Environmental Technology Verification Program

Advanced Monitoring Systems Center

Quality Assurance Project Plan
for Verification of
Picometrix, LLC T-Ray 4000® Time-Domain Terahertz System

Quality Assurance Project Plan
For
Verification of
Picometrix, LLC T-Ray 4000® Time-Domain Terahertz System

February 23, 2011

Prepared by

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SECTION A
PROJECT MANAGEMENT

A1 VENDOR APPROVAL PAGE

ETV Advanced Monitoring Systems Center

Quality Assurance Project Plan for Verification of Picometrix, LLC T-Ray 4000® Time-Domain Terahertz System

February 23, 2011

APPROVAL:

Name ________________________________

Company ______________________________

Date _________________________________

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A3 ACRONYMS AND ABBREVIATIONS

ADQ	Audit of Data Quality
AMS	Advanced Monitoring Systems
ATI	Alternative Technologies Initiative
DQI	data quality indicator
EM	electromagnetic
EPA	U.S. Environmental Protection Agency
ERS	External Reference Structure
ETV	Environmental Technology Verification
IAEA	International Atomic Energy Agency
gsm	grams per square meter
LRB	laboratory record book (the test logbook)
mm	millimeter = 10^{-3} meter
OAR	Office of Air and Radiation
pdf	Adobe portable document format
ps	picosecond = 10^{-12} second
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
QMP	quality management plan
RI	Refractive Index
SOP	Standard Operating Procedure
TD-THz	Time-Domain Terahertz
THz	terahertz
ToF	Time-of-Flight
TSA	Technical Systems Audit
μW	microwatt = 10^{-6} watt
VTC	Verification Test Coordinator

A4 DISTRIBUTION LIST

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A5 VERIFICATION TEST ORGANIZATION

The verification test will be conducted under the U.S. Environmental Protection Agency (EPA) Environmental Technology Verification (ETV) Program. It will be performed by Battelle, which is managing the ETV Advanced Monitoring Systems (AMS) Center through a cooperative agreement with EPA. The scope of the AMS Center covers verification of monitoring technologies for contaminants and natural species in air, water, and soil.

The day to day operations of this verification test will be coordinated and supervised by Battelle, with the participation of the vendor (Picometrix) who will be having the performance of their technology (Picometrix, LLC T-Ray 4000® Time-Domain Terahertz System) verified. The Picometrix technology offers an alternative to using sealed radioactive source nuclear gauges. Testing will be conducted at Appleton Paper in Appleton, Wisconsin. The vendor will provide and operate their technology.

The organization chart in Figure 1 identifies the roles and communication structure of the organizations and individuals associated with the verification test. Roles and responsibilities are defined further below. Quality Assurance (QA) oversight will be provided by the Battelle Quality Manager and also by the EPA AMS Center Quality Manager, at her discretion. This verification test is a Quality Category II test which requires a QA review of 25% of the test data (see Section C1).

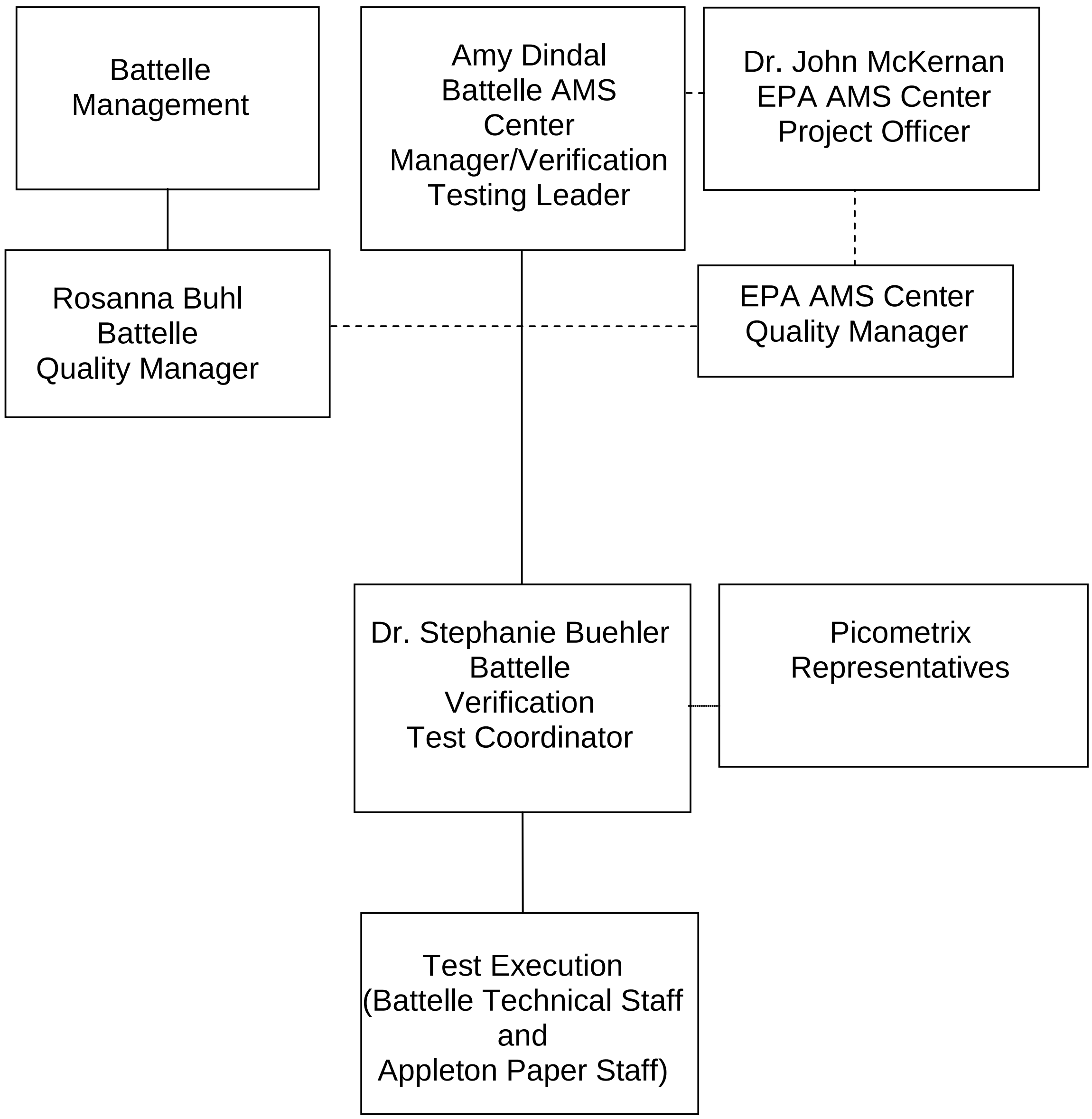


Figure 1. Organization Chart for the Verification Test (dotted lines indicate lines of communication)

