USCEA/NIST MEASUREMENT ASSURANCE PROGRAMS FOR THE RADIOPHARMACEUTICAL AND NUCLEAR POWER INDUSTRIES

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Abstract - In cooperation with the U.S. Council for Energy Awareness (USCEA), the National Institute of Standards and Technology (NIST) supervises and administers two measurement assurance programs for radioactivity measurement traceability. One, in existence since the mid 1970s, provides traceability to suppliers of radiochemicals and radiopharmaceuticals, dose calibrators, and nuclear pharmacy services. The second program, begun in 1987, provides traceability to the nuclear power industry for utilities, source suppliers, and service laboratories. Each program is described, and the results of measurements of samples of known, but undisclosed activity, prepared at NIST and measured by the participants are presented.

NIST has more than 100 Cooperative Research and Development Agreements (CRADAs) in which individual companies work directly with NIST researchers to achieve joint goals. NIST collaborates on two such programs with the USCEA: one is a measurement assurance program for the radiopharmaceutical industry; and one is a measurement assurance program for the nuclear power industry.

The USCEA is the commercial nuclear power industry's national communications and information association. Its main function is to disseminate information on nuclear power to the public using print and TV advertisements, and through several publications. USCEA represents a broad spectrum of nearly 400 companies in the U.S. and overseas (USCEA 1990). USCEA is involved with these measurement assurance programs only as a service to their members. The companies that were interested in starting these measurement assurance programs already belonged to USCEA, and this provided a common infrastructure already in place to hire people to work at NIST to provide the services that they desired.

The companies that started these programs did so because the programs that already existed did not fully meet their needs. There are several advantages that these programs have that others do not. First, they provide the participants with a direct link of their measurements to the national standards. Second, the participants can receive the kind of radionuclides that they want, at the activity levels that

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they want, in the mixtures that they want, with the interferences that they want, and in the form that they want. Third, the participants find the program cost effective because the cost of the program is offset by savings on customer complaints due to improved measurement accuracy, as in the radiopharmaceutical program, or the need to perform fewer audits on source suppliers, as in the power plant program. Finally, the participants have a ready access to an independent third party if a measurement dispute occurs between participants or between a participant and a customer or regulator.

The remainder of this paper will briefly describe both measurement assurance programs, concentrating on four areas: 1) the participants in each program; 2) the standards and other services they receive; 3) how the programs are supported financially; and finally, 4) a summary of the results of the participants measurements compared to the NIST values. Previous publications (Golas and Calhoun 1983; Hoppes 1990; Gray, Golas, and Calhoun 1990) can provide more detail on certain aspects of each program.

The radiopharmaceutical Measurement Assurance Program (MAP) began in the mid 1970s. The current participants are shown in Figure 1. Together, the participants represent the entire spectrum of the radiopharmaceutical industry from bulk radiochemical suppliers, to radiopharmaceutical manufacturers, to a radiopharmacy that provides unit doses of radiopharmaceuticals in the form of filled syringes and capsules to hospitals for individual diagnostic tests and treatments. The Food and Drug Administration also participates through an interagency agreement with NIST.

Figure 2 shows the distribution schedule for 1993. Ten different Standard Reference Materials (SRMs) are produced yearly, one per month, except for May and November. During these two months, known as "open months," participants can elect to submit samples to NIST for verification. This allows participants to get traceability on radionuclides that are important to them but are not on the schedule during the year, or to perform a test on some measurement problem that they may have had. Except for ^{99m}Tc, each monthly distribution consists of a high-level and a low-level SRM. The high levels are in the 0.1 to 10 GBq (multi-millicurie) range of activity while the low levels are usually from 5 to 800 MBq (one to several hundred microcuries). All the standards are in the form of 5 milliliters of solution in standard NIST glass ampoules except for ^{133}Xe, which is provided in a 5-mL Pyrex®[(1)] ampoule. The lack of availability of high-level standards was one of the original reasons for beginning the program. The other major reasons were that some standards that were needed by the industry were not available in the form they required and some decay data were not well known. This was a source of problems many years ago because if one company or supplier based an activity value on a measurement of a gamma ray with a poorly known probability per decay, and someone else used another method of calibration such as an ionization chamber, the activities sometimes did not agree. Because some of these companies purchased large quantities of radioactive material from each other, the economic consequence of being different from each other by a few percent was one of the major incentives for starting this program.

The SRMs are supplied to the participants with the NIST-measured activity undisclosed, or as "blinds." The participants make their measurements on the sources and report their results on a questionnaire supplied with the source. After the questionnaire is received and analyzed, NIST issues

(1) The mention of commercial products does not imply recommendation or endorsement by NIST, nor does it imply that the products identified are necessarily the best available for the purpose.

a report which compares the participant's measurement with the NIST value, providing traceability for the measurement. Usually, one beta-emitting SRM is issued every year, but this year it was replaced by ^{153}Sm. Also, ^{57}Co was substituted for ^{51}Cr which has been prepared in July during the last several years.

