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Primary Standards Laboratory Report 1st Half 1989

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Abstract

Sandia National Laboratories operates the Primary Standards Laboratory (PSL) for the Department of Energy, Albuquerque Operations Office (DOE/AL). This report summarizes metrology activities that received emphasis in the first half of 1989 and provides information pertinent to the operation of the DOE/AL system-wide Standards and Calibration Program.

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Foreword

This report was compiled and edited by W. G. T. Levy, Sandia National Laboratories. Additional information on the activities covered in this report may be obtained by contacting the Measurement Standards Department 7240, Sandia National Laboratories, Albuquerque, New Mexico 87185, Telephone (505) 844-3654.

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Primary Standards Laboratory Report 1st Half 1989

1. Introduction

The Primary Standards Laboratory (PSL) operates a system-wide primary standards and calibration metrology program for the US Department of Energy, Albuquerque Operations Office (DOE/AL). The PSL mission is to develop and maintain primary standards; to calibrate electrical, physical, and radiation reference standards for member laboratories (DOE/AL integrated contractors); to conduct technical surveys and audits of these laboratories; and to recommend and implement system-wide improvements.

This report summarizes activities of the PSL for the first half of 1989 and provides information pertinent to the operation of the DOE/AL Standards and Calibration Program. Specific areas covered include development projects, calibration and special measurements, surveys and audits, and significant events. Appendixes cover certifications and reports, commercial calibration laboratories, PSL memoranda, National Bureau of Standards (NBS)/National Institute of Standards and Technology (NIST) test numbers, and a DOE standards and calibration memorandum.

2. Development Projects

Time-and-Frequency System Status

The time-and-frequency system has maintained fairly reliable operation, with the only significant recent problem being a severe reduction in transmitted power for the time codes. Although the time code remained operational, errors sometimes occurred. The transmitter was repaired, and normal operation resumed. After replacing a defective time-code generator, we obtained a backup cesium-beam frequency standard from another SNL organization. This standard was serviced by the manufacturer and is now in operation in the PSL.

The fundamental comparison for maintaining the time-and-frequency system is through the NIST (Boulder, CO) Frequency Measuring System (FMS), which uses LORAN-C to compare SNL frequency standards with those of NIST. The FMS provides a daily chart of the short-term performance of the connected frequency standards; NIST provides the PSL with a monthly report of the operation of FMS-compared frequency standard. While this very effectively documents the frequency standard performance, the years of continuous operation produce, literally, reams of paper. NIST has introduced, and the PSL has acquired, a Frequency Analysis System (FAS) that stores on hard disk the digital data acquired by the FMS and allows future selection of all or part of the stored data for recall and analysis of a particular frequency standard. All the data for the PSL frequency standards stored from July 1985 have been entered into the FAS and can be recalled for viewing or analysis.

High-Voltage Pulse Generator Restored

The PSL 350-kV pulse generator was restored. Portions of the operating program need more work, including data acquisition, comparison with historical data, and certificate preparation. Minor oil leaks still plague the system, and we will try to replace the pipe fittings that seem to be the sources of these leaks. Also, the oil-transfer pump provides much less pumping capacity than expected, possibly because of cavitation or air leaks at the suspect pipe fittings. If the problems persist, we will replace the present centrifugal pump with a positive displacement unit. We have also had problems with triggering of the thyratrons that control the pulse generation, possibly because of aging that occurred in components that were not replaced.

Transient Waveform Digitizers (TWDs)

We ordered a fast-rise-time (45 ps) pulse generator to complement the equipment available for use in checking the operation of TWDs. This instrument can provide a 10-V fast-rise-time pulse to loads of 50 Ω,

with a repetition rate from a single pulse to 1 MHz. While this is a restricted-pulse generator, it will provide present state-of-the-art transition time stimuli to TWDs.

Josephson Voltage Standard Developed

We developed an ultra-high-accuracy dc voltage calibration system that uses a series array of Josephson junctions fabricated by NBS (now NIST) in Boulder, CO. The PSL designed and extensively tested the cryoprobe that supports this array of Josephson junctions. It features very low heat loss to the liquid helium bath, and cryogenic filtering on the precision voltage measurement leads. Both features are substantial improvements over the conventional design. This PSL system was compared against the NIST Josephson system used to maintain the US Legal Volt; both systems displayed the same noise level. The two sytems agreed to within 0.025 ppm, with the NIST measurements falling slightly higher. The manual version of this PSL system is now being used to calibrate dc solid-state voltage standards (Zeners) with an uncertainty of 0.1 ppm, at a confidence level of $>99.7\%$.

Development of the first fully automated Josephson voltage system continues. It will feature a dewar with a built-in liquid-helium refrigerator that will be computer-monitored and -controlled. When used with an automated measurement system similar to that under development at NIST, this combined system should require no operator intervention aside from scheduled semiannual maintenance.

Primary Pb Probe Calibration

The first primary Pb probe calibrations since replacement of the ion implantation accelerator were carried out in May 1989. The new accelerator's beam-alignment system made data collection much easier, and operation to >300 keV was possible. New beam deflector plates, designed to switch the beam in $\cong 10\ \mu s$, were slower than intended, but measurement of the time response together with calculations gave only a 0.6% correction to the calibration factor. A new power supply for the beam deflector is being tested and should eliminate the slow response. Old data collection instrumentation was used during the calibration to complete a history check; now we are developing new instrumentation for use on the next calibration.

Pb Probe Secondary Calibration Under Computer Control

Secondary calibrations of Pb probe neutron detectors are now automated. The data collection program prompts the operator through the necessary steps, collects and stores the data, and does a rough analysis after each data set so that the operator can check for proper behavior of the neutron generator. Data analysis is completed using a second program to analyze, summarize, and re-store the data in a more convenient format. A third program prints certificates and stickers. Although calibration uncertainty was reduced only slightly, the increase in efficiency and operator convenience is significant. Further, data analysis is now done on PSL computers rather than on Sandia's time-share system, which was removed from service June 1, 1989.