A5.1 Battelle

Dr. Stephanie Buehler is the AMS Center's Verification Test Coordinator (VTC) for this test. In this role, Dr. Buehler will have overall responsibility for ensuring that the technical, schedule, and cost goals established for the verification test are met. Specifically, Dr. Buehler will:

- Prepare the quality assurance project plan (QAPP), verification report, and verification statement;
- Revise the QAPP, verification report, and verification statement in response to reviewers' comments;
- Establish a budget for the verification test and manage staff to ensure the budget is not exceeded;
- Assemble a team of qualified technical staff to conduct the verification test;
- Direct the team in performing the verification test in accordance with this QAPP;
- Hold a kick-off meeting approximately one week prior to the start of the verification test to review the critical logistical, technical, and administrative aspects of the verification test. Responsibility for each aspect of the verification test will be confirmed;
- Ensure that all quality procedures specified in this QAPP and in the AMS Center Quality Management Plan[1] (QMP) are followed;
- Serve as the primary point of contact for vendor representatives;
- Ensure that confidentiality of sensitive vendor information is maintained.
- Assist vendors as needed during verification testing;
- Become familiar with the operation and maintenance of the technologies through instruction by the vendors, if needed;
- Respond to QAPP deviations and any issues raised in assessment reports, audits, or from test staff observations, and institute corrective action as necessary; and
- Coordinate distribution of the final QAPP, verification report, and verification statement.

Ms. Amy Dindal will serve as Verification Testing Leader and is also Battelle's Manager for the AMS Center. Ms. Dindal will:

- Support Dr. Buehler in preparing the QAPP and organizing the testing;
- Review the final QAPP;
- Attend the verification test kick-off meeting;
- Review the draft and final verification report and verification statement;
- Ensure that necessary Battelle resources, including staff and facilities, are committed to the verification test;
- Ensure that confidentiality of sensitive vendor information is maintained;
- Support Dr. Buehler in responding to any issues raised in assessment reports and audits.
- Maintain communication with EPA's technical and quality managers; and
- Issue a stop work order if Battelle or EPA QA staff discover adverse findings that will compromise test results.

Battelle Technical Staff will support Dr. Buehler in planning and conducting the verification test. The responsibilities of the technical staff will be to:

- Assist in planning for the test;
- Attend the verification test kick-off meeting;
- Assist vendor staff as needed during verification testing[1];
- Conduct and observe verification testing on-site, as appropriate. Immediately report deviations to this QAPP to the VTC[1];
- Coordinate and observe reference testing on-site, as appropriate. Immediately report deviations to this QAPP to the VTC[1];
- Perform statistical calculations specified in this QAPP on the technology data as needed;
- Provide results of statistical calculations and associated discussion for the verification report as needed; and
- Support Dr. Buehler in responding to any issues raised in assessment reports and audits related to statistics and data reduction as needed.

[1] Battelle's Quality Manager will perform these activities.

Ms. Rosanna Buhl is Battelle's Quality Manager. Ms. Buhl will:

- Review the draft and final QAPP;
- Attend the verification test kick-off meeting;
- Be present throughout testing to observe tests procedures;
- Conduct a technical systems audit during the verification test, or designate other QA staff to conduct the audit;
- Audit at least 25% of the reported verification data or designate other QA staff to conduct the data audit;
- Prepare and distribute an assessment report for each audit;
- Verify implementation of any necessary corrective action;
- Request that Battelle's AMS Center Manager issue a stop work order if audits indicate that data quality is being compromised;
- Provide a summary of the QA/QC activities and results for the verification reports; and
- Review the draft and final verification report and verification statement.

A5.2 Picometrix

The responsibilities of the technology vendor are as follows:

- Provide significant input into the draft QAPP;
- Review and provide comments on the draft QAPP;
- Accept (by signature of a company representative) the final QAPP prior to test initiation;
- Provide one unit with two measurement sensors of their technology for evaluation during the verification test;
- Provide all equipment/supplies/reagents/consumables needed to operate their technology for the duration of the verification test;
- Provide an appropriately trained person to operate their technology for the duration of the verification test;
- Provide maintenance and repair support for their technology, on-site if necessary, throughout the duration of the verification test;

- Provide data from testing in an understandable format from their technology to the Battelle VTC or Battelle technical staff;
- Review and provide comments on the draft verification report and statement for their technology; and
- Provide any applicable documentation related to testing of the vendor's technology, including calibration, testing results and observations, final images, supply or reagent certificate of authenticity (COA), etc.

A5.3 EPA

EPA's responsibilities in the AMS Center are based on the requirements stated in the "Environmental Technology Verification Program Quality Management Plan" (EPA ETV QMP).[2] The roles of specific EPA staff are as follows:

Ms. Michelle Henderson (or a qualified alternate) will act as the EPA's AMS Center QA Manager. For the verification test, the EPA AMS Center QA Manager will:

- Review the draft and approve the final QAPP;
- Attend the verification kick-off meeting, as available;
- Review checklists, reports, report responses, and closure statements of TSA, performance evaluation (PE) audits, and audits of data quality systems (ADQs) conducted by Battelle;
- Perform an external TSA of field and/or laboratory activities, PE audits, and/or an audit of data quality during the verification test;
- Notify the EPA AMS Center Project Officer of the need for a stop work order if evidence indicates that data quality is being compromised;
- Prepare and distribute an assessment report summarizing results of any external audit performed;
- Review the first day of data from the verification test and provide immediate comments if concerns are identified; and
- Review the draft and approve the final verification report and verification statement.

Dr. John McKernan is EPA's Project Officer for the AMS Center. Dr. McKernan will:

- Review the draft QAPP;

- Approve the final QAPP;
- Attend the verification kick-off meeting, as available;
- Be available during the verification test to review and authorize any QAPP deviations by phone and provide the name of a delegate to the Battelle AMS Center Manager should he not be available during the testing period;
- Review the draft verification report and verification statement;
- Oversee the EPA review process for the QAPP, verification report, and verification statement;
- Coordinate the submission of the verification report and verification statement for final EPA approval; and
- Post the QAPP, verification report, and verification statement on the ETV web site.

