THE NEXT-GENERATION IN TRACEABILITY: *e*-CALIBRATIONS

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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 provides an interactive feedback process between industrial processing and the national measurement standard. Moreover, an Internet calibration system offers global measurement harmonization that will remove measurement-related trade barriers.

INTRODUCTION

Many years ago the personal computer (PC) revolutionized metrology, despite the claims of some metrologists who contended that the computer could not equal the quality of measurements made under human control. The growing pervasiveness of computers in our society and the world is changing the nature of commerce. New measurement systems are needed to meet these new needs. Here, we contend that the next revolution in metrology is taking place. 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.

Recently, the world's first customer-based Internet calibration (*e*-calibration) service was created and successfully tested for the radiation processing industry. An overview of the industry and its measurement issues is given to provide perspective for the evolution of the service. The essential elements of this service are described here in detail so that they may serve as a template for the modernization of other metrology services.

Radiation Processing

Large-scale ionizing radiation sources, particularly accelerator electron beams and radionuclide (gamma-ray) sources, are now widely used by a number of processing industries. This is an industry that operates in the tens-of-billions of dollars per year, and has a large growth potential. This technology has been applied to the areas of medical device and pharmaceutical sterilization, as well as the curing of materials and coatings. An even larger market looms on the horizon, the irradiation of food. With many clearances in place and a growing public demand for a safer food supply, application of this rigorously tested technology seems inevitable.

Radiation sterilization of medical products is one of the most widespread and successful applications of radiation processing. It is based on the ability of ionizing radiation to kill pathogenic microorganisms. The relationship between absorbed radiation dose and the death of microorganisms is well characterized and predictable. Radiation sterilization developed rapidly and is applied currently to a broad range of disposable medical products (syringes, surgical sutures and utensils, implant materials and tissues, *etc.*). Sterilization is carried out by ^{60}Co gamma irradiation and, using a variety of electron accelerators, by electron-beam irradiation. Sterilization by irradiation comprises about 50% of the market share for sterilization; this represents an increase of nearly double that of 1990.

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 a 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.

Radiation Metrology (Dosimetry)

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 has led to production of a small, table-top EPR spectrometer dedicated to radiation dosimetry. The spectrometer was designed to be controlled through an ethernet card and is fully capable of being controlled by a local computer as well as remotely through the Internet.

THE PROBLEM

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 (Figure 1). 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.

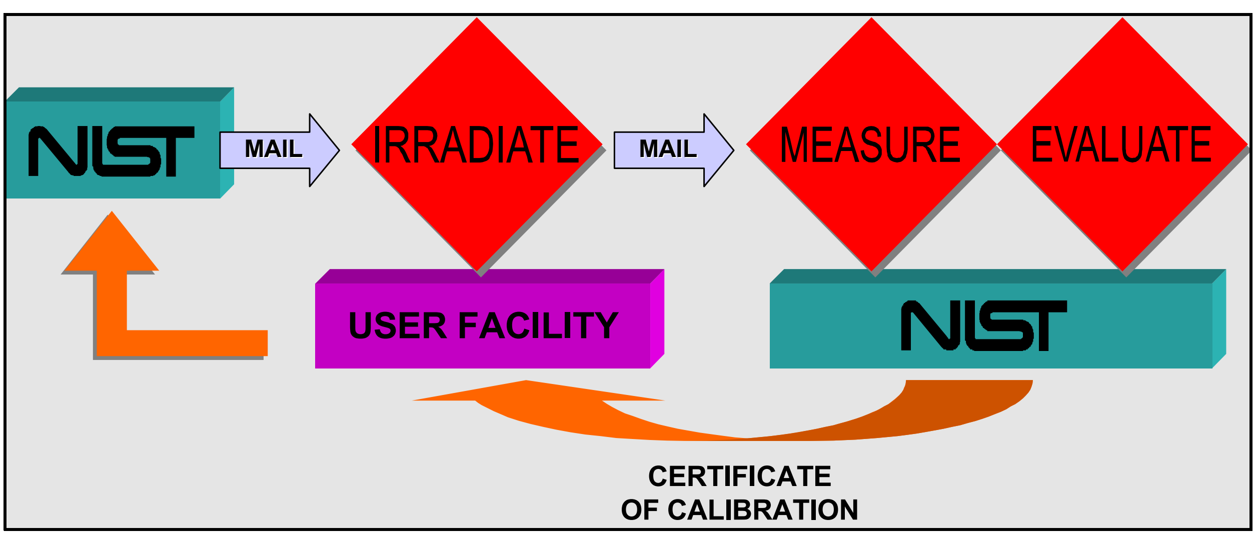

Figure 1. Traditional mail-based transfer calibration service.