The low-level standards are also available for sale to the general public, and the sale of these help to support the operation of the program. The SRMs provided to the public are not available as "blinds." The participants that created and support the program believe that allowing non-participants to receive the SRMs as blinds would remove the incentive to belong to the program. This would result in the program collapsing from lack of support, with the result that the SRMs might not be available at all since NIST would have no resources available to prepare the radiopharmaceutical SRMs.

Figure 3 is a diagram of how the radiopharmaceutical program operates. First, the participants meet at an annual steering committee meeting to decide on the 10 standards that are to be prepared the following year. The participants decide on which high- and low-level SRMs they want to receive and they pay USCEA for these along with a participation fee, currently $2,000. The $2,000 participation fee covers the cost of the two "open months," which are provided to the participants at no additional charge. Participants must also pay to belong to USCEA. USCEA charges a membership fee which is different for each company, based on a formula that takes into account the company's income related to the nuclear industry. This charge is separate from these measurement assurance programs and is charged whether or not they belong to one of these programs. The minimum membership fee is $500 and increases from there. USCEA uses the money collected from the participants to pay NIST for the SRMs provided to the participants and for supplies and raw materials needed to prepare the standards. This last category currently costs approximately $10,000 per year. Payment for the SRMs provided to the participants is done quarterly. The research associates are employees of USCEA and are paid as any other USCEA employee. USCEA employs two research associates, one for each of the measurement assurance programs. However, all the work for both programs is done cooperatively, so the costs are averaged between both programs. As previously mentioned, the low-level SRMs are advertised for sale to the general public to help support the program. The current price for these is $378 each. From the sale of each one of these, $115 is kept by NIST and the rest is returned to USCEA, also quarterly. The other arrows on the diagram indicate that the research associates help NIST personnel with some of their needs in the production of other SRMs, and NIST personnel assist the research associates with the preparation of the radiopharmaceutical SRMs, as well as the sources for the power plant program. All parties are in agreement that the contributions of each balance out.

The newest USCEA measurement assurance program was set up with the nuclear power industry in 1987. The reasons for setting up this program were similar to those of the other program, namely that other programs that existed at the time didn't fully meet their needs. This program was modelled after the radiopharmaceutical program but has several differences. The first difference is that there are different categories of participants rather than just one: source suppliers, service laboratories, and utility participants. Each participant pays the same amount each year, currently $8,500 but depending on the category, receives different benefits. Currently, there are 25 participants. Most, if not all, of the domestic source suppliers are members, only a small sample of service laboratories, and approximately one-quarter of the utilities that operate nuclear power plants, representing approximately one-third of all the commercial nuclear power plants currently in operation. A list of current participants is shown in Figure 4.

All of the participants receive six sources per year that are prepared by the research associates in cooperation with NIST. The distribution schedule for 1993 is shown in Figure 5. In addition to the samples prepared at NIST, there are "open months" similar to the case for the radiopharmaceutical program. In this program the companies pay for what are known as "credits" that the participants may use for having their own sources verified. Each credit is worth $1,000, which is the same as the cost of the "open months" for the radiopharmaceutical participants. However, in this case the samples that are submitted to NIST for verification are measured by NIST personnel instead of the research associates, and depending on the category of the participant, they may send in different numbers of sources each year. The source suppliers may have up to twelve credits worth of calibrations performed, service laboratories may have up to three credits worth of calibrations performed, and utilities get one credit, or may instead receive up to $1,000 worth of other NIST SRMs sent to them at no charge during the year or receive extra samples of the six sources prepared at NIST. Incidently, if a participant submits a sample that requires a greater amount of time to calibrate, such as mixed gamma sources or beta calibrations, the participant is usually charged more than one credit for the measurement.

The six sources per year prepared for this program are distributed approximately one every other month. In general, these sources are at lower activity levels than the radiopharmaceutical SRMs, but higher than those available in some other cross-check programs, so that the uncertainties due to background corrections will be minimized. The sources are supplied as a solution in our standard 5-mL ampoules, as simulated 47-mm-diameter filters for gamma-ray measurements, as rectangular paper filters when the filters must be dissolved for analysis, and as 33-mL double-stopcock glass spheres for gases. The gas sources are purchased from another supplier and calibrated at NIST. As in the radiopharmaceutical program, the participants decide on the sources that they want prepared at an annual steering committee meeting. At the beginning of the program, the sources were prepared as single radionuclides with no interferences so that the participants could use them to check their calibrations and measurement techniques. In subsequent years, the matrices have become more complex with mixtures of multiple radionuclides, often with interferences added, and sometimes without the radionuclides in the mixtures revealed so that the participants must also identify the radionuclides as well as report on the quantities measured. In 1993 we are returning somewhat to more single radionuclides and simpler mixtures to allow the participants that were not in the program the first two or three years to use the sources to also calibrate their measuring systems.