A5.4 Nuclear Gauge and Laboratory Reference Measurements

This test will require the use of a sealed source nuclear gauge and laboratory methods based on TAPPI standards as the reference instrument. Appleton Paper (Appleton), which routinely uses nuclear gauge technology and TAPPI laboratory measurements for their business and which maintains an appropriate safety license to operate a nuclear gauge, will conduct all reference measurements. The responsibilities of Appleton include the following:

- Provide a site and associated site equipment for conducting a test at an Appleton facility;
- Conduct all field reference measurements of materials using a nuclear gauge;
- Conduct all laboratory reference measurements of materials generated during the testing process, as specified in Appleton internal protocols;
- Supply all necessary equipment to obtain reference measurements using a nuclear gauge and laboratory protocols;
- Supply a licensed technician to operate the nuclear gauge and staff to conduct the laboratory measurements;
- Provide nuclear gauge as well as laboratory measurements resulting from this verification test to Battelle, including an accompanying description or analysis of the measurements as appropriate, including any QA/QC procedures that were followed;
- Perform testing activities and data acquisition as specified in this QAPP; and

- Allow the Battelle and/or EPA Quality Managers to perform a technical systems audit at their facility.

A5.5 Verification Test Stakeholders

This QAPP and the verification report and verification statement based on testing described in this document will be reviewed by experts in the fields related to nuclear gauges and alternatives to sealed-source technologies. The following experts have been providing input to this QAPP and have agreed to provide a peer review.

- Michael Barlament, Kimberly-Clark Corporation
- Paul Thomas, 3M Company
- Temeka Taplin, U.S. DOE
- Madeleine Nawar, U.S. EPA

The responsibilities of verification test stakeholders include the following.

- Review and provide input to the QAPP;
- Review and provide input to the verification report/verification statement.

A6 BACKGROUND

A6.1 Technology Need

Radioisotopes, such as sealed sources of Strontium-90, Krypton-85 or Promethium-147, can be found in industrial devices used to measure the mass per unit area (basis weight) of various manufactured products such as sheet metal, textiles, paper, photographic film, and other pressed or sheet (flat) material. Devices widely used in industrial and commercial applications are often small in size, and could potentially be lost, stolen, abandoned, or improperly disposed. In some instances sealed radioactive sources can be replaced by an alternative non-radioactive source of energy to accomplish the same function.

Currently, nuclear gauges are being used to measure or control material density, flow, level, thickness or basis weight. The gauges contain sealed sources that radiate through the substance being measured to a readout or controlling device. The radiation that is emitted from the gauge is attenuated by the matter between the radioisotope and the detector. The radiation reduction can be correlated to the quantity of matter between the source and the detector. The nuclear radiation is attenuated by the number of atoms between the source and the detector. The physical measurement is the magnitude of radiation that passes through the material. The actual amplitude of the detected signal must be compensated for a number of factors (e.g. moisture, air in the intervening space, geometry of the source and detector); however these factors are well understood and implemented in commercial nuclear gauges.

Nuclear gauges have been widely used (since the 1940's) for the measurement of basis weight and quantities that can be derived from basis weight (e.g. thickness, coat weight) because of their ability to penetrate most materials, their long life (depending on isotope) and their low cost. At this point in time they are a well established, low risk gauge that is well known in the industry. However, the radioactive sources in these gauges can present a safety risk. Minimizing the number of radioactive sources in the public domain will decrease the opportunity for individuals to use the radioisotopes for unintended, harmful (i.e. terrorist) purposes. In an effort to do so, the EPA's Office of Radiation and Indoor Air in the Office of Air and Radiation (OAR), established the EPA's Alternative Technologies Initiative (ATI) (http://www.epa.gov/radiation/source-reduction-management/alt-technologies.html). Part of the EPA-ATI is fostering the acceptance and voluntary market adoption of non-radioactive technologies; i.e., alternative technologies to those that currently use sealed sources. The EPA-ATI is focusing primarily on alternative technologies for devices with Category 3 and 4 radioactive sources as classified by the International Atomic Energy Agency (IAEA) . Commercial-ready or available alternatives to nuclear gauges (such as infrared, laser, and other technologies) are being considered. As with any new technology, the likelihood of acceptance can be significantly increased by independent evaluation and verification of a technology's capabilities.

The purpose of this QAPP is to specify procedures for a verification test applicable to commercially available alternatives to nuclear gauges that can replace technologies using sealed radioactive sources for the measurement of basis weight, the measurement of mass per unit area of a material. The purpose of the verification test is to evaluate the performance of the Picometrix T-Ray 4000® Time-Domain Terahertz (THz) System in an actual production environment for the measurement of basis weight. In performing the verification test, Battelle will follow the technical and QA/QC procedures specified in this QAPP and will comply with the data quality requirements in the AMS Center QMP.[1] Because this verification will be referenced by the Office of Air and Radiation's Alternative Technology Initiative, it was decided to establish the testing as a Quality Category II, requiring a QA review of 25% of the test data and additional peer-reviewers.

A6.2 Technology Description

In the last twenty years, significant advancements in the THz systems signal-to-noise performance have been realized, making the deployment of such systems to research laboratories and industrial factories possible. The most commonly deployed terahertz systems are Time-Domain Terahertz (TD-THz) units. Professors Daniel Mittleman (Rice University) and Daniel Grischkowski (Oklahoma State University) have published numerous articles and books on this method. Dan Mittleman maintains a list of researchers and groups active in THz study (http://www-ece.rice.edu/~daniel/groups.html).

TD-THz systems emit and detect a very narrow (<1 picosecond [ps]) electromagnetic (EM) pulse that forms photons in the terahertz frequency range. TD-THz systems measure the electrical field strength of the EM pulse as a function of time. The TD-THz frequency range falls between microwaves and the Far-IR. One very important aspect of these frequencies is that most dielectric materials are quite transparent in the region of study with TD-THz (0.05 – 3 THz). Plastics (regardless of color), paper, textiles, dry wood, packaging materials, rubbers, foams, non-polar liquids (oils), paints (including low observable "radar absorbing") and other coatings are all very transparent. Polar liquids (water, alcohols) are all strongly absorbing over the complete TD-THz region.

