OVERVIEW

Description of Service:
GEX provides complete and formalized dosimeter batch calibration services for a wide range of applications. The service involves helping our customers to plan the calibration exercise, coordinate the materials, capture the resulting data, perform data analysis and curve fitting, and final reporting. Some dosimetry users find it easier to use GEX's calibration services which may only be performed once or twice per year. GEX calibration services conform to current recommended methods published in ISO/ASTM 51261.

Applications:
GEX dosimeter batch calibration services can be used for any gamma, electron beam, or X-ray irradiation process as well as any type of dosimeter to include: GEX B3, Alanine, CTA, FWT-60, PMMA (Perspex), GAFCHROMIC, etc.

GEX Product No.	Service Description	Includes
S1101	Dosimeter Batch Calibration Service Package	• Calibration plan which includes guidance on the development of a formal calibration protocol, with record forms • Calibration curve fitting – 5 fits of calibration data: linear, log linear, quadratic, cubic, and quartic; with "best fit" selection and analysis • GEX Calibration Report Summary • Dose Estimation "Look Up Table" • Automated GEX Dosimetry Software update, if applicable
S1102	Dosimeter Batch Calibration Audit Package	• Audit plan which includes guidance on the development of a formal protocol, with record forms • GEX Audit Report Summary • Comparative analysis of audit data and current calibration

Definition / Purpose of Dosimeter Batch Calibration:
Dosimetry batch calibration is the process where the response of a dosimetry system is characterized through comparison with a traceable reference or standard. The calibration method for the dosimetry system is designed to ensure the integrity of the measurements made with the user's dosimetry system. Routine dosimetry systems should be calibrated in accordance with the requirements of ISO/ASTM 51261.

The practice of dosimetry involves the ability to estimate dose from a measurement of change in a given material (dosimeter) to a specific irradiation dose. The dosimeter response to dose often differs from manufacturing batch to batch. Each new batch of dosimeters must be calibrated by the user to account for this response difference. Dosimeter response also varies due to factors that influence and affect the response to dose. For example, the irradiation temperature affects the response of all chemical dosimeters. Although it is possible to measure temperature and apply a temperature correction, this practice is typically reserved for well-qualified calibration laboratories, and is simply not practical for routine dosimetry applications. Therefore, *routine dosimetry systems should be calibrated under conditions that approximate their actual use* in order to effectively capture temperature effects and other influence quantities that affect the measured response of dosimeters in the calibration exercise itself.

The dose accuracy of a dosimetry system is expressed in terms of an overall combined uncertainty, which is typically expressed at a confidence interval of approximately 95%, or two standard deviations. Most dosimetry users today can state an average dose uncertainty for their dosimetry systems at approximately ±6.0% or less at k=2 (~95%) or better.

Service Completion and Delivery:
The total time for completion of a calibration after GEX receives the supplies from a customer is approximately two to three weeks. Some calibration activities may take longer - *please plan ahead and provide ample time for shipping and logistical issues.* See the Service Timeline below for an approximate delivery schedule for each stage of the calibration:

Service Timeline:

SERVICE ACTIVITY	EXPECTED TIMELINE
Calibration supplies shipping from GEX to customer:	Within one (1) business day (depending on the laboratory selected)
Calibration laboratory provides results (reported doses):	Within three (3) business days of receiving irradiated alanines from the customer (depending on the laboratory selected)
GEX provides initial report of calibration curve fitting and analysis results to customer, for review:	Within three (3) business days of receiving reported doses from the laboratory
Final report from GEX:	Approximately three (3) business days after the review of the initial report
Complete calibration	2-3 weeks (10-15 business days)

Order supplies 60 (sixty) days prior to the final calibration due date!
Based on our experience, we recommend the customer order the calibration supplies 60 days before the final calibration scheduled due date. This allows time for all the calibration activities to be completed which includes any unknown factors or problems that may occur, and allows the customer to complete their calibration by the due date.

SERVICE DETAILS

Data Preparation and Analysis:
The dosimeter response data sets are evaluated by determining the mean, standard deviation, and coefficient of variation to verify that data sets satisfy specified dose point precision requirements, and for the identification of potential outlier data prior to undertaking the curve fitting process. In addition, the data sets are plotted and evaluated to verify that the response plot is appropriate based on the expectations for a particular dosimeter and dosimetry system.