Figure 6 is a diagram of how this program operates. In general, it is much the same as the radiopharmaceutical program, but simpler. First, there are no SRMs available for sale to the public, so the box that was at the bottom of Figure 3 is not there. Consideration was given to offering these sources as SRMs when the program was first set up, but several source suppliers thought that this would put NIST in direct competition with them and they did not want that. Because there are no SRMs for sale, there is no arrow for money to be returned to USCEA. Consequently, the participants pay more for the sources and calibrations that they do receive. More money is also paid to NIST because NIST does more work for the many calibrations that are performed for the source suppliers and service laboratories. Last fall a review of the program was made by representatives of the steering committee. After looking over the amount of work that was being performed, they recommended to the steering committee that the addition of a technician was appropriate to help in the production of the sources and assist NIST personnel in making measurements on the submitted samples. The technician is supported one-half by USCEA and one-half by NIST. USCEA employs the technician, the same as the research associates. NIST supports their half by charging less for the credits provided to the source suppliers and service laboratories up to an amount equal to one-half of the technician's salary. The steering committee has also paid additional money to NIST over the past few years to have special calibrations made that probably would not have been done without outside support. Because of this additional support, NIST has developed new calibrations for ^{141}Ce and ^{144}Ce, and calibrations for both a mixed gas and solid Marinelli beaker geometries.

A closer look at the benefits received by each category of participant reveals that the utility participants subsidize the majority of the open month "credits" used by source suppliers. The service laboratories are roughly in balance and get back what they put in. The participants set the program up this way from the start. One of the reasons advanced for creating this program was that if the utilities were confident that the source suppliers were making sources that were traceable to NIST then they would have to perform fewer or no audits on the source suppliers and everyone would save money and time.

Figure 7 is a histogram of the results received from the radiopharmaceutical participants since the beginning of the program. The histogram represents 1410 results submitted from June 1975 through January 1993. Approximately 84% of the results are within ±5% of NIST, and about 96% are with ±10% of NIST. Of the 1.3% that are greater than ±20% of NIST, most of these are results from the earlier years. It is likely that measurements have improved over the years, but it is also possible that companies that made poor measurements just stopped submitting results. Because the people that receive these SRMs know that they are making their measurements in order to become traceable to NIST, one could assume, certainly not "incorrectly," that these results represent "best efforts." It can only be guessed how well others not in the program routinely do. It is reassuring to see that the values are centered around the NIST value, proving that no matter what the different participants use to calibrate their measuring equipment, on the whole they agree with NIST.

The histogram from the nuclear power program, Figure 8, shows the results categorized by the type of participant. There are a total of 1388 results for sources distributed from June 1987 through October 1992: 1061 from utility participants; 230 from source suppliers; and 97 from service labs. The results are only for the distributed samples and not from sources submitted to NIST for verification from the source suppliers and service laboratories. The histogram shows a wider dispersion from NIST than the radiopharmaceutical results, but this should not be surprising. In almost all cases, the sources measured by the power plant participants were more complex, often with intentional interferences included to make the measurements difficult. In many cases, the samples required chemical processing before measurements could be performed, unlike the radiopharmaceutical SRMs. In spite of all this, the results still fall around the NIST value.

As well as providing calibrated sources and SRMs to the participants in these programs, another task is to assist any participant with any problems they may have encountered in measuring these sources. This is done directly, if someone calls with a problem, but participants can also obtain useful information from the summaries that are issued after everyone has submitted his result. The summary for the radiopharmaceutical program is fairly simple. For each radionuclide distributed, a list is generated which shows all deviations from the NIST value for each participant, and the type of detector used to make the measurement. An average difference of the results from the NIST value is also generated so individual participants can compare their difference from everyone else. The results are reported with the companies unidentified. The summary provides only a few details for this program, because it would be easy to identify a company after a while if too much of the measurement technique was described.

For the power plant program, the summary provides more detail. For each result, the difference from the NIST value, the type of detector used, a short description of how the source was measured is provided as shown in Figure 9. Again the participants are not identified. The sample number of the source is shown, but this is randomly assigned to each participant before the sources are shipped. The reason the sample number is provided is so that multiple results for one source can be identified. Some participants submit individual questionnaires for several different detectors or geometries for one source so that they can obtain traceability for particular geometries or measuring equipment. Also included is a graph of the participant's results compared to the other results submitted, as shown in Figure 10. The vertical line above and below the participant's value indicates the one sigma uncertainty on the participant's result as reported on the questionnaire. The long, horizontal dashed lines mark the NIST combined standard uncertainty.