The THz pulse is low energy (approximately 1 microwatt [μW]), completely safe, and can be focused, reflected and treated essentially in the same manner as any pulsed "light" source. After this pulse has interacted with matter (transmission, reflection, scatter), the changes in the pulse have lead to two methods of analysis. Spectroscopic methods of investigation are possible with THz. The Fourier transform of the time-domain data allows frequency (i.e., spectroscopic) analysis. The second common methods of analysis is to directly study the time-domain data measuring changes in the Time-of-Flight (ToF) of the pulse as it interacts with matter.

The time-domain analysis of THz pulses (ToF) has demonstrated the ability to measure the basis weight (mass per unit area) of manufactured products. This is important because the measurement of basis weight is the most common use for nuclear gauges in industry.

A laboratory basis weight measurement consists of the measurement, with an analytical balance, of the mass of a specific area of a sheet product (e.g., paper). The specific area cut from the sheet product varies depending on the product. For paper products, a common unit area is 800 square inches. Example basis weight units of measure are pounds per square yard or grams per square meter. For the paper industry, a common unit is pounds per ream, where a ream represents 3300 square feet of paper.

The THz method to measure a material's basis weight is to measure the increase of the Time-of-Flight (ToF) of the EM pulse as it transmits through the material of interest. This ToF value is calibrated against accepted values of the material's basis weight. The THz method is a time-based measurement, as opposed to the amplitude attenuation-based measurement method of nuclear gauges.

The material's ToF value is found in the following manner: when photons transmit through a material, the transit time of the photon will be increased due to the increased refractive index (RI) of the material compared to the RI of photons in air or vacuum (~1). The ratio of the velocity of photons in a vacuum to the velocity of photons in the material of interest, defines the RI for that material. Because the velocity of the EM is less in a material, the amount of time required for the EM to transmit through the material will be longer. The difference in time between the EM pulse transmitting through the material, compared to the same transmission through air, can be precisely measured with THz instrumentation. This difference is the Time-of-Flight (ToF) delay. This ToF delay (typically in ps) is calibrated against accepted values of basis weight for the sample material, which will be supplied by Appleton as a result of their laboratory measurements.

A wide range of material basis weight values (5 grams per square meter (gsm) to greater than 100,000 gsm) can be measured with this single source instrument. The terahertz system directly measures the time delay increase due to the pulse passing through the material under test. Formally this is the volume of material in the beam times the index of refraction of the material at the terahertz frequency minus 1. Not to be tested in this plan, but of interest, is the ability of THz to make simultaneous non-contact sample total and individual layer thickness measurements and percent moisture content. The total sample thickness measurement, again a time based measurement, requires the use of an External Reference Structure (ERS). Finally, the amplitude of the transmitted THz pulse does contain simultaneous complementary information regarding chemical and physical properties of the sample, as mentioned, the moisture content. In this work we distinguish between a measurement of thickness (sometimes called caliper thickness) or the physical dimension of the material under test, and the basis weight, which is the mass per unit area. A nuclear gauge measures the amount of matter between the source and detector, which is most directly converted to a basis weight. In circumstances where the material has a uniform density, the nuclear gauge measurement can also be correlated to physical thickness.

Most THz measurements are made in reflection, as this geometry simplifies the system configuration and reduces cost. Often, a fixed metal plate is installed behind the sample. The THz pulse, reflected off a rear metal plate, will have transmitted through the sample twice. This measurement mode is equivalent to double pass transmission and will have the measured ToF delay increased by a factor of two.

The use of a beam splitter in the reflection sensor allows the transmitting and reflecting THz pulses to remain collinear throughout the inspection (see Figure 2A). Therefore, the sensor operates best when aligned orthogonal to the inspection surface. However, for illustration purposes, an angle is often shown between the transmitter and receiver (see Figure 2B). This display method helps to clearly separate the incoming and reflecting THz pulses and thus better illustrates the origin and timing of the reflection pulses.

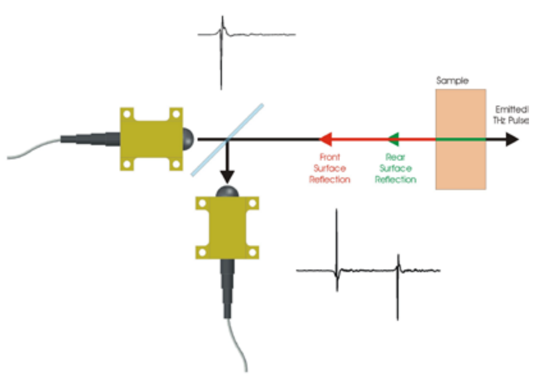


Figure 2A. Collinear THz Reflection Sensor

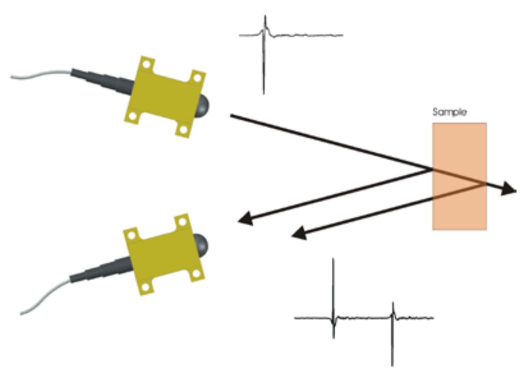


Figure 2B. Illustration of Pulse's Origins

Again, the most common use of nuclear gauges in industrial settings is the measurement of basis weight. Thus, this verification test will address the comparison of the two measurement systems only for basis weight measurements.

The fundamental method of nuclear gauges to find mass per unit area (basis weight) is to calibrate the measured attenuation of the nuclear particle flux when passing through a sample against a standard-method-determined or accepted basis weight value. If the average density of the product is known and remains constant, then the average sample thickness can be calculated from the measured basis weight value.

The fundamental measurement with a THz sensor is the delay in the ToF of the THz pulse as it passes through a sample. In a transmission measurement, the delay can be directly measured and then calibrated against basis weight, a similar process to nuclear gauges. The sample's ToF is calibrated against a standard method determined or accepted basis weight values.