AC/DC Precision Calibration System (PCS) In Use

The PCS built for SNL, Albuquerque, by NIST, Gaithersburg, MD, was delivered during FY 88 and is now in service. This sytem, designated the PCS-II, was designed to have the capability to calibrate precision ac sources or precision ac measuring instruments to the same accuracy levels as achievable at NIST. To do this, it must have standards equal to those used at NIST and the same data analysis capabilities. Implementation of PCS-II at the PSL was delayed by software problems we found; these were corrected by NIST. Unfortunately, problems still exist that must be investigated and corrected before complete operation is attained. Despite these problems, several functions of the PCS-II were proven and are in use. A second version of the PCS-II is being assembled by NIST for the PSL that will have a wider operating range for both generating and measuring voltage and current.

Low-Frequency Automatic Network Analyzer (LFANA) Restored

The LFANA is being restored to state-of-the-art performance. New instruments for the system are being evaluated and will be placed in service during 1989. Test results so far show significant improvements over the original units. For example, the new LCR meter has comparison capabilities on inductance measurements to picohenries as compared to the nanohenry resolution of the original instrument. The new instruments also use synthesized (not

crystal-controlled) frequencies to allow testing at hundreds of frequencies over the range rather than the original 12 frequencies.

Software Development

Four calibration data tables were set up in the recall system database, and the calibration data input programs were written. The calibration data tables were for pressure measurements, standard platinum resistance thermometers, noble-metal thermocouples, and tungsten-strip lamps. Past data for standard platinum resistance thermometers, noble-metal thermocouples, and tungsten-strip lamps were loaded from the temperature laboratory computer. All the calibration data input programs use a common design, which will also be used for all subsequent calibration data input programs. These programs and data tables will serve as the foundation for the calibration history database.

The following group of programs was written in FORTRAN for use on the LSI-11 computers in the radiation laboratory. Program PBCALB was written for real-time data acquisition of lead probe calibration data. Program ALPSET was written to make preliminary preparations for the measurement of alpha-particle emission rates, and Program ALPACQ was written for measuring alpha-particle emission rates. We completed Program PBCALC to perform calculations on lead-probe calibration data and print the results.

Development of a High-G Mechanical Shock Calibration Standard at the NIST

A contract has been initiated to provide for the development of a high-g mechanical shock calibration standard at the NIST. The system will permit calibration of accelerometers and other transducers used for mechanical shock measurements up to 100,000 g.

Sandia is funding the project at the 50% level, with remaining funds supplied by NIST and other industrial laboratories.

The development program is to be completed in three phases; (1) feasibility study, (2) construction of a prototype, and (3) fabrication and delivery of a completed shock standard to the PSL. Completion date is estimated to be March 1992.

Modification of the Dual 6-Port Network Analyzer (D6PNA)

Four new 6-port reflectometers, two with 7-mm test ports and two with Type N test ports, were completed. Operating over the range of 2 to 18 GHz, they feature a "power conservative" design that lowers source power requirements by ~3 dB, and a novel mechanical design that inexpensively provides for precise positioning and alignment of the test ports.

Two new synthesizers featuring good spectral purity and an output of at least 13 dBm over the band 10 to 18,000 MHz have been acquired. They are performing very well.

Proof-of-principle measurements have been demonstrated on a new concept for excitation of the D6PNA. Dubbed the Twin Synthesizer D6PNA (TSD6PNA), it uses two coherent synthesizers to generate the required microwave phase shifts without incurring any loss in the microwave power levels. Unfortunately, even though the synthesizers have very good spectral purity, the stability of the TSD6PNA is degraded by the remaining close-in (<10 Hz) phase noise on the synthesizers. One possible solution requires use of eight digital voltmeters (DVMs) on the TSD6PNA instead of two as at present; we hope a better solution will soon be found.

Two major programming efforts on the D6PNA are under way:

1. All data acquisition software is being rewritten in Rocky Mountain BASIC to install the HP ES/12 Vectra computer as the instrument controller. This controller will replace the 1980 vintage Fluke 1720A controller. Among many advantages afforded by the Vectra, the major one is permitting efficient transfer of instrument data directly to the departmental MicroVax II for processing. The data are acquired under BASIC control, then transferred to the VAX using the VTERM application program that operates under MS-DOS. The KERMIT file-transfer protocol is used to assure integrity of the data transfer. This system is operational and performs flawlessly.
2. Because of the demise of the CDC NOS time-sharing system, all processing software for the D6PNA is being revised/upgraded to run on the MicroVAX II. Much work remains to be done; nevertheless, we hope to complete a project review on the entire package for making thermistor mount and other one-port calibrations on the D6PNA by the end of CY 1989. Two-port calibrations will come much later, but the workload is much smaller there.

Development of HP-8902S Measuring Receiver System for Attenuation Calibrations

Largely because of the long downtime record of the Weinschel VM4-B system, an HP-8902S Measuring Receiver was procured to provide a backup facility. Two programs (running on the HP-310 computer) to certify the HP-8902 system for attenuation and reflection coefficient measurements were developed. Three more programs for making attenuation and reflection coefficient calibrations on customers' units are also complete and fully functional. The software was written so that the operator interface is very similar to that for the VM4-B system. Work is well under way on the documentation required for a project review of the system. After calibration of some required standards by the refurbished D6PNA and completion of the documentation, we anticipate a project review in the fall of 1989.

40,000-psi Ruska System Upgrade Status

The design of the refurbishment and upgrading of the 40,000-psi cross-float calibration system by Ruska has been completed. The current calibration pressure bench is being modified primarily to provide automated weight loading and unloading for the 10 26-lb weights needed to develop pressures up to 40,000 psi. Additional modifications will update other critical components in the calibration system to the current state of the art. Delivery of the modified system is scheduled for September.