In conclusion, this has been a summary of both USCEA/NIST measurement assurance programs: who the participants are; how the programs are structured; how the programs are financed; and how well the participants are making their measurements. The programs fill a need in each industry and the participants find the programs useful, necessary, and cost effective. They also have an opportunity each year at the annual steering committee meeting to change their programs to adapt to any changes that are taking place to meet new demands or regulations.

REFERENCES

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Gray, D. H., D. B. Golas, and J. M. Calhoun. 1990. "Review of the USCEA/NIST Measurement Assurance Program for the Nuclear Power Industry." Radioactivity and Radiochemistry 2:1.

Hoppes, D. D. 1990. "Demonstrated Measurement Traceability for Nuclear Power Radiochemistry Departments." Radioactivity and Radiochemistry. Winter 1990.

U.S. Council for Energy Awareness. 1990. Annual Report. Washington, D.C.

PARTICIPANTS IN THE NIST/USCEA RADIOACTIVITY MEASUREMENTS ASSURANCE PROGRAM FOR THE RADIOPHARMACEUTICAL INDUSTRY

Bristol-Meyers Squibb Company

Cintichem, Incorporated

DuPont Merck Pharmaceuticals Company

Hybritech, Incorporated

Mallinckrodt Medical, Incorporated

Medi+Physics, Incorporated

Nordion International, Incorporated

Syncor International Corporation

Figure 1

1993 DISTRIBUTION SCHEDULE FOR THE NIST/USCEA RADIOPHARMACEUTICAL PROGRAM

Month	Radionuclide	High Level	Low Level
January	^{131}I	750 MBq (20 mCi)	25 MBq (700 μCi)
February	^{99}Mo	3 GBq (80 mCi)	75 MBq (2 mCi)
March	^{133}Xe	7.5 GBq (200 mCi)	750 MBq (20 mCi)
April	^{67}Ga	375 MBq (10 mCi)	20 MBq (500 μCi)
May	OPEN	n/a	n/a
June	^{201}Tl	225 MBq (6 mCi)	35 MBq (900 μCi)
July	^{57}Co	75 MBq (2 mCi)	10 MBq (250 μCi)
August	^{111}In	375 MBq (10 mCi)	20 MBq (500 μCi)
September	^{99m}Tc	7.5 GBq (200 mCi)	n/a
October	^{153}Sm	375 MBq (10 mCi)	20 MBq (500 μCi)
November	OPEN	n/a	n/a
December	^{125}I	750 MBq (20 mCi)	6 MBq (150 μCi)