A7 VERIFICATION TEST DESCRIPTION AND SCHEDULE

A7.1 Verification Test Schedule

Table 1 shows the planned schedule of testing and data analysis/reporting activities to be conducted in this verification test. As shown in Table 1, preparation to test the technology began in January 2011. Testing will be conducted over 2-3 days during February, 2011. The exact test dates will be determined the week before testing. After testing, an ETV verification report and verification statement will be drafted for the participating technology. The report will be reviewed by the technology vendor and peer reviewers that will be selected from the paper and coatings industry and the EPA. The final report and statement will be submitted to EPA for final signature, and these documents will be made publicly available on both the EPA/ETV and the Battelle AMS Center websites.

Table 1. Planned Verification Test Schedule

Month Year	Testing Activities	Data Analysis and Reporting
January 2011	▪ Coordinate with vendor representative ▪ Coordinate schedules for technologies and reference testing	▪ Begin preparation of ETV report template
February 2011	▪ Perform testing	▪ Compile data ▪ Compile testing environment conditions ▪ Collect and analyze data from reference samples
March/April 2011		▪ Analyze and finalize all data ▪ Complete common sections of reports ▪ Prepare draft report/verification statement
May 2011		▪ Internal review of draft report/verification statement ▪ Vendor review of draft report/verification statement
June 2011		▪ Revise draft report/verification statement ▪ Peer review of draft report/verification statement
July 2011		▪ Revise draft report/verification statement ▪ Submit final report/verification statement for EPA approval

A7.2 Test Sites

Testing will be conducted at Appleton Paper (Appleton) in Appleton, Wisconsin. In performing this verification test, Battelle will ensure that the vendor and Appleton staff follow the procedures specified in this QAPP and will comply with quality requirements in the AMS Center QMP.[1]

A7.3 Health and Safety

The vendor and Appleton staff will conduct all verification testing and reference measurements following the safety and health guidelines in place for the Appleton facility. This includes maintaining a safe work environment and a current awareness of radiation exposure potential. Testing involving the release of radiation will be performed by appropriately trained personnel according to the Appleton facility safety guidelines.

A8 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

The data quality objectives of this verification test are to:

- Evaluate the ability of THz sensor(s) to obtain on-line measurements of the mass per unit area (basis weight) of different weight coated paper products; Picometrix expects that the accuracy will be within 10% of an existing nuclear gauge (MeasureX company) measurement system;
- Evaluate if THz sensors are able to replace nuclear sensors in the measurement of basis weight; and
- Evaluate the ability of a THz sensor to make off-line, static measurements of the mass per unit area (basis weight) of different weight coated paper products in a controlled laboratory environment; Picometrix expects that these to be made with precision and an accuracy within 10% of laboratory measurements.

The verification of the THz performance will be compared to laboratory measurements of basis weight using TAPPI standard methods. Comparisons will also be made versus results obtained using the nuclear (beta) gauges currently used as the in-line process monitoring device at Appleton.

Data quality indicators (DQIs) ensure that this verification test provides suitable data for a robust evaluation of performance. DQIs have been established for paper basis weight measurements and the laboratory analyses that must be controlled for this performance-based measurement. The DQI for these supporting measurements are quantitatively defined in Table 2 along with the acceptance criteria. Quantitative performance parameters for vendor technology performance are discussed in Section B.

Appleton staff will operate the nuclear gauges and perform the laboratory testing; vendor staff or vendor-appointed representatives will operate the THz gauges. These staff will follow the manufacturer's recommended set-up, operations, and maintenance and any internal operating procedures to ensure that the data generated during testing are representative of gauge capabilities. Because the THz and nuclear gauges will not be operated or handled by Battelle staff, Battelle staff will be present throughout testing to observe testing and document the procedures and performance of gauges as well as the laboratory equipment used for the basis weight determination (reference method). The requirements for these observations are described in the discussion of documentation requirements and data review, verification, and validation requirements for this verification test. System model, nuclear gauge model, isotope type and current activity of the Appleton nuclear gauge will be noted.

The Battelle Quality Manager will perform a technical systems audit (TSA) of the THz, nuclear gauge, and laboratory testing activities to verify that testing and analysis were performed according to the QAPP and internal Appleton test methods. The EPA quality manager also may conduct an independent TSA, at their discretion. TSAs are described in detail in Section C1.2.

Battelle will rely on the vendor's data quality indicators for their THz technology in order to insure that the technology is performing properly during testing. The technology data quality relies on proper operation and maintenance of the THz technology. Vendor DQIs specific to this test are provided in Table 2. The results from the technology are expected to be quantitative and will be reported as basis weight and compared to measurements of paper sampled during the test for analysis at Appleton's in-house laboratory. Results from the nuclear gauges will also be compared to the THz data as well. There are realistic factors in the production environment which will limit how ideal the overlap of laboratory and gauge measurements will be. Laboratory measurements of a production sample's basis weight can only be made on samples at the end of the production roll. There is no method to perfectly synchronize the gauge measurements to the paper roll position.

Table 2. DQI and Criteria for Critical Supporting Measurements

Phase	DQI/Critical Measurement	Method of Assessment	Frequency	Acceptance Criteria	Corrective Action
Laboratory[1] Confirmation	Accuracy / Balance[2]	Certified weights	Quarterly by professional balance service and Prior to testing	NIST tolerances for analytical balances	Maintenance and recalibrate
Laboratory Confirmation	Accuracy / TAPPI Room Temperature	Thermometer	Continuously during testing	23±1.0 °C (73±1.8 °F)	Repeat measurement
Laboratory Confirmation	Accuracy / TAPPI Room Relative Humidity	Hygrometer	Continuously during testing	50%±2.0%	Perform maintenance Repeat measurement. Apply correction factor

Phase	DQI/Critical Measurement	Method of Assessment	Frequency	Acceptance Criteria	Corrective Action
Laboratory Confirmation	Hygrometer	ISO 17025 Certified Laboratory	Annually and within one week of testing	±1.0% at 23°C and 50% RH	Replace
Laboratory Confirmation	Thermometer	ISO 17025 Certified Laboratory	Annually and within one week of testing	Graduated to 0.20 °C (0.50 °F)	Replace
In-line Production	Completeness/ Amount of THz data collected per second	Time stamp of THz data stream	Each run: number of data points/ second	>99%	Investigate and correct; repeat run
In line Production	Accuracy/ THz instrument calibration	Calibration standard results vs. initial calibration	Daily Mid-day check	$\pm 20^{-15}$ sec	Investigate and correct; repeat run
In line Production	Accuracy/ Amount of accurate data collected	Review data for anomalies (> ±5% from average)	Each run	<20% of data average	Confirm data filtering; correct if necessary; repeat run
Off-sheet Production	Accuracy/ mass in chamber	Off-sheet check of optics	Before and after each run	$< \pm 50^{-15}$ sec difference between readings	Investigate for sensor assembly damage; if none, use new value

[1]Laboratory Confirmation is the reference method for this test.

