RF P-2997

CONF-800315--1

APPLICATION OF AMERICAN NATIONAL STANDARDS OF CALIBRATION TECHNIQUES OF BULK MEASUREMENTS FOR NUCLEAR MATERIALS CONTROL

MASTER

Louis W. Doher - ANSI-N15
and
Kenneth B. Gerald
Rockwell International
Rocky Flats Plant
Energy Systems Group
P. O. Box 464
Golden, Colorado 80401

1. Abstract

In 1975 Subcommittee ANSI-INMM-8, "Calibration Techniques for Nuclear Material Control" under the guidance of the American National Standards Institute Committee N15 and sponsored by the Institute of Nuclear Materials Management, published four standards for calibration of bulk measurement of nuclear materials. The calibration techniques include those for mass, volume, nondestructive assay, and plutonium calorimetry measurements. Since that time, calibration and research personnel of the Rocky Flats Plant operated by Rockwell International for the United States Department of Energy and workers at other facilities have applied the direction and guidance of these standards.

The results of the applications are reported, and the value of each standard is discussed. Examples are included together with certain shortcomings and future revision plans.

2. Introduction

The Institute of Nuclear Materials Management (INMM) is the secretariat of the American National Standards Institute for development of American National Standards for Nuclear Materials Control. This INMM committee is designated N15 and currently is expanding its scope to increase the subcommittees beyond the present twelve active groups.[1]

One of these subcommittees is designated ANSI-INMM-8, "Calibration Techniques for Nuclear Material Control" and has developed four American National Standards which provide guidance for the calibration of bulk measurements of Special Nuclear Materials (SNM). These standards are designated ANSI N15.18-1975, "Mass Calibration Techniques for Nuclear Material Control,"[2] ANSI N15.19-1975, "Volume Calibration Techniques for Nuclear Material Control",[3] ANSI N15.20-1975, "Guide to Calibrating Nondestructive Assay (NDA) Systems"[4] and ANSI N15.22-1975, "Calibration Techniques for the Calorimetric Assay of Plutonium-Bearing Solids Applied to Nuclear Materials Control."[5]

The guidance and rationale provided by these standards has been applied at the Rocky Flats Plant (RFP) operated by Rockwell International, Energy Systems Group, for the United States of Department of Energy (DOE) as well as at other nuclear facilities. These applications have been instituted during both normal plant operation and experimental measurements. The results of these applications are discussed in their numerical order.

3. Discussion

ANSI N15.18-1975 (Mass)

At Rocky Flats Plant the Chemistry Standards Laboratory (CSL) has instituted (1) the concept of mass measurement process, (2) characterization of that process and (3) procedurization of assigning mass values of test objects with the associated uncertainty determination as outlined in ANSI N15.18-1975, Sections 4, 5, and 6 respectively. This effort supports a checkweighing program to estimate the uncertainties of the mass measurement processes for nuclear material control at the RFP.[6]

The task force designated INMM 8.1, which developed ANSI N15.18-1975, instituted a unique concept in Section 7 of the Standard for the establishment and maintenance of the control of mass measurements of uranium hexafluoride (UF_6) both within and between facilities. Emphasis is placed on the role of control of the measurements between facilities, and thus establishes decision points for detection of measurement problems and making safeguards judgments.

The unique concept include the use of artifacts of UF_6 packaging cylinders, cal-

DISCLAIMER

ibrated by a central authority, the National Bureau of Standards (NBS), to introduce the mass unit into all of the industries' weighing processes.[1] These are called Replicate Mass Standards (RMS). This feat is accomplished by comparing the RMS to each facility's In-House Standards (IHS), also artifacts, and thence the usage of these IHS to quantify the systematic and random errors of each UF_6 mass measurement process. Section 7 of the Standard also provides an Administrator for a UF_6 measurement assurance program (MAP) and the guidelines of the program appear in Section 8 of the Standard.

The results of application of the Standard have shown realistic and acceptable results and have been reported.[7,8,9] These demonstrations have shown that the MAP administrator can immediately update the data base, and thus detect possible safeguards and internal mass measurement problems. All of these actions and judgments can then be communicated to the shipper-receiver facilities and/or regulatory bodies for immediate action. Further, these actions have demonstrated that the methods used for detection of diversion and inspection of UF_6 transfers are applicable to both national and international systems.