Curve Fitting:
Analysis of fit involves evaluation of the difference between values derived from the regression model against actual values. GEX provides the individual equations derived for each curve fit along with a statistical analysis of fit to the customer for review and evaluation.

GEX uses the Terastat *Calibrate!* Software platform for regression analysis (curve fitting) of dosimeter response data sets in order to derive mathematical response functions used to estimate dose from a measured response value. Response functions are generated for each set of measurement instruments and/or instrument data in combination. Terastat developed the "Calibrate!" software specifically for use in performing calibration of dosimetry systems. The software performs curve fitting and fit analysis of the calibration data sets using built-in MS Excel functions.

The *Calibrate!* Software performs up to five simultaneous curve fits of the data depending on the quantity of dose levels:

- Log-linear
- Linear (1st order)
- Quadratic (2nd Order)
- Cubic (3rd Order)
- Quartic (4th Order)

This software compares the fit analysis results of all the response functions and determines a "best fit" response function based on an internal software algorithm. It should be noted, however, that a user may request a specific regression order or GEX may choose to override the results for various reasons.

Goodness of Fit Testing:
The Calibrate! Software provides the following quantitative curve fit statistical analysis functions and displays the results for each of the generated curves:

- r-Square: Value from the regression equation used to fit the data. It can be read as the fraction of variability in the response due to changes in the standard value. For almost all calibrations, r-square will be 0.990 or greater.
- Regression F Statistic: The F value of the regression equation. A larger F statistic value indicates the model fits the data well.

- Significance of Regression: Probability that the observed F statistics may have occurred by chance. For almost all calibrations, the p-value will be near zero.
- Lack of Fit F Statistic: F-value of the lack of fit component of the residual variability. This statistic is used to evaluate whether a better fit to the data can be obtained by using a higher order polynomial. A low value typically indicates that a higher order fit is not better.
- Lack of Fit Significance: Probability that the observed lack of fit F statistic may have occurred by chance. If this value is greater than 0.05 then the software decides that it may have occurred by chance.
- MSE (mean square error): The variability remaining after fitting the regression equation. The lowest value is typically best.
- Estimated MDL at 95% confidence: Refers to "minimum descriptive length". The MDL principle is used in selecting a best model from a group of possible candidate models. The MDL algorithm takes personal preference out and uses specially determined weighting factors so the software can automatically select a so called "best fit" function from the number of response functions or curves generated (usually four) by the "Calibrate!" software. For example, it weights the F Statistic far higher than, for example, the R squared factor. The algorithm applies a set of criteria developed specifically to evaluate the sensitivity of the various response functions in order to determine the curve with the "best fit". An MDL value greater than 3.0 may indicate that that particular response function is not appropriate for estimating dose and alternative response functions should be evaluated.

Plot of Residuals Analysis:
Calibrate! Software also provides a plot of residuals that is used for qualitative analysis of the "best fit" regression model. The residuals plot is an invaluable tool used to verify the quality of fit of the selected response function. Residual plots should demonstrate generally distributed results around zero without a detectable pattern. Residual plots also provide a means to verify absence of outlier data that should have been resolved prior to fitting.

Determining the Usable Dose Range of the "Best Fit" Response Function:
Once the "best fit" curve has been determined, the Calibrate! Software is used by GEX staff to create a table of dose values covering the calibrated range derived from the calibration response function. Calibrate! Software estimates a dose for each designated response value and also generates 95% lower and upper prediction limit for each dose.

A percent uncertainty value at a 95% confidence level is calculated for each dose estimate value using these prediction limits (upper prediction limit - lower prediction limit) / (2 * dose estimate * 100). This can help the user to set the minimum and maximum allowable dose limits of the calibration based on a maximum allowable uncertainty.

Determining the Uncertainty of the Calibration Curve:
Once the calibration curve dose range limits have been established, a median or average uncertainty value can be calculated for the usable portion of the calibration range. This becomes the Type A calibration uncertainty value and can be used in determination of an overall calibration uncertainty by adding other components in quadrature. Refer to *GEX TIR# 100-209, Developing and Using Uncertainty Statements* for discussion and ISO/ASTM 51707 for guidance.