[2] Basis weight determined using Appleton Spec. No. 10001.00 which is based on TAPPI T 410.

A9 SPECIAL TRAINING/CERTIFICATION

Documentation of training related to technology testing, field testing, data analysis, and reporting is maintained for all Battelle technical staff in training files at their respective locations. The Battelle Quality Manager may verify the presence of appropriate training records prior to the start of testing. Battelle technical staff supporting this verification test will have a minimum of a bachelor's degree in science/engineering or equivalent years of experience.

The verification test described in this QAPP will be performed at an Appleton site. All participants in this verification test (i.e., Battelle, EPA, and vendor staff) will adhere to the health and safety requirements of the Appleton site. Operation of each technology (THz technology and nuclear gauge) will be carried out by a trained vendor and Appleton representative during testing. Due to restrictions in the testing agreement, no Battelle or EPA personnel are permitted to touch or handle the Appleton or vendor equipment. Vendor staff will operate only their THz technology during the verification test. They are not responsible for, nor permitted to operate the Appleton test site equipment (i.e. nuclear gauge and laboratory instruments). Appleton staff operating the nuclear gauge and laboratory test equipment must be trained, experienced operators. Data generated by the nuclear gauge and the THz gauge will not be shared between operators during testing.

A10 DOCUMENTATION AND RECORDS

The documents for this verification test will include the QAPP, vendor instructions, reference methods, verification report, verification statement, and audit reports. The project records will include copies of calibration records, sample forms, records laboratory record books (LRB; log book) containing the data collection forms, electronic files (both raw data and spreadsheets), and QA audit files and all other quality records. All of these documents and records will be maintained at the test site during the test, will be transferred to the VTC at the end of testing, then to permanent storage at Battelle's Records Management Office (RMO) at the conclusion of the verification test. Electronic documents and records will also be uploaded to a SharePoint site designated for this test and will be provided to EPA upon request. All Battelle project raw data files are stored for at least 10 years by RMO. EPA will be notified before disposal of any files. Section B10 further details the data recording practices and responsibilities.

A bound, pre-paginated test logbook that contains the data recording forms created for this test (Attachment 1) will be used to document all data and observations made by Battelle during the test. All data generated during the conduct of this project will be recorded directly, promptly, and legibly in ink. All data entries will be dated on the date of entry and signed or initialed by the person entering the data. Any changes in entries will be made so as not to obscure the original entry, will be dated and signed or initialed at the time of the change and shall indicate the reason for the change. Project-specific data forms will be developed prior to testing to ensure that all critical information is documented in real time. The draft forms will be provided to EPA prior to testing.

Table 3. Summary of Data Recording Process

Data to Be Recorded	Where Recorded	How Often Recorded	By Whom	Disposition of Data
Dates, times, and details of test events, technology maintenance, down time, ease of use, etc.	ETV LRBs or data recording forms	Start/end of test procedure, and at each change of a test parameter or change of technology status	Battelle or technology operator	Used to organize and check test results; manually incorporated in data spreadsheets as necessary
Technology calibration information	ETV LRBs, data recording forms, or electronically	At technology calibration or recalibration	Technology operator	Incorporated in verification report as necessary
Technology readings (THz and nuclear gauges)	Recorded electronically by the technology and downloaded to an independent computer, or hard copy data printed by the technology and taped into the ETV LRB, or hand entered into ETV LRBs or data recording forms	Recorded continuously for electronic data, printed after each measurement for hard copy print-outs, or recorded manually with each reading	Technology operator	Converted to or manually entered into spreadsheet for statistical analysis and comparisons
Hobo continuous-monitoring temperature and relative humidity records	Recorded electronically by the instrument and downloaded to independent computer after each data collection event	Recorded continuously during each collection event (production run, laboratory measurement event)	Battelle Quality Manager	Used to verify the temperature and relative humidity of the production and TAPPI rooms
Reference method analysis conditions, procedures, calibrations, QC, etc.	LRBs, or other data recording forms (Battelle will retain copies of all applicable Appleton records for these measurements)	Throughout reference analysis	Collaborator, Battelle, or others assisting in reference analysis	Retained as documentation of sample collection or reference method performance
Reference method results	Electronically or manually into ETV LRBs or data recording forms	Every weight measurement	Collaborator, Battelle, or other reference analysis technician	Transferred to spreadsheets for calculation of results, and statistical analysis and comparisons

SECTION B

MEASUREMENT AND DATA ACQUISITION

B1 EXPERIMENTAL DESIGN

A non-sealed source THz technology (Picometrix T-Ray 4000® Time-Domain Terahertz System) will be tested at Appleton. This will allow for performance evaluation under real world manufacturing conditions. Overall, the performance of the THz technology will be verified based on the following factors:

- Accuracy
- Precision
- Comparability
- Operational factors (ease of use, sampling time, sampling costs).

The verification test is planned to take place over a period of 2-3 days. The evaluations will be performed according to the vendor's approved procedures as described in the user's instructions or manual and will be carried out by a trained and licensed operator provided by the vendor. Similarly, calibration and maintenance of the technologies will be performed by the vendor or vendor-provided operator. If possible, calibration of the vendor technology will be conducted onsite so that Battelle technical staff can observe the procedure. The technology will be evaluated based on comparable metrics to the nuclear gauge. Results from the technology being verified will be recorded manually by the operator on appropriate data sheets or captured in an electronic data system and then transferred manually or electronically to spreadsheets and/or forms for further data analysis.

B1.1 Test Procedures

The following sections describe the test procedures that will be used to evaluate the Picometrix T-Ray 4000® Time-Domain Terahertz System at the Appleton facility.