Figure 2

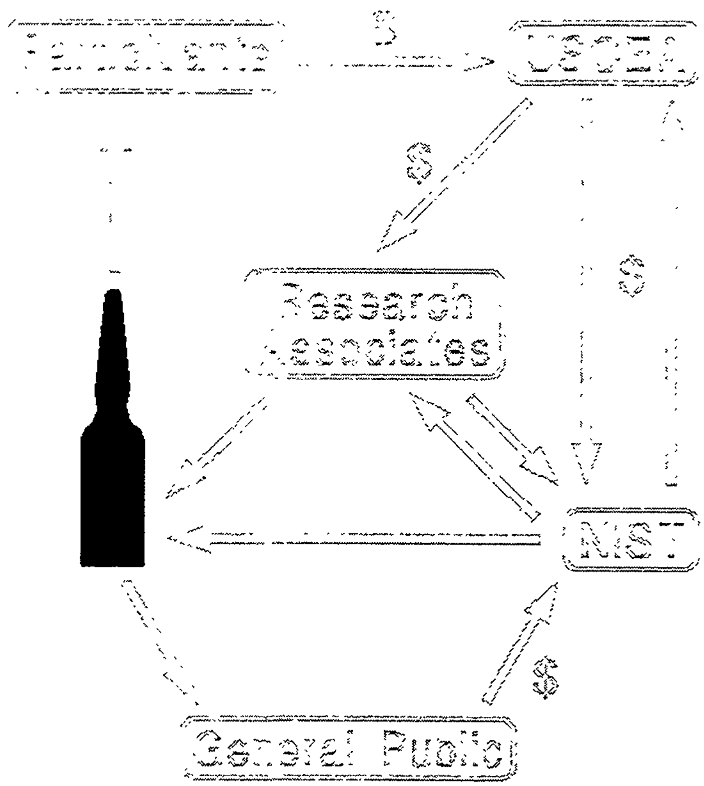

Figure 3 - Diagram of Radiopharmaceutical Program Operation

PARTICIPANTS IN THE NIST/USCEA RADIOACTIVITY MEASUREMENTS ASSURANCE PROGRAM FOR THE NUCLEAR POWER INDUSTRY

SOURCE SUPPLIERS

Amersham Corporation
Arlington Heights, Illinois

Analytics, Incorporated
Atlanta, Georgia

Isotope Products Laboratories
Burbank, California

North American Scientific
North Hollywood, California

The Source
Santa Fe, New Mexico

TMA/Eberline
Albuquerque, New Mexico

SERVICE LABORATORIES

Atlan-Tech, Incorporated
Roswell, Georgia

Battelle, Pacific Northwest Laboratories
Richland, Washington

Institute of Nuclear Energy Research
Lung-tan, Taiwan

Scientech, Incorporated
Gaithersburg, Maryland

UTILITIES

American Electric Power Service Co.
Bridgman, Michigan

Babcock & Wilcox
Lynchburg, Virginia

Baltimore Gas and Electric Company
Lusby, Maryland

Commonwealth Edison Company
Maywood, Illinois

Connecticut Yankee
East Hampton, Connecticut

Houston Lighting & Power Company
Houston, Texas

New York Power Authority
White Plains, New York

Northeast Nuclear Energy Company
Waterford, Connecticut

Omaha Public Power District
Fort Calhoun, Nebraska

Pacific Gas & Electric Company
San Ramon, California

Pennsylvania Power & Light
Berwick, Pennsylvania

Portland General Electric
Rainier, Oregon

Public Service Electric & Gas
Hancocks Bridge, New Jersey

Rochester Gas & Electric Company
Ontario, New York

Yankee Atomic Electric Company
Bolton, Massachusetts

Figure 4

1993 DISTRIBUTION SCHEDULE FOR THE NIST/USCEA POWER PLANT PROGRAM

MONTH	RADIONUCLIDE(S)	SOURCE DESCRIPTION
February	^{131}I	37 kBq or 3700 kBq in 5 mL of solution
April	^{60}Co, ^{65}Zn, ^{134}Cs, ^{137}Cs	100-200 Bq each, on an air filter
June	^{230}Th	Less than 200 Bq in 5 mL of solution
August	^{110m}Ag	5000 $\gamma s^{-1} g^{-1}$ in 5 mL of solution
October	^{85}Kr, ^{127}Xe, ^{133}Xe	1 MBq, 100 kBq, and 200 kBq, respectively, in a 33 mL double-stopcock borosilicate-glass sphere
December	^{55}Fe	37 kBq in 5 mL of solution