Medium-Pressure Air Cross-Float Calibration System

Detailed specifications were written, and a request for quotes was issued, for an upgraded medium-pressure air cross-float calibration system. The modified system will provide for the fully automated calibration of pressure transducers in the range 0.2 through 2500 psi using highly accurate piston gage technology. The system will also allow for a semiautomated cross-float calibration of piston gages. The new system will be located on an air-clean bench.

3. Calibration and Special Measurements

Pb Probe Calibration History Analysis

Primary calibrations were carried out on the oldest lead probe neutron detector standards still in use: JD88 and JD89. Previous results indicated the possibility of a substantial linear increase in calibration factor, starting with a low result at LANL in early 1966. However, present data show the calibration factor to be approximately constant since 1970, and so the original calibration appears anomalously low. To provide a consistent history, we took the 1989 data on the same equipment used in the 1970s. This old data-gathering electronics will now be retired.

Primary calibrations use a neutron pulse of 0.1 s duration, whereas the probes are used to measure microsecond neutron bursts. To test whether saturation problems were affecting the photomultiplier, we tested JD88 by gathering on neutron production for 6.4 s (eight halflives of radioactive Pb). Calibration results differed by 1% from those using a 0.1-s neutron burst. Further, pulse height analysis of the Pb decay showed no deviation from the expected 0.8-s half-life. Therefore, at least for longer neutron exposures, photomultiplier fatigue does not appear to be a problem.

Detection of 2.5-MeV Neutrons

Arsenic activation detectors, used to measure pulses of 2.5-MeV neutrons, were calibrated by intercomparing several detectors, first using 14-MeV and then 2.5-MeV neutrons. A previously determined response ratio between the two energies could then be used to relate the As response to that of the Pb probe at 14 MeV. The resulting uncertainty of $\cong 5\%$ can be improved only by using direct accelerator calibrations that rely on the associated particle technique.

The particle beam fusion group uses Pb probes to monitor neutron output, and they expressed interest in probe response to 2.5-MeV neutrons. Tests showed the probes are $\cong$25X less responsive to 2.5-MeV neutrons than to the usual 14-MeV neutrons.

Neutron Detector Intercompared With the UK's Detectors

Two scintillation detectors were received from the UK for an intercomparison of pulsed neutron detectors and readouts. Direct comparison showed that the US and UK neutron scales differed by 39% when uncorrected for scattering. Since scattered neutrons strongly affect the UK probe, a neutron attenuation cone has been built to UK specifications to measure the absolute contribution of scattered neutrons by the PSL. Then only the difference in source attenuation seen by the US and UK probes needs to be measured before we can determine the intrinsic difference of our neutron scales.

Capacitance Measurements

The Automated Capacitance Measurement System being constructed by the NIST for the PSL is still not finished, and no realistic projection can be made of the final checkout date. A commercially available 1-kHz Capacitance, Inductance, Resistance Bridge (GR1693 RLC Digibridge) was obtained, placed in operation, calibrated, and is being used for much of the PSL capacitance calibration measurements. This instrument operates only at 1 kHz, which limits its applications, but performance has been far better than other available instruments for our application. Another commercially available impedance measuring instrument was obtained for use from 12 Hz to 200 kHz. This instrument is specified to provide primary parameter measurements, generally to ±100 ppm; it is operating well within the tolerances on NISTcalibrated reference standards. This is one of the few instruments of this type and quality produced by a major manufacturer that has connectors from the unknown (four-terminal pair) which are completely compatible with those of another major manufacturer. This arrangement allows direct comparison of measurement results made on bridges from the two manufacturers without necessary corrections for the requisite adapters between series.

Inductance Measurements

A newly available impedance measuring instrument (see capacitance discussion) was ordered, received, and placed in service in the PSL for use in parallel with the present inductance bridge to determine its suitability as a replacement instrument for calibrating Standard Inductors. Evaluation of this instrument is expected to be completed during 1989.

Initially, the instrument provided results within 50 ppm of the calibrated values from NIST for PSL Standard Inductors. Since the NIST uncertainties were ±200 ppm, the results are so far quite encouraging. The instrument can be used to provide measurement results with resolutions of better than 1 ppm.

Temperature Measurement System Comparisons

A variety of measurement systems are used by the 11 Contractor Standards Laboratories (CSLs) to measure the resistance of a standard platinum resistance thermometer (SPRT). To determine the measurement characteristics of the different systems (especially ac types), the PSL is conducting a measurement round-robin using a 25-Ω Tinsley resistor to simulate a 25-Ω SPRT. The resistor is assumed to have negligible ac-dc differences at an ac frequency of 30 and 90 Hz based on test results for two 100-Ω resistors of the same type that were tested at NIST for ac-dc differences. The 25-Ω resistor is more resistant to the mechanical shock of shipping than an SPRT. The resistor has been shipped to four CSLs, measured on 10 measurement systems, of which six different types of instruments were used, and returned to the PSL for between-laboratory measurements. To date, all CSL measurements have been dc. Currently it is at the fifth CSL for measurement.

Primary Standards Recall System

Transaction logging has been added to the recall system. Any time a record is logged, posted, or modified either by the PSL clerk, the database administrator, or automatically by the system, the transaction with the initial status, resulting status, and the transaction date is recorded in a table (PSL_TRANS). This table is perpetual. A submenu with six reports on transaction logging was added to the report menu.

The D (delete) status was changed to I (inactive) status. Records for items not submitted for times specified by individual projects after recall are automatically moved to I status as the specified time elapses.

Three reports involving records of items tested or certified between dates were modified to include scanning of the two recall history tables. These reports required a separate program to extract the data from the main recall table and the two history tables and then to build a new table with the data. Then the report writer was used to prepare the various reports from the new table.