The proposed comparison testing method is to temporarily set up a THz sensor at a fixed position near the edge of a moving paper sheet in the Appleton production facility. The existing production nuclear gauge system is on a scanner frame and can either be moved back-and-forth across the sheet or "parked" at a position. The THz sensor will be aligned with the nuclear gauge. Both the THZ sensor and nuclear gauge will remain stationary during testing. Figure 3 shows the positioning of the gauges.

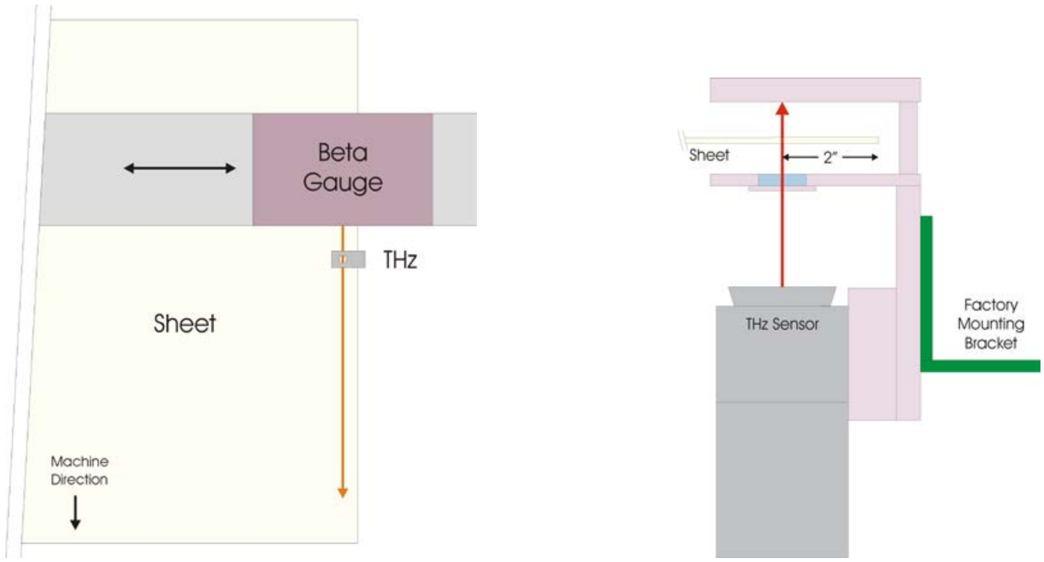


Figure 3. Relative Position of Sample Sheet, Nuclear (Beta) Gauge and THz Sensor

The nuclear gauge and THz sensor will be placed as close to each other as reasonably possible (less than 2-3 feet) at the point in production after the last coating application (inspection position). The THz sensor will remain static (two to four inches from the edge of the paper). The nuclear gauge will both be scanning across the sheet and later parked aligned with the THz sensor. The Appleton production line used for testing involves multiple coating applications.

The nuclear scanning will be needed by the Appleton production staff for the first few (approximately three) rolls once a new product is beginning its production run. Once production conditions permit the nuclear gauge to be temporarily taken off-line, the nuclear gauge will be "parked" in a fixed position aligned with the THz gauge. Continuous overlapping measurement data will be collected and logged by both the gauge and sensor over at least a five minute period. The current nuclear gauge acquires measurements at a 1/5 Hz rate. Thus, a five minute period will result in a log of 60 nuclear gauge measurements. The THz system acquires measurements at a 100 Hz rate, thus 30,000 measurements will be collected over the five minute period.

At the mid-point of each day of testing, the THz system time measurement calibration will be checked. This calibration procedure will follow protocols established by the vendor for the technology. The reason for the test is to confirm the measured ToF delay through a calibration block of fused silica is within specified tolerances. The nuclear gauge will be operated and calibrated according to manufacturer recommendations and Appleton established protocols.

At the start of a new roll of the material under test, the nuclear gauge will operate normally; scanning across the product sheet and providing feedback to the production equipment and staff. If feasible, THz measurements will be acquired frequently during this period for later exploratory analysis. Once the Appleton production staff has become confident of the operation stability, the nuclear gauge may be moved to a static position near the edge of the roll for trial testing. The range of times required to coat an entire roll of paper varies from 15 – 30 minutes. The nuclear gauge will be available to monitor the coating for the second half of a roll. Appleton production staff requires the use of the nuclear gauge during the first half of a roll coating process to ensure proper product quality. All steps within a single measurement run will be completed within a single roll of product. Five rolls each of two different products, a midrange and heavyweight, will be measured.

For all measurement runs, the general data collection procedure is as follows:
once proper manufacturing stability is confirmed by the Appleton production representation, the nuclear scanner will be moved and parked at a predetermined position to align with the THz sensor, somewhere between 2 and 4.25 inches in from the edge of the sheet. The sheet edge position will vary from product to product, hence the variation in the distance from sheet edge. The THz sensor will be positioned as close as possible, within 2-3 feet, directly in front of or behind the nuclear gauge.

Before sample data collection, the THz sensor assembly will be rotated “off-sheet”, i.e., so the product is not in the measurement path. This step simulates a scanning operation; where the sensor moves past the paper sheet (off-sheet) before reversing direction to continue scanning. In this off-sheet position, a system measurement check will be conducted. This check will involve measuring the “open-air space” ToF value. This data will be collected for two seconds, which represents a typical time for a sensor to go off-sheet, reverse direction, and move back onto the sheet. This measurement will be stored for use in the basis weight calculation. The sensor will then be rotated back to the fixed position aligned with the nuclear gauge. On-line data collection should begin within one minute after the completion of this procedure.

Data will then be simultaneously collected and logged for both the nuclear and THz systems for a continuous period of at least five minutes. The THz data collection is triggered with a key press. The method of triggering the logging of the nuclear gauge data will be determined by Appleton staff, and will be started at the same time as the THz system. If the logging start of the nuclear gauge is imprecise, a slightly longer data set will be collected to ensure there is at least five minutes of overlapping data between the nuclear and THz gauges. If necessary, a hand trigger can be used to mark the THz data at the start of nuclear gauge measurements. The clocks for both technologies will be synchronized, and data from both the gauge and sensor will be time stamped to allow the results to be more precisely overlapped. The measurement system clocks used to timestamp the data will be compared within ± 10 minutes of each data collection run.

The nuclear gauge must move off-sheet (i.e., ending on-line measurements) 16 seconds before the end of a roll. This step is required to prevent the individual that pastes the end of the previous paper roll to the beginning of the next roll from being caught in the relatively narrow gap between the top and bottom plates of the nuclear gauge. Once the start of the new production roll is past the inspection point the nuclear gauge will return to scanning the product.