Figure 5

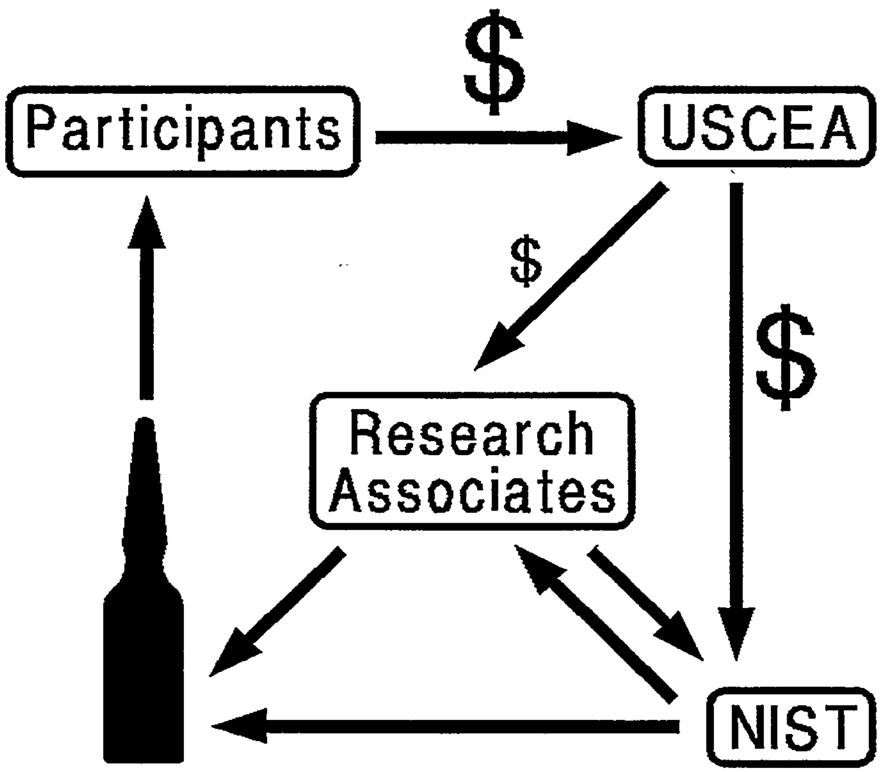

Figure 6 - Diagram of Program Operation

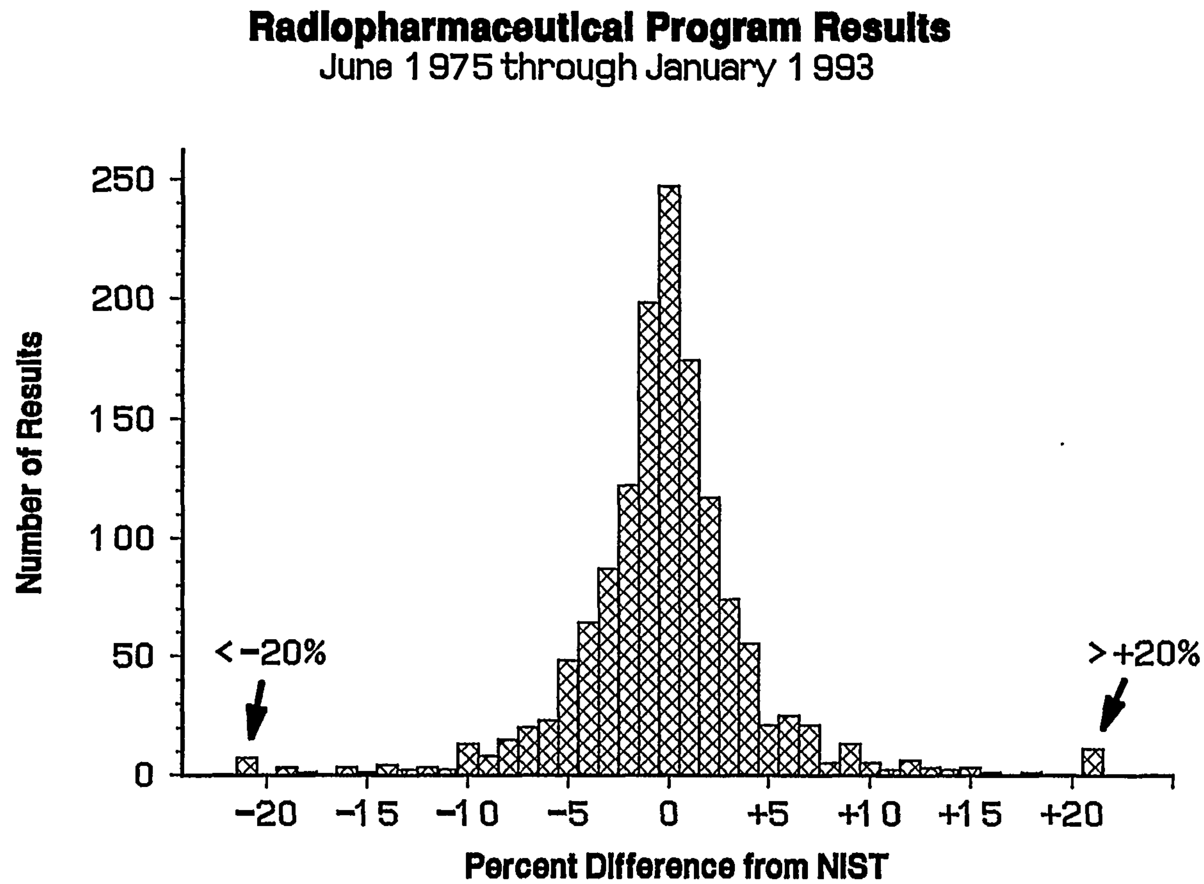

Figure 7 - Histogram of the Results Received from the Radiopharmaceutical Participants

