Version 5, March 30, 2001

e-Calibrations: Using the Internet to Deliver Calibration Services in Real Time at Lower Cost

Marc Desrosiers[a], Vitaly Nagy[a], James Puhl[a], Robert Glenn[b], Robert Densock[b], David Stieren[c], Brian Lang[d], Andreas Kamlowski[e], Diether Maier[e], Arthur Heiss[f]

[a]Ionizing Radiation Division, Physics Laboratory, National Institute of Standards and Technology, Gaithersburg, Maryland 20899, USA
[b]Information Technology Laboratory, National Institute of Standards and Technology, Gaithersburg, Maryland 20899, USA
[c]Manufacturing Engineering Laboratory, National Institute of Standards and Technology, Gaithersburg, Maryland 20899, USA
[d]Physics Department, Miami (Ohio) University, Oxford, Ohio 45056, USA
[e]EPR Division, Bruker Analytik GMBH, D-76287 Rheinstetten/Karlsruhe, Germany
[f]EPR Division, Bruker Instruments, Billerica, Massachusetts 01821, USA

Abstract

The National Institute of Standards and Technology (NIST) is expanding into a new frontier in the delivery of measurement services. The Internet will be employed to provide industry with electronic traceability to national standards. This is a radical departure from the traditional modes of traceability and presents many new challenges. The traditional mail-based calibration service relies on sending artifacts to the user, who then mails them back to NIST for evaluation. The new service will deliver calibration results to the industry customer on-demand, in real-time, at a lower cost. The calibration results can be incorporated rapidly into the production process to ensure the highest quality manufacturing. The service would provide the U.S. radiation processing industry with a direct link to the NIST calibration facilities and its expertise, and provide an interactive feedback process between industrial processing and the national measurement standard. Moreover, an Internet calibration system should contribute to the removal of measurement-related trade barriers.

Keywords

Alanine; Calibrations; Dosimetry; Electron paramagnetic resonance; Internet; Remote control

Contact

Marc F. Desrosiers, Mailstop 8460, Ionizing Radiation Division, National Institute of Standards and Technology, Gaithersburg, Maryland 20899, FAX: 301-869-7682, marc.desrosiers@nist.gov

INTRODUCTION

When the personal computer (PC) was first introduced many years ago there were metrologists who contended that the computer could not equal the quality of measurements made under human control. Despite these prejudices, the PC revolutionized metrology. We are now faced with a new revolution based on the rapid exchange of information between PC's. The growing pervasiveness of computers is changing the nature of science and commerce. New measurement systems are needed to meet these new needs. It is now possible to use the instant connectivity of the Internet to link measurements made at industrial facilities to the standards maintained at standards laboratories and/or national metrology institutes. This connectivity offers the ultimate in quality assurance and traceability. The calibration of manufacturing processes can be rapid; in some cases, where it is possible to perform measurements in-line with the process, a real-time link to the national standard can be made.

More than 200 industrial irradiators are currently operating throughout the world and approximately one-third of these are in North America. Large-scale sterilization is performed in a commercial or semi-commercial production plant operating as part of the manufacturing system. In the routine operation of a radiation processing facility, the dose measurements made in the product at regular intervals provide the facility operator and regulatory authorities with an independent quality control of the process. In some radiation processes, especially those of concern for public health and safety, the release of irradiated product for public use depends on dosimetry measurements demonstrating that the required treatment has been achieved. Thus, it is important and often required that dosimetry in radiation processing be suitably accurate and traceable to a primary standard.

The public health authority will insist on the use of "good manufacturing practice" to reduce the pre-irradiation bacterial contamination of the medical products to a minimum. At the same time, the public health authority (and/or the manufacturer) may insist that the product is not adversely affected by the radiation treatment, and therefore a maximum dose limit may have to be imposed. The operator of an irradiation facility sterilizing medical products will be required to demonstrate that all the products are irradiated to the dose defined by the public health authority. The dosimetry used has to meet certain requirements with respect to accuracy, precision and calibration, and validation of the traceability of the calibration to national or international standards. It is important to realize that establishment of accurate dosimetry provides a completely independent measure of certification of each irradiation procedure and forms the basis for the regulation of radiation processing.

Alanine dosimetry is the most accurate system available to industry for routine (daily, in-house) use and transfer calibrations (documented traceability to national standard). The dosimeters are composed of microcrystalline alanine (an amino acid) which is usually held in a polymeric binder. The mixture is made into a rod, pellet, film, or cable. The absorption of ionizing radiation induces fragmentation of the molecule to form stable free radicals (molecules containing unpaired electrons, also known as paramagnetic). Electron Paramagnetic Resonance (EPR) spectrometry is a non-destructive method sensitive to free radicals. Standardizing the EPR absorption intensities of the free radicals can be accomplished by measurement of a standard sample having a stable and relatively simple EPR signal. Recent advances in spectrometer technology and design have led to production of a small, table-top EPR spectrometer dedicated to radiation dosimetry. The spectrometer was designed to be fully capable of being controlled by a local computer as well as remotely through the Internet.