REQUIREMENTS

The customer will need to provide the following information:

1. **TYPE OF CALIBRATION**
   Reference ISO/ASTM 51261, NPL CIRM 29 and *GEX TIR #100-203, Dosimetry System Calibration* for complete requirements and guidance.

   Specify the type of calibration as one of the following:

- **Full in-plant calibration* (recommended):** Full in-plant calibration using reference transfer standard dosimeters.
- **In-plant verification audit:** Periodic verification to assure validity of the dosimeter batch calibration.
- **Laboratory calibration:** Irradiation of dosimeter samples performed at an accredited calibration laboratory with in-plant calibration verification also performed by the user.

NOTE: For customers performing a laboratory calibration, GEX will send instructions for shipping dosimeter samples to the laboratory. GEX will communicate with the laboratory on the customer's behalf to determine scheduling of irradiations.

2. CHOOSE A LABORATORY

For laboratory calibration using reference transfer standard dosimeters, select a National Standards Laboratory from one of the following:

- GEX Corporation (Colorado, USA) – NVLAP Accredited
- Risø High Dose Reference Laboratories (Denmark)
- NPL – National Physics Laboratory (England)
- NIST – National Institutes of Standards and Technology (Washington DC, USA)
- MDS Nordion (Canada) – NVLAP Accredited

For pricing, please see the GEX Pricelist or contact GEX Sales for a Customer Service Representative.

3. DOCUMENT CALIBRATION DETAILS

a. Type of facility: Gamma, Electron beam, or X-ray.
b. Energy of the radiation source (if electron beam or X-ray).
c. Material handling system (e.g. continuous, batch, rolling conveyor, hanging tote, etc.)
Different process "pathways" through the irradiator may require a separate calibration for each pathway if the dose rates, process geometry, and temperature ranges are significantly different.
d. Calibration irradiation geometry.
Identify the calibration fixture/phantoms used (e.g. Risø phantoms, ethafoam sandwich, etc.).
e. Type of dosimeter: GEX B3, FWT-60, PMMA (Perspex), GAFCHROMIC, Alanine, etc.
f. Manufacturer's dosimeter batch ID number, thickness or mass, etc.
g. Instrumentation make, model, and ID numbers.
If there is more than one instrument, separate dose response curves will be prepared for each. Data sets can be combined if requested by the user.
h. Specify measurement settings (e.g. wavelength (nm) for spectrophotometers).
i. Thickness measurement device, if any.
j. Calibration laboratory to be used.
k. Type of transfer standard dosimeter to be used.
l. Average thickness or mass to be used, or statement about specific thickness or mass measurement.
m. Average original absorbance (A_o) to be used in the response calculation.
A_o is typically not used except when calibrating FWT films.
n. Specify if an update to the customer's software file is needed.
o. Method of post-irradiation heat treatment or time control.
p. Determine if multiple time measurement records are required for time control.
The Calibration Data Workbook may require added worksheets to record the additional measurements.
q. Record the calibration irradiation procedure number (if available).
GEX recommends use of *GEX TIR #100-263, Performing a Dosimeter Batch Calibration*, which is modified for the customer's specific application and irradiation plan. Additional information is also available in *GEX TIR #100-203, Dosimetry System Calibration*.

4. SPECIFY CURVE FITTING AND DOSE ESTIMATION TABLE REQUIREMENTS

a. The required dose range in which the dosimeters will be used.
b. Record whether multiple ranges (for example, "low dose" and "high dose") are requested.
c. Specification of the irradiation target doses. See ISO/ASTM 51261 and NPL CIRM 29 for details.
d. Request for a combined data curve, if applicable.
e. Special Instructions and/or Requests. Record any additional information needed. For example, the degree of fit will be determined by GEX software; however, a customer may specify a fit type (e.g. 2rd order polynomial) or any other special instructions that should be communicated to GEX.
f. Request calculation of measurement uncertainty (reference *GEX TIR #100-209, Developing and Using Uncertainty Statements*). GEX can provide assistance creating customized uncertainty values.