The implementation of the Standard's concept for UF_6 measurements in an American industry-wide program has now been inaugurated. The program has been made possible with the Nuclear Regulatory Commission providing funding to allow the National Bureau of Standards' Office of Measurements for Nuclear Safeguards to actively provide the administration and technical support for the implementation of ANSI N15.18-1975 UF_6 mass measurements throughout the industry.[10] The goal of this implementation is to provide an efficient means for obtaining uniform mass measurement of UF_6 based on the national measurement system. This program is underway and has been well received by the industrial participants as recently reported.[11]

Future revision plans include expansion of Sections 7 and 8 based on the information gleaned from this implementation.

ANSI N15.19-1975 (Volume)

In Section 5 of ANSI-N15.19-1975 "Volume Calibration Techniques for Nuclear Material Control" guidance is provided for selection of equipment including tank configuration, response systems and calibration equipment. Further, the Standard details criteria for calibration of test measures (provers) for tank volume calibration procedures in Sections 6 and 7. Sections 8 and 9 deal heavily with treatment of volume calibration data, data analysis and evaluations.

The RFP CSL has designed a positive displacement piston prover (PDPP) for use in calibrating tankage. The device was calibrated for delivering known volumes of liquid in accordance with appendix C of the Standard.[12] The laboratory used the guidance of ANSI N15.19-1975, Sections 8 and 9 for estimating the uncertainties of volume calibration of process tankage in a plutonium processing facility.[13]

While the guidance in ANSI N15.19-1975 has been shown to be adequate for vertical straight-walled tanks, it is not always applicable to tanks requiring unusual engineering configurations. The statistical guidance provided in the Standard has been found to be overly involved for calibration personnel. Also, the limited scope of the standard, i.e., a linear least squares fit of the calibration data, has been found to be inadequate for all cases. A revision, scheduled for 1980, will correct these deficiencies.

Further application of ANSI N15.19-1975 was performed by the Barnwell Nuclear Fuels Plant (BNFP) operated by Allied General Nuclear Fuel Services.[14,15] During these studies, the operating personnel of BNFP relied heavily upon the expertise, counsel and direction of selected members of INMM 8.2. Their direction consisted of (1) planning for tank calibration, (2) choice of recently developed measurement response systems, (3) procedural guidance, with emphasis on details to provide a base for future measurement and calibration judgments, (4) data collection and (5) data treatment to produce an adequate calibration error budget.

This guidance was valuable in achieving the goals of the volume calibration of the plant. However, ANSI N15.19-1975 error analysis procedures required expansion and revision in order to obtain realistic error statements concerning the calibration function as applied to the physical conditions experienced at BNFP. Again, those expansions and revisions are planned to be incorporated in the future revision of ANSI N15.19-1975.

Another exercise was conducted at the Savannah River Plant, operated by du Pont, in calibrating a replacement tank[16]. Again, the counsel of the INMM 8.2 members was used and again similar expansions and revisions were required.

The Mass and Volume Section of the NBS undertook a study to determine the accuracy of a nuclear fuel reprocessing tank volume calibration comparing several instruments as liquid level detectors and generat-

ing data for testing ANSI N15.19-1975.[17] The study included the Standard's analysis technique for application to tank calibration data. The study concluded that other more descriptive models should be offered as options in future publications of ANSI N15.19-1975.

The Technical Support Organization (TSO) of Brookhaven National Laboratory (BNL) used the guidance and rationale of ANSI N15.19-1975 in the design of volume calibration and operational measurements using specialized equipment at BNFP.[18] This experience proved the value of the Standard and promoted thoughts for expanding the "Equipment" section of ANSI N15.19-1975.

ANSI N15.20-1975 (Nondestructive Assay)

As the title of ANSI N15.20-1975, "Guide to Calibrating Nondestructive Assay (NDA) Systems" implies, its intent is only that of a guide rather than a definative procedure. As such, the text provides general guidance for selection, precalibration and qualification of NDA systems in Section 4, a discussion of measurement errors in Section 5, details of physical standards in Section 6, general and analytical concepts of calibration methods in Section 7, plus some descriptions of assay errors and a measurement assurance plan in Sections 8 and 9, respectively. Valuable aids to the user, including guidance for solution of problems encountered when using specific NDA techniques, are given in the appendix. Other appropriate mathematical aids are also included in the appendix.

The value of the Guide is exemplified by its use by writing groups of INMM-9 "Nondestructive Assay (NDA)." These proposed Standards all heavily reference ANSI N15.20-1975. These NDA American National Standards will include procedures for (1) Material Categorization, (2) Container Standardization, (3) Physical Standards, (4) Measurement Controls, (5) Techniques, and (6) Automation.