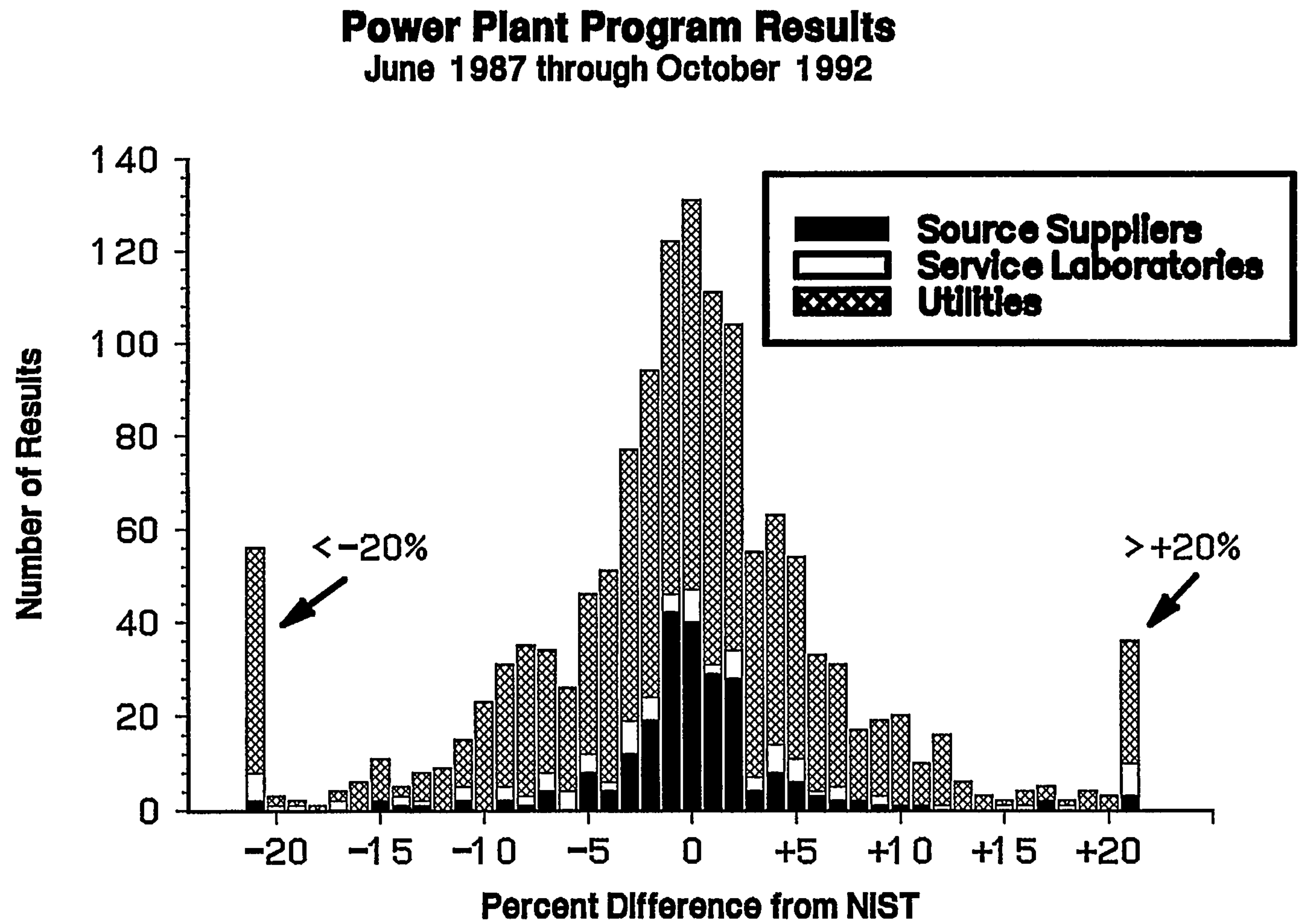

Figure 8 - Histogram from the Nuclear Power Program

ZN-65 June 1992 (8731)				Results by Date (As of 5/19/93)
	Sample	% Difference	Detector Used	Summary of Source Preparation Technique Used
*	1	2.31	Ge(Li) detector	Counted three times "as is" for 1000 seconds each 4 cm from detector.
	2	2.39	High purity Ge detector	Counted "as is" for 30 minutes.
	6	5.92	Ge(Li) detector	Counted "as is" 2 cm from Ge(Li) crystal.
	9	0.77	Ge(Li) and HPGe detectors	Counted "as is" six times: two shelf positions on three detectors.
	10	5.22	High purity Ge detector	Sample measured "as is" in 47 mm filter geometry for 600 seconds.
	11	-1.29	Intrin. Ge closed-end coaxial	Filter was counted "as is" on four detectors at approximately 6 cm from detector for approximately 3000 seconds each.
	13	-1.73	High purity Ge detector	Sample measured "as is" at distances from 50 to 100 mm from detector. Counting times ≥ 54,000 seconds.
	14	-10.56	Germanium detector	Counted "as is" four times, 2,000 seconds at 10 cm and 10,000 seconds at 20 cm on two detectors.
	15	3.16	High purity Ge detector	Sample was placed in planchet and counted "as is" 2000 seconds each at an elevated geometry on three different detectors.
	20	-1.39	Intrin. Ge closed-end coaxial	Counted "as is" 11 times, 900 seconds each measurement, 43 mm from detector.
	21	3.21	High purity Ge detector	Filter was counted "as is" one time each on five detectors at 2.91" from detector, 10,800 to 60,000 second counts.
*	22	1.13	Intrinsic Ge detector	Placed in 2" stainless steel planchet and counted "as is" on shelf #2.
*	22	-6.96	Intrinsic Ge detector	Placed in 2" stainless steel planchet and counted "as is" on shelf #2.
*	22	12.46	Intrinsic Ge detector	Placed in 2" stainless steel planchet and counted "as is" on shelf #2.
	23	-0.08	High purity Ge detector	Counted "as is" 20 times on two detectors for 2000 seconds each.
	24	-1.70	Low-energy Ge detector	Sample measured "as is" in petri dish placed about 1.5" above detector end cap. 14,400-second count time.
	25	-4.88	Intrin. Ge closed-end coaxial	Counted "as is" 11 times, 900 seconds each measurement 43 mm from detector.
	27	5.16	Reversed electrode Ge detector	Filter was placed in a petri dish and counted three times "as is" at approximately 13 cm above detector end cap.
	27	3.82	Reversed electrode Ge detector	Filter was placed in a petri dish and counted three times "as is" at approximately 13 cm above detector end cap.
	28	-5.34	High Purity Ge detector	Sample measured "as is" in planchet. Counted at 3, 6, and 10 cm.