The radiation processing industry operates its facilities 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.

THE SOLUTION

An Electronic Calibration Service

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 (Figure 2). 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.

Internet calibrations will provide industry with on-demand calibrations, immediate turnaround times, lower cost, and improve the quality of the manufacturing process. NIST would be able to respond to the growing need for industrial radiation calibrations while using less staff time to service it.

Attributes of the *e*-Calibration Service

Accessibility

The *e*-calibration service is operated by a NIST server and its execution does not require the presence of NIST staff (Figure 3). Therefore, it is accessible on a 24-hour, seven-day basis. Furthermore, as an Internet-based service, it offers worldwide connectivity. International companies can be calibrated identically.

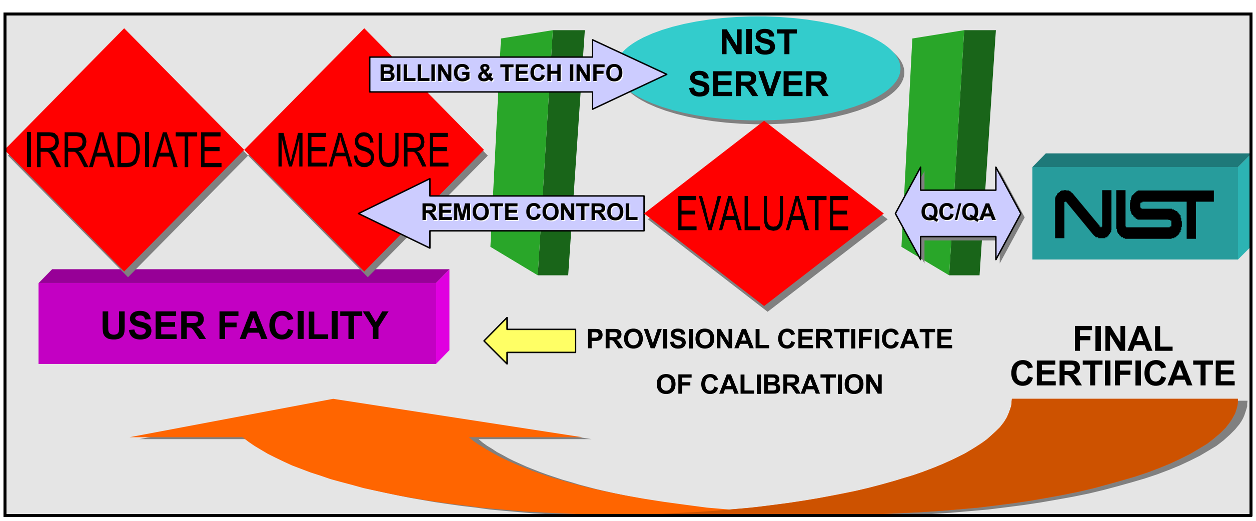

Figure 2. Internet-based transfer calibration service.

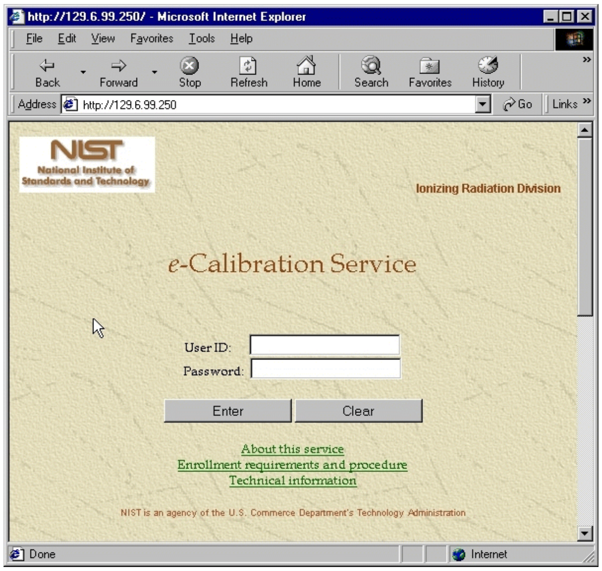

Figure 3. NIST *e*-Calibration website.