Conversion of Software for the Microwave Power Calibration System

We have begun a massive project for converting and upgrading the software for the Microwave Power Calibration System from the 1980 vintage Fluke 1720A to the HP310 computer. Conversion of the system for intermediate level (—10 dBm to +10 dBm) power calibrations is ~90% complete. Advantages of the new software include (1) a monolithic program for the entire calibration process, from streamlined data acquisition to Deskjet certificate printing, (2) a more friendly operator interface, and (3) mass storage of data in DOS-compatible format.

Restoration of PSL's 20,000 g Mechanical Shock Calibration Standard

After more than 10 years of use, degradation of the mechanical shock calibration standard at PSL has prompted a restoration effort to return the existing standard to the original accuracy and repeatability, and, if possible, to decrease its uncertainty by employing laser interferometry for acceleration measurements. Arrangements for the restoration were made at the Physical Science Laboratory of New Mexico State University.

During restoration, mechanical shock calibration will be discontinued at the PSL. However, calibration can be performed by the Sandia Standards Laboratory with an uncertainty of ±10%, and limited to 15,000 g. The restoration is estimated to extend from July 1989 to July 1990.

New High-Humidity Standard at the PSL

The PSL has received a new humidity generating standard, Thunder Scientific Model 9000, modified and instrumented for enhanced accuracy and repeatability. Installation of the humidity standard is scheduled for completion by October 1, 1989, at which time the PSL's final approval testing will be performed.

During acceptance testing at the factory, the humidity generator demonstrated the ability to control at a constant dew point, ±0.1°C, or at a constant relative humidity, ±0.1% RH, using the HP-9122 computer control system. The humidity standard is expected to generate and control dew-point temperature from —10°C to +65°C, and relative humidity of from 5% to 95% RH. Since the new generator will provide automation for a range of temperatures and humidities, it will be used to evaluate new products and to develop a more reliable humidity sensing instrument to be used as a transfer standard for weapons manufacturing and surveillance.

Enhancements to HP-8510 and HP-8753 VANA Calibration Software

Enhancements have been made in four programs for the HP-8510 and HP-8753 Vector Automatic Network Analyzers (VANAs) that calibrate directional couplers and fixed one- and two-port devices. The enhancements minimize overall uncertainties by tailoring the interval uncertainty component to the frequency and value at each calibrated point. A decision will be made soon regarding the level of project review required before placing these enhancements in service.

Faster Certification of the Reflection Coefficient Measurements

A faster version of the program for certifying reflection coefficient measurement performance of the VM4-B has been written. It uses two standards, two "half-standards" and a Monte Carlo technique for assessing the effects of the uncertainties on those standards. The new program, CERT4B_BO, shortens the time required for certification by nearly an order of magnitude. Unfortunately, a power supply in the VM4-B failed, and we are trying to get a replacement unit. We hope to have a project review on CERT4B_BO within a couple of weeks after receipt of the power supply.

Glassware Stability Study

The PSL, in collaboration with the Analytical Chemistry group at SNL, is continuing the 2-year study of the variability of glass volumetric flasks as a function of time and use. Initial results were presented at the 1989 Pittsburgh Conference (March 1989) and at the 5th DOE Conference on Analytical Methods (March 1989 at Rocky Flats). During June 1989, a team from the PSL and the Analytical Chemistry Division traveled to Allied Signal, Kansas City Division (ASKC) to discuss the calibration of volumetric glassware. As a result of these meetings, ASKC formulated a plan for the calibration of glassware and will begin implementation soon.

New Short-Gage-Block Measuring System

The PSL recently purchased a new short-gage-block comparator system to replace an aging system. The new system uses a digital display and an RS-232 output for direct data transfer from the comparator to a computer. The old comparator will be kept in storage as a backup and will possibly be refurbished in the future.

Computer Programs Transferred

The computer programs used for data analysis and certificate/data sheet preparation in the DC Project were transferred from the Sandia network operating system (NOS), which went out of operation in June 1989, to the department MicroVax system. The transfer required recoding portions of the programs to accommodate differences in the FORTRAN and BASIC versions and the system commands used on the two systems.

Backup Temperature Monitors Installed

A relatively simple and inexpensive backup temperature monitor/power cutoff system for the constant-temperature oil baths used in the DC Project was fabricated and tested successfully. All the oil baths are now being fitted with the backup system.

DC Automatic Calibration System

An HP-9000 workstation-based computer system was ordered and will be used to operate and control the DC Project's automated calibration systems. The computer system will acquire and analyze data and will be used to write operating programs for these systems. It will also be used to maintain, update, and manage our calibration history files and to print certificates. The system will also transfer required data to the department MicroVax and will use the MicroVax to back up programs and files.

Accumulate Dump Leak Calibration System Qualification Complete

The accumulate dump leak calibration system Phase I testing was completed, and the system was reviewed and qualified for certifying leaks. Comparisons between three leak calibration systems, and verifications with the comparison leak calibration system, were used to generate the data and crosschecks necessary for qualification. The system is now being used to calibrate a variety of CSL gas leaks from 10^{-14} mol/s. The gas-leak calibration backlog should be completed sometime in July. Now that the instrument can do routine calibrations, additional operational testing will be investigated to enhance the use of the equipment and to fully use the performance functions of the magnetic sector mass spectrometer and computer-controlled manifold and data acquisition system. This testing, called Phase II testing, will be ongoing between calibrations as time permits and will concentrate first on measurements of the linearity of the mass spectrometer and associated electronics.