RFP research personnel have fabricated, calibrated, and tested a crate assay counter to estimate the plutonium content in waste for safeguards verification measurements.[19] Calibration of the instrument was achieved using a specially built crate with thirty-five tubes for insertion of physical standards. Neutron and gamma-ray response calibrations were obtained using guidance of Section 7 of ANSI N15.20-1975. Future calibrations, together with detection of associated errors, are planned using the rationale of Sections 5, 7 and 8 of the Standard.

A commercial segmented gamma-ray scanning device has been installed at RFP for measurement of plutonium and americium-241 in molten salt residues.[20] Both isotope assays are required. Calibration of the system was achieved using the physical standard guidance of N15.20-1975, i.e., both synthetically obtained and residue material, measured calorimetrically.[5]

A DOE requirement at RFP is that of verification of inventory measured by NDA systems. Verification is done using calorimetry. Recent verification packages have been used to provide more accurate NDA calibrations by postulating a log model and parameter estimates obtained by linear least squares procedure after a log transformation as prescribed in Section 7 of ANSI N15.20-1975.[21]

Application of this Standard has been well received by the calibration personnel but has been found to be lacking in some accepted options for calculation of both calibration and assay errors. Revision to the standard, planned in 1980, will include such options.

ANSI N15.22-1975 (Calorimetry)

ANSI N15.22-1975 titled "Calibration Techniques for the Calorimeteric Assay of Plutonium-Bearing Solids Applied to Nuclear Material Control" has as its scope the establishment of methods for calibration of heat-flow calorimetry systems within the confines described in the title. Within this scope, guidance and methodology is provided for equipment types and their operation and procedures in Sections 5 and 6, calibration techniques, including physical standards, in Section 7 and determination of plutonium content and the error analysis associated with this determination in Sections 8 and 9 respectively. Of primary value are the tables included in the standard which define values of nuclear constants for the radionuclides of interest, sample calculations, summaries of error sources and the uncertainties of the nuclear decay parameters. Clarity of the document is enhanced by the use of figures displaying equipment types and typical calibration and sensitivity graphics. The rationale used for obtaining the assignment of values for half-life and specific power of plutonium isotopes and americium-241 appear as an appendix.

RFP has used ANSI N15.22-1975 as both a calibration and assay guide for calorimetric

measurements of plutonium since its first day of publication. The sixteen calorimeters of RFP which measure plutonium-bearing solids operate equivalently because of the usage of this standard. Of notable importance, the Standard provides best estimates for the heat producing isotopes of plutonium and americium (as of 1975) for (1) disintegration energy, (2) decay mode, (3) total energy, (4) half-life, (5) decay constant, and (6) specific power.

A four litre calorimeter which was designed for use in preparing physical standards for the RFP NDA package counters has been calibrated using ANSI N15.22-1975 guidelines.[22] The Standard was also used to write a computer program, that calculates the weight of plutonium in a sample from the power emitted. The Standard was particularly useful by supplying the equations for the determination of plutonium content and for the corrections for radionuclide decay as well as the necessary constants and their standard errors. The program follows the sample calculation procedure illustrated in Table 2 of ANSI N15.22-1975. Through the guidance of ANSI N15.22-1975, all meaningful sources of error were incorporated into the program.[23] The derivation of the plutonium measurement uncertainties associated with the four litre calorimeters was obtained by using the general technique for estimating the variance of an arbitrary function of the random variables[24] and quantifying the errors of the specific power of the isotopes given in Table 4 of ANSI N15.22-1975 and by equations 21 and 22 of the Standard.

Plans for revision of this standard during 1981 will include acceptance of recently published values of plutonium isotopes half-lives by the DOE Plutonium Half-Life Committee[25,26], standardization of certification of physical standards by NBS and expansion of error treatment options.

4. Conclusions

The applications of these four calibration standards have been found to be of immense value throughout the American safeguards program. Interaction has been initiated at all levels within the European Safeguards Research and Development Association (ESARDA) and the INMM organizations to discuss possible mechanisms for extending such experience to the international level.[1] Based on such cooperation, implementation of forthcoming International Atomic Energy Agency requirements is expected to proceed on a comprehensive basis.

5. References

1. Bishop, Dennis M., "USA-INMM Safeguards Consensus Standardization Program Status," proceedings of First Annual Symposium on Safeguards and Nuclear Material Management, ESARDA, Brussels, Belgium, April 25-27, 1979, p. 348-351.

2. American National Standard N15.18-1975, "Mass Calibration Techniques for Nuclear Material Control," American National Standards Institute, 1430 Broadway, New York, NY 10018.

3. American National Standard N15.19-1975, "Volume Calibration Techniques for Nuclear Materials Control," American National Standards Institute, 1430 Broadway, New York, NY 10018.