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 (Figure 4). 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*).

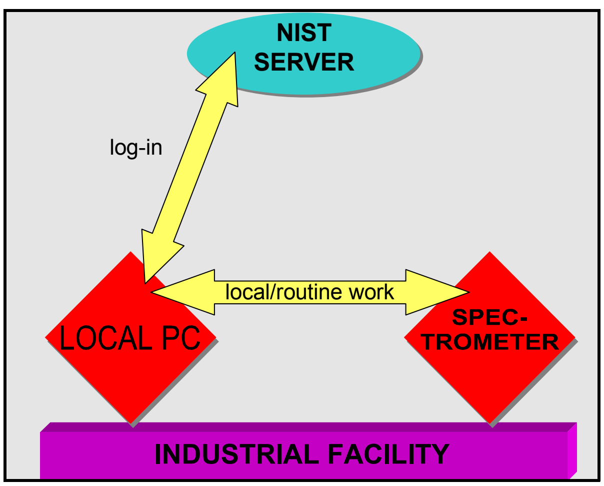

Figure 4. Operational scheme for measurements.

Remote Control

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, in our opinion, 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. 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 (Figure 5). 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 and spectrometer internal reference standard data until all the dosimeters are measured.

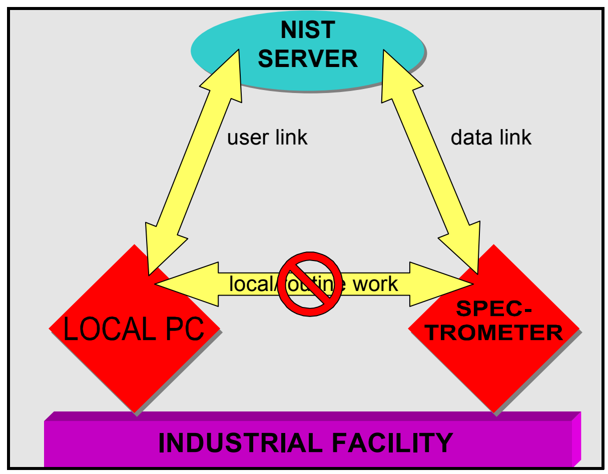

Figure 5. Communication links for *e*-calibrations.

Performance Verification

The spectrometer reference standard is installed internally by the manufacturer. It 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 critical element of the service.

Real-Time Quality Monitoring

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 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.

Electronic Certification

Upon completion of the set of dosimeter measurements, the doses are calculated and displayed in a provisional certificate of calibration. This certificate is sufficient documentation to continue the manufacturing process. After the calibration is complete, a message is sent to the responsible NIST staff. A complete set of calibration events (time stamped) along with spectra and calculated doses is available for review. These data are reviewed for accuracy and a final certificate is prepared and signed by the authorized NIST staff. Eventually, this document could be electronic as well, through the use of digital signatures.

Low Cost

Since most of the NIST labor has been removed from the equation, the cost savings are substantial. A minimum of an order of magnitude reduction in cost is projected, and additional savings are included (*vide infra*). Moreover, the unit price designation 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.

Added Features

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.

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.

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.

THE FUTURE

One of the most important issues to address next is the use of firewalls to protect local PC's and networks. Firewalls may prevent connectivity and control. However, our current solution is to create "holes" in firewalls that open specific paths between the communicating computers. This approach was used in our first successful demonstration of this service. In this demonstration, an instrument in a conference hotel in San Diego was remotely controlled by a NIST server in Gaithersburg (through the NIST firewall) and a calibration was performed without the direct involvement of NIST staff. With the fundamental features 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. Upon implementation of this service, the nature of traceability will be changed forever.