Low-Pressure Calibration System Delivered

A low-pressure calibration system was delivered and is undergoing performance and initial acceptance testing. The system will be used to calibrate capacitance diaphragm gages from 10^{-3} to 10^{+3} torr, and spinning-rotor gages from 10^{-6} to 10^{-4} torr using the comparison method. Because the system is fully automated, it will greatly reduce the amount of operator time required for pressure generation, pressure stabilization, data recording, and data analyses. Final testing and acceptance should be completed by early FY90.

Ultrasonic Interferometric Manometer (UIM) Testing Continues

The UIM has been in continuous operation at NIST for nearly a year. Delivery of the Sandia UIM and the Navy UIM are being coordinated with NIST and are tentatively scheduled for early FY90. Results obtained with the Sandia UIM indicate that pressure uncertainties are near the predicted values of 20 ppm. The Sandia UIM results have also been compared with two other manometers at NIST; these results show that the Sandia manometer's performance is completely equivalent to the others. Personnel from the Pressure/Vacuum/Leak project are scheduled to travel to NIST for training in the use of the UIM during August or September.

Pressure-History Database Data Entry

About half of all pressure-history data were entered into a computerized pressure-history database

that interfaces directly with the PSL computerized recall system. The pressure-history database contains all historical data on any device or class of devices calibrated in the PSL. A summary of results can then be obtained through the use of various menu-driven reports.

Safety of Leak Artifact Handling and Transportation Enhanced

Safe handling of gas-leak artifacts has been enhanced by transporting the leaks using foam-cushioned cases and, where possible, equipping the leaks with a metal vent cap/plug. The safety of pressurized-gas leaks (some have internal pressures of 1500 psia) has been an ongoing issue because of the fragile nature of the glass leak elements, even though no known catastrophic or dangerous failure has occurred. An agreement was reached with the Safety Organization at Sandia that encouraged the use of a foam-padded container when transporting the leaks, and the use of a venting cap/plug for leaks with reservoir pressures >30 psia. The cap/plug will keep personnel protected from any glass fragments in case of failure, and a small hole in the cap will allow the high-pressure gas in the leak to be vented safely. Several foam-cushioned cases have been received and are being used for internal handling of these leaks. All customer leaks are being returned with a vent cap/plug when necessary, and existing hardware allows for easy attachment.

Calibration Times Reduced for Slow Gas Leaks

Calibration times have been reduced significantly for slow leaks (10^{-13} to 10^{-14} mol/s) by taking advantage of the flexibility of the computer-operated measurements on the accumulate dump leak calibration system. The new technique requires only a single fundamental accumulate/dump measurement. The result of this measurement is then verified with multiple comparisons to another calibrated leak. Using this new technique, we reduced the calibration time for a 10^{-14} mol/s leak from ~3 months to 2 weeks with only a slight increase in the assigned uncertainty.

4. Surveys and Audits

Technical Surveys

The PSL performed three Technical Surveys of CSLs for conformance to AL Standards and Calibration Order 57XA and to good laboratory practice. All contractors were in conformance with AL Order 57XA. In addition, one Technical Survey was made of a proposed Commercial Calibration Source (CCS). Approval was given by DOE/AL based on the survey and PSL recommendation. Starting in CY88 the Technical Survey began evolving from a survey of each contractor every year to a survey every other year. By the end of CY89 this evolution will be completed.

Technical Audits

Forty-two measurement audit exercises were initiated with the CSLs/CCSs to verify dimensional, physical, and electrical measurement capabilities. These included technical audits, blind audits, and audit packages handcarried to the contractors for measurement during Technical Surveys. The blind audits were randomly selected during the surveys for measurement at the PSL and for comparison of these measurements with the CSLs' current calibration data. Forty-three reports of audit results were written, including 14 for handcarry audits that were included in Technical Survey Reports, all of which were successfully completed.

Audit Report Program

An audit report-generating computer program is being written. The program, called RPTADT, is written in VAX BASIC, which executes on the PSL MicroVAX computer and generates audit reports based on data obtained from the audit items that are periodically sent to 13 CSLs. The program uses the EQUEL language to retrieve data from several INGRES databases during program execution and required only the entry of a single audit number that uniquely identifies the audit. Input of most recent data of PSL and CSL measurement must be done by the appropriate project leader or other designated person. Procedures for doing this have yet to be formalized. Since there are many different combinations of audit items, laboratories, distribution lists, etc., the program designs the report during execution and has automatic page and table layout. To ensure program independence, we use four external data files that can easily be modified to accommodate changing information such as the names, addresses, etc. The program determines a grade based on data and associated uncertainties. Under certain conditions the program allows entering report modification text from a terminal during program execution. Currently, reports can be written for force, length, resistance, and capacitance audit devices. The modular design allows

adding other sections for other types of audit items. The final reports can be sent to several computer locations during a single program execution. Another program, DATADT, was developed to aid in updating INGRES tables that contain the measurement data associated with each different audit. A spin-off is program PTABLE2, which can generate tables from data in external data files or from terminal input.

5. Significant Events

Measurement Technology Course Taught to Standards Laboratory Personnel

A 4-day course in Measurement Technology, oriented primarily toward dc and ac measurement, was taught to standards laboratory personnel in early May. The course was taught by Dr. Andrew F. Dunn, the president of Measurements International Limited. Dr. Dunn worked at the National Research Council of Canada (the Canadian equivalent of NIST) for 35 years and was chief of electrical measurements there from 1971 to 1987. The course addressed new technology and automation, classical measurement techniques, and "tricks of the trade." It was attended by nine standards laboratory employees and one CSL employee.

The New Weapons Production PSL (WPPSL) Building

The PSL staff reviewed the Title I drawings and submitted changes and corrections to Facilities Engineering on February 9, 1989. After a review meeting with the Architect/Engineer (A/E), Facilities Engineering authorized them to proceed with Title II. Title II was completed in July. One of the open questions is the use of an integrated shield to protect the laboratories from electromagnetic interference. The A/E will contact a consultant for recommendations.