Despite these new improvements to dosimetry practices, a major issue remains — turnaround time for transfer calibrations. At present, the procedure of calibrating a source for a customer comprises sending unirradiated alanine pellets from NIST to the customer, who irradiates them with the industrial radiation source to be calibrated. The irradiated pellets are returned to NIST, the EPR signals are measured at NIST, compared with the signals from pellets irradiated with the NIST standard calibration source, and the dose values are calculated. Finally, a NIST Certificate of Calibration containing the NIST-interpolated dose values is sent to the customer. This process takes several days and considerable labor at NIST, which makes the cost of the calibration relatively high and discourages customers from frequent use of this NIST service.

Some radiation processing industrial facilities operate 24 hours per day, 365 days per year, in a just-in-time manufacturing mode. When problems arise that require resolution through transfer dosimetry, the turnaround time can be critical. Measurement errors leading to product rejection or processing stoppages can be costly. The NIST Ionizing Radiation Division has been attempting to respond to the call by industry to address this pressing need. Over the past few years, NIST and industry have hosted several workshops, from which a few non-technical solutions were attempted, but these eventually failed.

DISCUSSION

The objective is to create a system for fast remote calibration of high-dose radiation sources against the U.S. national standard gamma-radiation source using the Internet. An Internet-based system will deliver immediate calibration results to the industry customer on-demand at a lower cost. The calibration results can be incorporated rapidly into the manufacturing process to ensure the highest quality.

NIST has taken the first steps toward automating this operation on the basis of modern technologies and commercially available products. A high-quality alanine-EPR measurement system has been coupled with the connectivity of the Internet to deliver services in real time. The source calibration process will be made faster, less laborious, much cheaper and, consequently, much more accessible for present and potential customers, both nationally and internationally.

The Internet-based transfer calibration service can be described as follows. A company will subscribe to the NIST service and receive a user identification and password. The industrial sites will have dosimeters on hand and own their own EPR spectrometer to read the dosimeters. They will have 24-hour, seven-day access to the NIST server. Before the service is initiated, remote measurements of the internal EPR reference standard will establish a record of the EPR spectrometer performance. They will begin by irradiating pre-supplied dosimeters, then connect to the NIST server, initiate the Internet program and request a calibration. The NIST server will communicate with the user via the local PC and establish control of their EPR spectrometer. The NIST server will also instruct the industry technician to perform sample manipulations in a defined sequence. The raw data will be transferred and evaluated by the server and a provisional certificate of calibration will be transmitted to the customer. These calibration data can then be used immediately in the industrial process. After appropriate quality checks are made at NIST, an official certificate will be issued. This step fulfills the regulatory requirement of a signed calibration certificate, and would not slow the application of the calibration data by the customer.

There are five essential features of the *e*-calibration service: accessibility; performance verification; real-time quality monitoring; electronic certification; and low cost.

The *e*-calibration service is fully accessible because it is operated by a NIST server through the World Wide Web. Execution of the service does not require the presence of NIST staff. Therefore, it is accessible on a 24-hour, seven-day basis. Furthermore, as an Internet-based service, it offers worldwide connectivity. Foreign and domestic facilities can be calibrated identically. Since this dosimetry system has the advantage of having both routine and reference class qualities, measurements can be made in a local or remote mode. Measurements made by the NIST server are only necessary if certification of the measurement is desired. However, the server connection is useful for other features of the service (*vide infra*).

Without question, *the* most critical element of the *e*-calibration service is remote control of the industry spectrometer. NIST certification requires a level of control that demands that NIST have the exclusive ability to fully control the measurement process. To initiate an *e*-calibration, the user logs into the NIST server with a unique name and password (Figure 1). The user is asked to specify the dosimeter type and lot number, as well as irradiation parameters and other associated measurement parameters. After the user provides the general calibration information, the server program instructs the user to number the dosimeters to be measured in a specific sequence. Then, the local PC-spectrometer communication link is blocked by the NIST server to prevent user intervention. The exclusion of the local user from changing the spectrometer settings is a key component of the remote control process that is essential to the certification process. The server begins by setting the appropriate measurement parameters on the industry spectrometer. It then queries the spectrometer for the parameters to verify that they were set accurately.

The NIST server continues to communicate commands to the user to insert and remove dosimeters via the user communication link. The server initiates measurement commands to the spectrometer via the data link and receives dosimeter data and spectrometer internal reference standard data until all the dosimeters are measured.