5. PERFORMING THE CALIBRATION

Documents utilized for execution of the calibration:

- Calibration Procedure/Protocol (reference *GEX TIR #100-263, Performing a Dose Batch Calibration*).
- Calibration Data Workbook (QF-77-01) – Prepared by GEX and sent to the customer prior to calibration. Consists of MS Excel workbook with pre-formatted worksheets designed to provide consistent data recording.
- Technical References – as needed. See 'References' section below for the complete list.

Materials needed:

- Representative dosimeter batch samples
- Transfer-Standard dosimeters (e.g. laboratory alanine)
- Calibrated measurement instruments
- Dosimeter holder or inserts, etc.
- Dosimeter handling equipment – forceps, tweezers, PenVac.
- Temperature monitoring labels or equivalent (i.e., calorimeter, irreversible temperature labels, IR temperature gun)
- Calibration phantoms/fixtures
  NOTE: calibration phantoms should be pre-validated for use to confirm that the target doses and their associated temperatures are consistent with those specified for the calibration. Reference GEX TIR #100-263 for details.

Materials available from GEX:

GEX Part No.	Product Description	Purpose
S1000	Transfer-Standard Alanine Dosimeters	Reference dosimeter for calibrations
P1010	Large Capacity Risø Gamma Calibration Phantom Holder	Positioning of dosimeter samples during calibration
P1020	10 MeV Electron Beam Risø Calibration Phantom Holder	
P1030	5 MeV Electron Beam Risø Calibration Phantom Holder	
P8003	Irreversible Temperature Indicators 27.5°C - 65.0°C (2.5°C intervals)	Maximum calibration temperature measurement
P8005	PENVAC Dosimeter Handling Tool	Dosimeter handling
P8006	Dosimeter Handling Forceps – 4.5 inch curved	

LIMITATIONS/PRECAUTIONS

The liability for the use of calibration equations and reports is the sole responsibility of the user. GEX requires customer review of all materials, and the user must accept the final report in writing before use.

WARRANTY/GUARANTEE

Guarantee:
GEX provides a 1 year satisfaction guarantee on all products and services.

DEFINITIONS

Curve Fitting: Regression analysis of calibration dose data sets in order to derive mathematical response functions used to estimate dose from a measured response value. Response functions are generated for each set of measurement instruments and/or instrument data in combination.

Dosimeter Batch: Quantity of dosimeters made from a specific mass of material with uniform composition, fabricated in a single production run under controlled, consistent conditions, and having a unique identification code.

Dosimeter Stock: Part of a dosimeter batch held by the user.

Dose Point : Also known as a Target Dose for a calibration.

A_o : Abbreviation used for Absorbance original, i.e. original absorbance, the pre-irradiation background optical density of the dosimeter film.

A_I : Abbreviation used for Absorbance irradiated, i.e. irradiated absorbance, the post-irradiation optical density of the dosimeter film.

T or t - Abbreviation used for dosimeter film thickness.

REFERENCES

References:

- ISO/ASTM Standard 51261: Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing
- NPL CIRM Report 29: Guidelines for the Calibration of Dosimeters for Use in Radiation Processing
- ASTM Standard E170: Standard Terminology Pertaining to Radiation Measurements and Dosimetry
- ISO/ASTM Standard 51261: Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing
- ISO/ASTM Standard 51275: Practice for Use of a Radiochromic Film Dosimetry System
- ISO/ASTM Standard 51276: Practice for Use of a Polymethylmethacrylate (PMMA) Dosimetry System
- ISO/ASTM Standard 51607: Practice for Use of an Alanine-EPR Dosimetry System
- ISO/ASTM Standard 51631: Practice for Use of a Calorimetric Dosimetry System
- ISO/ASTM Standard 51607: Practice for Use of a Cellulose Acetate (CTA) Dosimetry System

Associated GEX Documents:

- QF-77-01, Calibration Data Workbook
- TIR 100-203, Dosimetry System Calibration
- TIR 100-205, B3 Radiochromic Film Dosimetry
- TIR 100-209, Developing and Using Uncertainty Statements
- TIR 100-211, Calibration of Dosimetry Systems Used in Low Energy Electron Irradiation (Energies of 300 keV or less)
- TIR 100-263, Performing a Dosimeter Batch Calibration
- WI-75-05, S1000 Order Preparation & Shipping

To learn more about GEX products and services, visit www.gexcorp.com or contact a GEX representative at +1 303 400-9640.