUREMENTS Food and Human Urine	October 1977 6. PERFORMING ORGANIZATION CODE

7. AUTHOR(S)	8. PERFORMING ORGANIZATION REPORT NO.
D. G. Easterly, R. R. Kinnison, A. N. Jarvis, and R. F. Smiecinski	

9. PERFORMING ORGANIZATION NAME AND ADDRESS	10. PROGRAM ELEMENT NO.
Environmental Monitoring and Support Laboratory Office of Research and Development U.S. Environmental Protection Agency Las Vegas, Nevada 89114	1HD621 11. CONTRACT/GRANT NO.

12. SPONSORING AGENCY NAME AND ADDRESS	13. TYPE OF REPORT AND PERIOD COVERED
U.S. Environmental Protection Agency-Las Vegas, NV Office of Research and Development Environmental Monitoring and Support Laboratory Las Vegas, NV 89114	Preliminary, 1972-1975 14. SPONSORING AGENCY CODE EPA/600/07

15. SUPPLEMENTARY NOTES

16. ABSTRACT

As part of the radiation quality assurance program conducted by the U.S. Environmental Protection Agency, calibrated radionuclide solutions are distributed to participating laboratories for instrument calibration and yield determinations. Laboratory performance studies involving the analysis of radionuclides in environmental media are also conducted.

A summary is given of the results for the food and human urine cross-check programs for 1972-1975. For tritium, which was the least difficult to analyze, eighty-two percent of the laboratories were within the control limits for accuracy and ninety-nine percent within the control limits for precision over the 3-year period. For strontium-89, and most difficult to analyze, thirty-three percent were within the accuracy control limits and seventy-seven percent within the precision control limits.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS		b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
barium-140	potassium	laboratory performance	07E
cesium-137	quality assurance	intercomparison studies	12B
food analysis	quality control	cross-check	14D
human urine analysis	radiation chemistry		18B,D,H
iodine-131	strontium-89		
isotopes	strontium-90		
	tritium		

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS *(This Report)*	21. NO. OF PAGES
RELEASE TO PUBLIC	UNCLASSIFIED	44
	20. SECURITY CLASS *(This page)*	22. PRICE
	UNCLASSIFIED	

EPA Form 2220-1 (9-73)