NOTE: * means that the result was not counted for traceability.

Figure 9 - USCEA/NIST Radioactivity Measurements Assurance Program for the Nuclear Power Industry

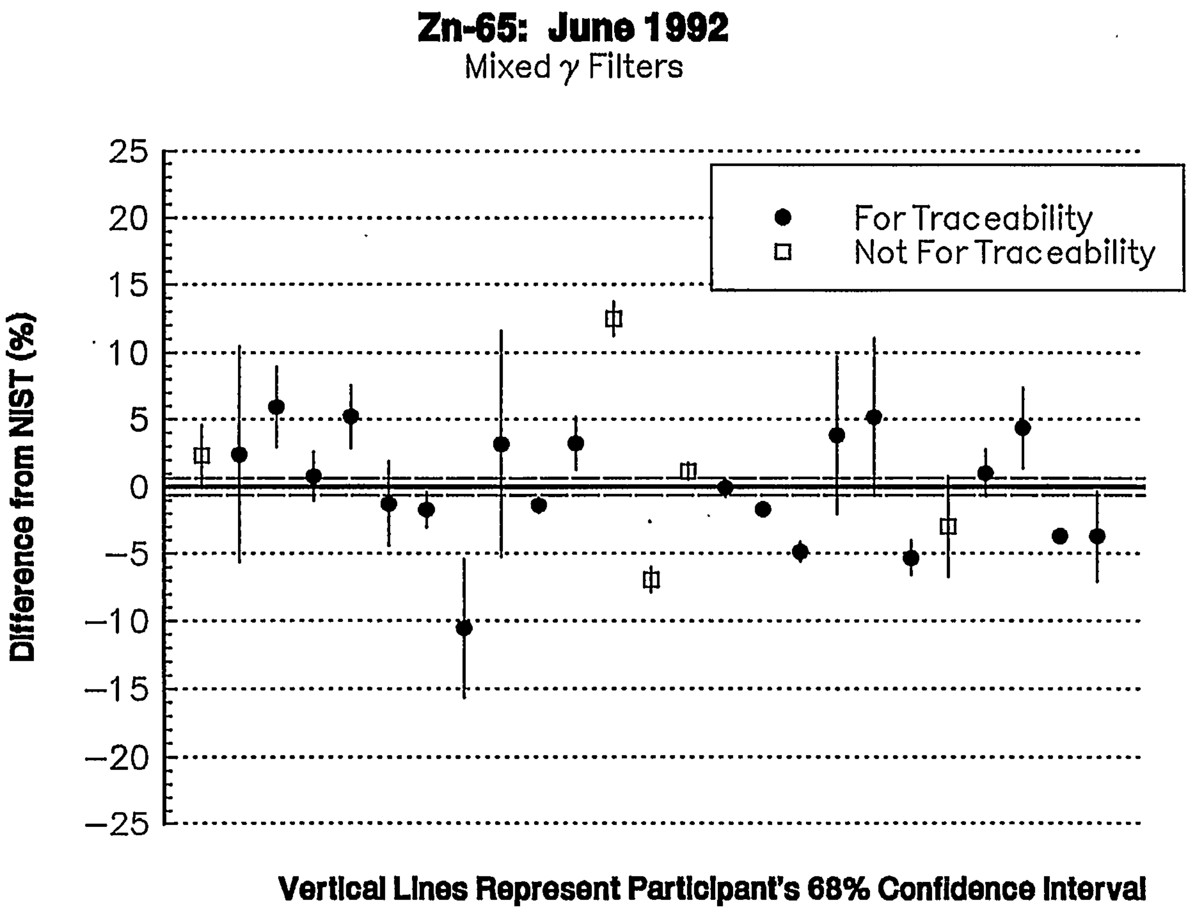

Figure 10 - Graph of Participant's Results Compared to the Other Results Submitted