A list of required equipment was provided to Facilities Engineering in May. Procurement of long-lead-time items may begin soon.

We are also in the process of preparing a plan for acceptance testing of the WPPSL. The testing is intended to verify the environmental control of the building; then adjustments will be made as the building is occupied.

Presentations

S. L. Husa, "Pressure Calibration and Certification of a Quartz Pressure Transducer," ISA International Symposium, Orlando, FL, May 4, 1989.

S. L. Kupferman, S. R. Booker, and H. J. Meissner, "A Computer-Controlled 300-kV Pulse Generator," at 1989 Instrument and Measurement Techniques Conference, Washington, DC, April 25-27, 1989.

C. F. Pace, S. H. Weissman, and E. V. Thomas, "The Degree of Variation of Calibrated Volumetric Glassware in an Analytical Chemistry Laboratory,"at the Fifth DOE Conference on Analytical Methods, March 29-31, 1989. Also presented to the chemistry and standards lab meeting on the calibration of volumetric glassware at ASKC, June 14, 1989.

S. H. Weissman, C. F. Pace, and E. V. Thomas, "Calibration of Volumetric Flasks—Variation With Use in an Analytical Chemistry Laboratory", presented at the 1989 Pittsburgh Conference, March 6-9, 1989.

APPENDIX A
Certificates and Reports Issued

The certificates and reports issued by the PSL are summarized in this appendix.

Reference Standards for

Allied Signal, Inc., Kansas City Division		80
Albuquerque Microelectronics Operations		14
EG&G, Energy Measurements Inc. Las Vegas, NV		17
EG&G Idaho Inc., Idaho Falls, ID		23
General Electric Neutron Devices Department		42
Lawrence Livermore National Laboratory		8
Los Alamos National Laboratory		55
Mason and Hanger		59
Mound Facility		55
Martin Marietta Energy System Inc.; Oak Ridge Y-12 Plant		5
Rockwell International Rock Flats		27
Sandia National Laboratories, Albuquerque		152
Sandia National Laboratories, Livermore		20
Savannah River Laboratory		6
Westinghouse Hanford		4
	Subtotal	567
Other Reference Standards and Delegated Instruments		282
Calibration to Support Audits & Surveys		32
Special Measurements		10
	Subtotal	324
	Total	891
Total Active Items Currently in Recall System		6647

APPENDIX B
Commercial Calibration Laboratories

Auxiliary Calibration Source Clarification

AL Order 57XA provides for two general types of auxiliary calibration sources to aid the DOE Contractor Standards Laboratories (CSLs) in implementing their calibration programs, i.e., other than the NIST, the PSL, or the CSLs. These are Commercial Calibration Sources (CCSs) approved for calibrating acceptance equipment, and Designated Commercial Standards Laboratories (DCSLs). As explicitly stated for CCSs, measurements for acceptance may be made on equipment calibrated by them, whereas measurements made with equipment calibrated by DCSLs are in-process type measurements. A table summarizing the attributes of CCSs and DCSLs is included.

DOE/AL-Approved Calibration Sources Other Than NIST, PSL, and DOE Integrated Contractors (CSLs)

(CCS) – Commercial Calibration Source: Approved for Calibrating Acceptance Equipment
(DCSL) – Designated Commercial Standards Laboratory: Approved for Calibrating In-Process Measuring and/or Test Equipment

	Attribute Summary	
	CCS	DCSL
AL Order 57XA(3/31/86) Reference Paragraphs	4.g,5.a.(3),5.c.(2) 6.a.(4)(8)(10),6.b.(5)(d),6.b.(14), 7.a.(3),7.b.(2)(3)	4.k,5.c.(4), 6.a.(4),6.b.(5)(e),6.b.(14), 7.a.(3),7.b.(2)(3)
Can calibrate in-process measuring or test equipment	Yes	Yes
Can calibrate acceptance equipment	Yes	No
Initially certified by*	PSL	CSL
Recertified by*	CSL	CSL
Survey interval	2 Years	2 Years
Monitored by	PSL	PSL
Can calibrate any device	No**	No**

*AL Order 57XA provides for initial approval by the PSL, with the reapproval to be accomplished by the principal using CSL.

**Approved calibation parameters defined at time of initial approval and updated where necessary after each survey. Contact W. G. T. Levy when use of a CCS or DCSL is considered for the first time or when new or extensions of existing calibration parameters are contemplated.

CCSs

The following is a current list of the approved Commercial Calibration Sources authorized to calibrate acceptance equipment:

Source	Expiration Date
General Electric Company Integrated Communications Services Operations Compton, CA	*
Honeywell, Ordnance Div. Hopkins, MN	*
Honeywell, Electronic Test Center New Hope, MN	*
Honeywell, Avionics Div. Ridgeway, MN	6/89*
NM Department of Agriculture Standards & Consumer Services Division Las Cruces, NM	*
Rockwell International Tulsa, OK	11/90
Sierra Instruments Carmel Valley, CA	12/90
SIMCO Electronics Co. Santa Clara, CA	12/89
Teledyne Systems Co. Northridge, CA	8/91
ATEC Engineering** Brea, CA	8/91

Use of these sources, or information update should be coordinated with W. G. T. Levy, Surveys and Audits Representative, (505) 844-3654.