Performance verification is achieved with a spectrometer reference standard. This is installed internally by the manufacturer. The standard cannot be adjusted or changed by the user. The internal standard is useful to correct for environmental readout influences. However, it more importantly serves as a monitor of the spectrometer performance. A check of the standard prior to the calibration process will ensure that the instrument is operating within the boundaries set by initial and periodic monitoring. The ability to independently monitor the spectrometer performance is regarded as a key element of the service.

Critical to real-time certification is real-time analysis of the measurement data. The acquired data must be evaluated online so that measurements can be repeated if necessary. Several checks based on spectrum pattern recognition are employed to ensure the measurement data is valid. Dosimeter movement or removal during the measurement can be detected and an error message is sent to the user with a request to repeat the measurement. For signals that are too weak, too strong or offscale are detected, the instrument parameters are adjusted and the measurement repeated. Similar checks are performed on the internal reference standard. The measurement of the standard should take place in the absence of a dosimeter (*i.e.*, with an empty sample chamber); if the dosimeter is present, it is automatically detected and an error message will be sent to the user to remove it and initiate a new scan. Also, the intensity of the standard is compared against a history of measurements performed on that instrument.

Upon completion of the set of dosimeter measurements, the doses are calculated and delivered electronically in a provisional certificate of calibration. Upon termination of the calibration, a message is sent to the appropriate NIST staff member. A complete set of calibration events (time stamped) along with spectra and calculated doses is available for review. These data are examined for accuracy and a final certificate is prepared and signed by the authorized NIST staff. Eventually, this final document could be exclusively electronic as well, through the use of digital signatures.

Since most of the NIST labor has been removed from the process, the cost savings are substantial. A minimum of an order of magnitude cost reduction is projected, and additional savings are included (*vide infra*). Moreover, the unit price designation for calibrations will also change. Currently, the charge is on a per dose basis; it is expected that pricing based on server time will be more appropriate.

To maintain quality and be pro-active in tracking changes that could affect future calibrations, it is anticipated that periodic automatic monitoring of the internal standard will be performed to maintain a history of the spectrometer performance. This could be done in a no-cost mode and be electronically scheduled to be done at times that are convenient for the user.

The *e*-calibration website will be designed to be a resource to industrial subscribers. It will contain utilities and reference data useful to the radiation processing industry. There is a built-in added bonus for this system to radiation processors. Typically, routine dosimeters are sent to a calibration facility for irradiation to calibrated doses to create an in-house dosimeter response function. However, since calibration response functions are already an integral part of the service, subscribers can download a NIST calibration function and use it for routine measurements. This produces an added cost savings.

One of the most important issues to address is the use of firewalls to protect local PC's and networks. Potentially, firewalls may prevent connectivity and control. We were able to overcome this obstacle in our first system test in the latter part of 2000. In this demonstration, an instrument in a conference hotel in San Diego was remotely controlled by a NIST server in Gaithersburg (through a NIST firewall) and a calibration procedure was simulated. The software resident on the NIST server performed the calibration in its entirety without any intervention by NIST staff. When complete, the system will reside on an independent, firewall-protected network. With these fundamental components now in place, additional features and safeguards are being incorporated before field testing of this technology. Several industrial partners are poised to begin collaborative trials at their facilities. These tests should foster confidence in the system prior to its inauguration as NIST's first *e*-calibration service. The implementation of this service will open a new era in traceability.

ACKNOWLEDGMENTS

The authors thank the Bruker EPR Division staff for their efforts to make this service possible. Brian Lang was funded by the National Science Foundation Research Experience for Undergraduates program (Grant #9605093).

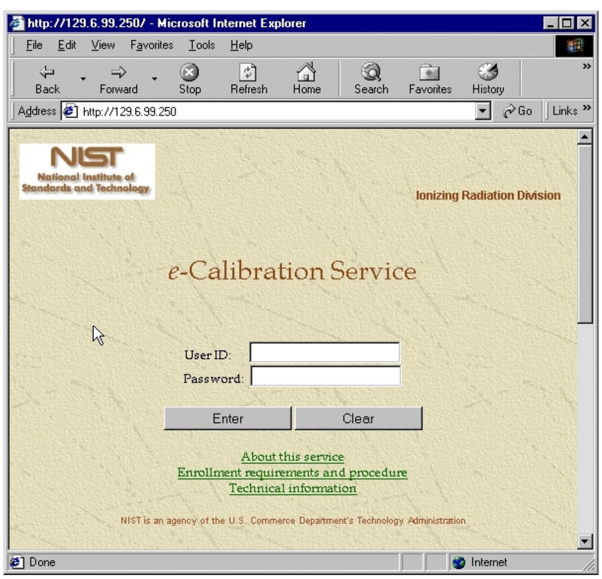

Figure 1. NIST *e*-Calibration website.