4. American National Standard N15.20-1975, "Guide to Calibrating Nondestructive Assay Systems," American National Standards Institute, 1430 Broadway, New York, NY 10018.

5. American National Standard N15.22-1975, "Calibration Techniques for the Calorimetric Assay of Plutonium-Bearing Solids Applied to Nuclear Materials Control" American National Standards Institute, 1430 Broadway, New York, NY 10018.

6. Rocky Flats Plant Chemistry Standards Laboratory Procedure, "Weight Measurement Control Program," personal communication, Nov. 1975 (Revised Dec. 1979).

7. Doher, Louis W., "INMM-8 Pilot Program," Nuclear Materials Management, Vol. V, No. 1, Spring, 1976, p. 47.

8. Pontius, P. E., and Doher, L. W., "The Joint ANSI-INMM 8.1 Nuclear Regulatory Commission Study of Uranium Hexafluoride Cylinder Material Accountability bulk Measurements," Proceedings, 18th Annual Meeting, Institute of Nuclear Materials Management, Volume 6, No. 3, p. 480-487.

9. Doher, L. W., Pontius, P. E., and Whetstone, J. R., "A New Approach for Safeguarding Enriched Uranium Hexafluoride Bulk Tansfers," Proceedings, International Atomic Energy Agency International Symposium on Nuclear Materials Safeguards, Vienna, Austria, October 2-6, 1978.

10. Doher, L. W., "Implementation of ANSI N15.18 Uranium Hexafluoride Mass Measurements," Nuclear Materials Manage-

ment, Vol. VIII, No. 2, Summer, 1979, p. 33.

11. Doher, L. W., Johnsen, E. G., "Status-Implementation of ANSI N15.18 Uranium Hexafluoride Mass Measurements," Nuclear Materials Management, Vol. VIII, No. 4, 1979-80, p. 37.

12. Shuler, L. E., Doher, L. W., Morrow, L. H., "A Displacement Technique for Calibration of Special Nuclear Material Tankage Volumes," Proceedings, 18th Annual Meeting, Institute of Nuclear Materials Management, 1977, Vol. VI, No. 3, p. 472-479.

13. Hume, M. E., personal communication, March 29, 1978.

14. Ehinger, M. H., Crawford, J. M., "Measurement System Performance During Cold Checkout Runs at the BNFP," Proceedings, 19th Annual Meeting, Institute of Nuclear Materials Management, Vol. VII, No. 3, 1978 p. 475-489.

15. Ehinger, M. H., Crawford, J. M., Madeen, M. L., "Redundant Measurements for Controlling Errros," Proceedings, 20th Annual Meeting, Institute of Nuclear Materials Management, Vol. VIII, No. 3, p. 564-572.

16. Suda, S. C. (Chairman INMM 8.2), "Minutes of Meeting-INMM 8.2," personal communication, Aug. 13. 1977.

17. Schoonover, R. M., Ku, H. H., Whetstone, J. R., Houser, J. F., "Liquid Level Instrumentation in Volume Calibration," NBSIR 75-900, NBS, Dept. of Commerce, Washington, D. C. July, 1976.

18. Suda, S. C., "An Automated Electromanometer System for Accountancy Tanks," Proceedings of First Annual Symposium on Safeguards and Nuclear Materials Management, ESARDA, Brussels, Belgium, April 25-27, 1979, p. 325-329.

19. Harlan, R. A., "Uranium and Plutonium Assay of Crated Waste by Gamma Ray, Singles Neutron, and Slow Neutron Coincidence Counting," Proceedings of Conference on Measurement Technology for Safeguards and Material Control, November 27-30, 1979.

20. Haas, F. X., Lawless, J. L., Herren, W. E., and Hughes, M. E., "Measurement of Plutonium and Americium in Molten Salt Residues," Proceedings of Conference Measurement Technology for Safeguards and Material Control, November 27-30, 1979.

21. Gerald, K. B., personal communication, August 31, 1979.

22. Tento, S. W., personal communication, September 1975.

23. Gerald, K. B. and Doher, L. W., personal communication, April 12, 1979.

24. Jaech, J. L., "Statistical Methods in Nuclear Material Control," Technical Information Office, Office of Informational Services, Dept. of Energy, 1973, p. 152.

25. Lucas, L. L., Mann, W. B., (editors) "The Half Life of Plutonium-239", The International Journal of Applied Radiation and Isotopes, Vol. 29, No. 8, August 1978.

26. Prindale, A., Evans, J., Dupzyk, R., Nagle, R., Newburgy, R., "Status of the Half-Lives of Plutonium Isotopes," DOE Plutonium Half-Life Committee Status Report, personal communication, November 28, 1978.