* Reapproval in process

** Formerly Sheffield Measurement Division Warner Swasey Company

DCSLs

The following is a current list of the approved Designated Commercial Calibration Laboratories in use by the CSLs shown:

Certifying Contractor	Expiration Date
Allied Signal	
American Electronics Labs Montgomeryville, PA	10/89
Boonton Electronics Parsippany, NJ	5/90
C. P. D. Engineering, Inc. Oconto, WI	9/91
Delta Electronics Laboratory Orlando, FL	3/90
Endevco Corp. San Juan Capistrano, CA	7/90
Eppley Laboratory, Inc. Newport, RI	5/90
ESSCO Standards Laboratory Woburn, MA	6/91
GenRad Co. West Concord, MA	6/91
Hayes Instrument Services Billerica, MA	6/91
Hewlett-Packard Co. Palo Alto, CA	1/90
Honeywell, Avionics Div. Minneapolis, MN	10/89
John Fluke Co. Everett, WA	7/90
Leeds & Northrup Co. North Wales, PA	10/89

Certifying Contractor	Expiration Date
Allied Signal	
Lockheed Electronics Plainfield, NJ	5/90
Lockheed Missile & Space Sunnyvale, CA	1/90
Rosemount Engineering Minneapolis, MN	10/89
SIMCO * Burbank, CA	8/89
SIMCO Electronics Co. Santa Clara, CA	12/90
Teledyne System Northridge, CA	8/91
Viking Labs/Honeywell Mountain View, CA	5/91

* Formerly Comtel Metrology

APPENDIX C
Listing of PSL Memoranda (PSLM)

Over the years the PSL has issued various memoranda that provide general technical requirements and standards of good practice for the CSLs. These memoranda are listed here by number. A review for possible update, addition and/or obsoletion of these PSLM is continually under way at the PSL. A copy of the latest index of the PSLM will appear in each semiannual report. The current issue reflected herein is Rev. 5 dated 7/19/89.

PSLM No.*	Date	Title
1A	7/09/80	Exceptions to Recall Requirements
2	1/14/70	(standard leak terminations)**
3B	5/16/88	Laboratory Environments
5E***	7/19/89	Calibration Obtained from Other than the Primary Standards Laboratory
11A	3/06/72	Gage Block Materials
13B	11/20/86	Packaging Requirements for Shipment of Standards
14	11/20/74	Designated Commercial Standards Laboratories
20B	1/30/89	Rating and/or Reporting for Technical Surveys and Measurement Audits
23	6/05/80	NBS Calibrated Standards
24A	7/18/86	Weights to be Calibrated by the PSL
26	3/30/83	Checking and Cross-Checking of Standards
27***	7/19/89	Reference Materials

* Missing PSLM numbers were obsoleted in this or earlier revisions.
** Denotes the subject matter of the PSLM since no official title was given when written in 1970.
*** PSLMs still in force affected in this revision (PSLMs 5E and 27, together, replace PSLM 5D, Calibration Service Not Available from the Primary Laboratory, PSLM 15, Cross Utilization of Contractor Standards Laboratories, and PSLM 22, Calibration Below Reference Standard Level).

APPENDIX D
NBS/NIST Test Numbers

Following is a list of some of the presently valid NBS/NIST test numbers assigned to reports of tests performed for the PSL, including some of the more frequently used standards. However, where traceability is questioned by first-tier suppliers and others outside the DOE/AL Weapons Complex, it is the position of both the DOE/AL and PSL that the memorandum on traceability dated November 26, 1986, written with the concurrence of the DOE/AL, be used to answer such questions. A copy of this memorandum along with DOE's cover memorandum and transmittal letter are reprinted herein for ready reference.

Standard	NBS/NIST Test No.
DC Resistance	243183
DC Voltage	243273
DC High Voltage	728/243576-89
High-Voltage Pulse	722/228883
Potential Transformer	722/228500
AC Current Transformer Tester Set (60 Hz)	722/228500
AC Potential Transformer Tester Set (60 Hz)	722/228500
Gage Blocks, Long English	738/238897-87
Gage Blocks, Long Metric	731/238897-87
Gage Blocks, Angle	731/241753-88
End Standards	731/239286-87
Length (Threadwires)	731/241485-88
Length, Metric (Threadwires)	731/241134-88
Master Balls	738/235826
Master Metric Balls	731/239111-87
Force (Proving Rings)	
5000 lbf	737.04/237388
10,000 lbf	737.04/236524
20,000 lbf	732.07/241956
60,000 lbf	737.04/237155
100,000 lbf	737.04/233240
Pressure (Ruska-F/N 4174A)	P-8032
Pressure, Oil (Ruska-F/N 5327A)	P-8288
Pressure, Air (Ruska-F/N 2409E)	P-8196
Ion Gage 6508H	241647
SRG 6507H	241647
SRG 6965H	241647
Capacitance Diaphragm Gages	
2889H	238439
3273H	238439
3092H	238439
1870H	241875
1871H	241875
1875H	241875
Accelerometer (Vibration)	732/241365-88
Lamps Tungsten Strip	534-242010-88-1

APPENDIX E
DOE/PSL Memoranda on Standards and Calibration Program
(These memoranda are still in effect)

DOE F 1325.8

United States Government — Department of Energy

memorandum

Albuquerque Operations Office

DATE: NOV 26 1986

REPLY TO ATTN OF: PPB:QED:PEO (183)

SUBJECT: AL Standards and Calibration Program

TO: Those on Attached List

There have been some inquiries of AL contractors from other federal offices and contractors concerning how traceability of calibrated items is accomplished at their facilities. In response to these questions, the Primary Standards Laboratory has prepared a memo describing how traceability is accomplished throughout the AL complex, and this office has prepared a memo identifying each contractor facility participating in the AL calibration system. These memos are attached and should be provided to your contractor(s) for answering future inquiries.

Stephen J. Guidice
Director
Quality Engineering Division

Attachments

DOE F 1325.8

United States Government **Department of Energy**

memorandum

Albuquerque Operations Office

DATE: NOV 26 1986

REPLY TO ATTN OF: PPB:QED:PEO (178)

SUBJECT: AL Standards and Calibration Program: Traceability

TO:

One of the responsibilities of the Albuquerque Operations Office is to insure the accuracy of test and measurement equipment used for weapons design, development, and production throughout the AL complex. To assist us in meeting this responsibility, AL has tasked the Primary Standards Laboratory (PSL) at Sandia National Laboratories, Albuquerque, to provide calibration traceability for each of the following contractors:

Allied Bendix Aerospace, Bendix Kansas City Division
General Electric Neutron Devices Department, Pinellas Plant
Los Alamos National Laboratory
Mason & Hanger Silas-Mason Co., Inc., Pantex Plant
Monsanto Research Corporation, Mound
Rockwell International, Rocky Flats Plant
Sandia National Laboratories, Albuquerque
Sandia National Laboratories, Livermore
EG&G Idaho, Inc.
EG&G Energy Measurements, Inc.
Reynolds Electrical & Engineers Co., Inc.
Martin Marietta Energy Systems, Inc., Y-12 Plant
Westinghouse Hanford
E. I. duPont de Nemours & Company Inc., Savannah River Laboratory.

Attached is a memo prepared by the PSL which contains excerpts from AL Order 57XA, "Standards and Calibration Program". It describes how traceability is achieved throughout the AL complex. If you have any further questions concerning this matter, please contact this office.

Stephen J. Guidice
Director
Quality Engineering Division

Attachment

PRIMARY STANDARDS LABORATORY

Sandia National Laboratories, Albuquerque, New Mexico 87185

November 12, 1986

TO WHOM IT MAY CONCERN:

The Department of Energy has the overall responsibility for all phases of the design and production of nuclear weapons, and it has placed the specific responsibility for these activities with its Albuquerque Operations Office, DOE/AL.

Considering the nature of these nuclear devices, the reliability requirement is extremely high. All aspects of Quality Assurance are given high priority by DOE/AL in carrying out its responsibilities for the nuclear weapons program. One of the particularly important quality elements is the assurance that test and measuring equipment used throughout all phases of the program is providing proper measurements.

One of the documents relating to the requirements for the prime contractors engaged in the various phases of the nuclear weapons program, one which addresses in substantial detail the means for assuring that proper measurement results are being obtained, is AL Order 57XA, "Standards and Calibration Program", dated March 31, 1986. This Order provides for a Primary Standards Laboratory (PSL) and reference level calibration laboratories at each of the prime contractors, i.e., Contractor Standards Laboratories (CSLs) and states the requirements of the PSL and CSLs, as well as DOE/AL, their Area Offices, and other DOE Operations Offices, in the implementation of the standards and calibration program to insure the accuracy of measurements made for the nuclear weapons program.

The purpose and scope of AL Order 57XA as stated therein is as follows:

1. Purpose. The purpose of this order is to set forth the requirements of, and assign responsibilities related to, the AL Standards and Calibration Program. This program provides for a standards and calibration system to insure the accuracy of instruments, gages, and testers used in the pre-production, production, and stockpile evaluation of weapons material throughout the AL weapons design and production system.

3. Scope. This order sets forth the minimum quality requirements for DOE contractors and DOE offices engaged in the calibration of measurement and test equipment used in the design, production, and evaluation of weapons unless specifically exempted by AL in writing.

One of the most important aspects of the AL Standards and Calibration Program is "Traceability", which is defined in paragraph 4.s. as follows:

4.s. Traceability. The ability to relate individual measurement results through an unbroken chain of comparisons to national standards maintained by the National Bureau of Standards (NBS) or to accepted values of fundamental physical constants or values

derived by the ratio-type of self-calibration techniques or those obtained using nationally accepted measurement systems.

Paragraph 5.d. of AL Order 57XA establishes Sandia National Laboratories as the Primary Standards Laboratory for the DOE/AL Weapons Complex as follows:

5.d. Sandia National Laboratories is assigned the task of providing a PSL function for the AL Standards and Calibration Program. This function shall be performed in accordance with the technical requirements (see 6.a.) contained herein.

The requirements most closely concerned with traceability for the PSL and CSLs in this regard are covered in paragraphs 6.a.(1) and (2) and 6.b.(1) and (2), respectively as follows:

6.a. Primary Standards Laboratory:

(1) develops and maintains, or specifies, primary reference standards for the weapons production system, periodically certified as being traceable per paragraph 4.s., Traceability.

(2) calibrates and/or certifies the reference standards for the AL production system contractors and approves procedures for obtaining calibration and certification from other sources; maintains documentation of the calibration/certification; and reports significant out-of-tolerance conditions to appropriate users.

6.b. DOE Contractors:

(1) maintain certified reference standards and associated laboratory equipment to properly calibrate transfer standards, working standards, and measuring equipment. Ultimate traceability shall be as defined in paragraph 4.s., Traceability. In most cases the traceability to the NBS will be through the PSL.

(2) select and maintain the calibration of transfer and working standards which are used to calibrate measuring devices. Each item calibrated shall be labeled and records of calibration shall be maintained. A supplementary label, or other documentation properly identified and dated, may be used to convey certification information when space is not available on the calibration label.

The calibrations certified by the Primary Standards Laboratory and Contractor Standards Laboratories at their respective levels are covered by the above requirements.

In accordance with AL Order 57XA, the Director of DOE/AL's Quality Engineering Division reviews and performs periodic surveys of the operation

of the PSL and the CSLs for conformance to its requirements. In addition, the PSL performs periodic technical surveys of the CSL operations and operates a technical audit measurement program for all of the CSLs per AL Order 57XA. The PSL also routinely participates in Measurement Assurance Programs (MAPs) sponsored by the NBS to insure that the continued calibration capabilities are maintained at a high point of proficiency.

Evidence of traceability is maintained on file in the Primary Standards Laboratory along with detailed documentation of each calibration. The Primary Standards Laboratory publishes a semiannual report of its major activities which includes a section on applicable National Bureau of Standards Test Numbers. Each CSL also maintains detailed evidence of traceability in their files for instruments calibrated in their laboratory.

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Department 7240

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