At the end of all rolls for which an inspection was completed, an end-of-roll "tear-off" paper sample will be collected. These samples will all be marked and saved for laboratory and later THz inspection.

Within one minute of the end of the data collection, the THz sensor assembly will again be rotated off-sheet and a two second data set of 'open-air space' (i.e., nothing between the THz transmitter and receiver) ToF values will be recorded.

This process will be conducted at least five times in the production run of the two products (i.e., midrange and heavyweight stock) to be tested.

The laboratory results from the first data collection run will be used to temporarily calibrate the THz sensor to output basis weight values. All "raw" THz measurement values will be saved allowing this calibration to be refined once all measurement results are available.

Testing is planned for two to three days during the week of February 14, 2011. Additional days may be required for system setup and to complete the Technical Systems Audits described in Section C. A summary of the testing is as follows:

- Five data collection runs will be completed per product grade. In general, data logging will begin as soon as reasonably possible once the nuclear gauge can be taken off-line for trial testing.

- All data collection runs will collect and compare simultaneous nuclear / THz and THz / laboratory measurement results, but not nuclear / laboratory results. The paper sample collected for laboratory analysis will also be analyzed off-line with the THz technology in order to further establish measurement accuracy and precision.

  Specific steps of the data collection process for Appleton and Picometrix personnel are:

  - → An Appleton representative will be required to park the nuclear gauge and begin and end data logging.
  - → A Picometrix and Appleton representative will be required to rotate the THz sensor off-sheet, collect a 2-second air space ToF measurement and return the sensor to on-sheet.
  - → A Picometrix representative will be required to begin and end data logging, and to press a trigger to mark the THz data when the nuclear gauge goes off-sheet

Appleton personnel will collect a typical end of roll sample for laboratory basis weight measurement for later off-line THz measurements.

Conditions of testing:

- The materials manufactured at this facility are coated paper products. All samples tested will be coated final products.
- The tested parameter will be product basis weight.
- Two differing bare stock basis weight materials, a mid-range and heavyweight, will be tested.
- TAPPI standard method guidance will be used to determine basis weight.
- The nuclear gauge will be required to be scanning and operating (i.e., not available for trial testing) at the start of every new roll. This is to guarantee production quality control. This period of use will be variable depending on the amount of time necessary to establish confidence in the product quality. A typical roll time is 15 – 20 minutes. It is assumed that the second half of the roll can be used for trial testing.
- The nuclear gauge will go off-sheet 16 seconds before the end of roll, preventing the “paster” that attaches the two rolls from being caught in the relatively narrow gap of the nuclear gauge.

- There could be a significant imprecision in removing the exact sample section from the paper roll. Assuming a manufacturing rate of 3000 ft/min, a 1-second offset between the data logging and selected sample position will result in a mismatch of 50 feet (600 inches).
- The laboratory results for the first measurement run on each product grade will be used to temporarily calibrate the THz sensor to provide real-time measurement results for subsequent runs of that product.
- All THz "raw" measurement results (i.e., Time-of-Flight values) for all runs will be logged to allow a separate comparison of the on-line and off-line measurements.
- It is assumed that the static THz sensor configuration (1.5" gap) can remain in place through the end of all rolls.
- Comparisons of on-line measurements between the nuclear and THz technologies are easy to perform, and are possible whenever the nuclear gauge is available for trial testing purposes.
- It is assumed that the THz sensor configuration can be rotated so as to simulate going off-sheet. While off-sheet, calibration checks of the THz sensor will be made.
- It is assumed that a THz sensor can easily be used in the "TAPPI room" laboratory testing area.
- Additional accuracy and all precision THz measurements will be made in the TAPPI room on the same samples used for the laboratory basis weight testing. In paper products, the paper "formation" creates basis weight variation on a short length scale. The large spot and web movement creates natural averaging of the nuclear gauge's measurements so that formation does not have an effect. In the laboratory measurements, sheets of paper are weighed to determine basis weight, again naturally averaging over variations due to formation. Due to the relatively smaller inspection spot of the THz sensor, multiple measurements over an area will be performed to avoid any issues with the product's formation. Another option is to cut and stack multiple sheets of the laboratory samples for inspection with the THz sensor. The stacking of samples should reduce formation induced product basis weight variability.

Data Collection Runs:

On-line Data Collection

1) For a mid-range basis weight product:
   a) Run 1: Acquire simultaneous nuclear and THz sensor measurements for at least the last five minutes of a production roll. The nuclear gauge will move off-sheet (and thus end its data collection) 16 seconds before the end of the roll. The THz sensor will continue to collect data past the end of the roll.
      i) Use a hand trigger switch to mark the THz data when nuclear gauge starts to move off-sheet, thus aligning the end of nuclear gauge measurements to the THz results.
      ii) Collect an end-of-sheet tear off sample for later laboratory and THz analysis. The laboratory results can be simultaneously compared to the nuclear and THz sensor results.
      iii) Use the laboratory results from this first data run to temporarily calibrate THz sensor to provide real-time results for subsequent measurement runs.
      iv) Use laboratory sample to perform static THz measurements to further confirm accuracy and establish static measurement precision.
   b) Runs 2 - 5: Are the same as Run1 except the THz system will now be programmed to directly report an estimated basis weight value (while still logging the ToF raw data).
   c) Use laboratory samples to perform static THz measurements to further confirm accuracy and establish static measurement precision.

2) Repeat all above steps for heavyweight product.

A typical single product production run can range from 12 hours to > 50 hours. The five repeated measurements will occur within these ranges and will be spread out to encompass as large a variation of the two basis weight materials (medium and heavy) as possible. The timing of the repeated measurements will be determined by Appleton.

There will not be any control of the environmental conditions during testing. That is, both gauges will be operating in a manufacturing factory environment. Environmental conditions present during testing will be noted based on information provided by Appleton staff.

Battelle staff present at the test site will also provide a temperature and relative humidity gauge to record conditions on the production floor as well as in the TAPPI conditioning room.

All laboratory results will be collected and run per Appleton protocols, which are based on TAPPI standards[3]. The protocols are considered business confidential information. In general, the samples will be bagged after collection and allowed to condition in TAPPI rooms on the manufacturing floor. The environment of these rooms is tightly controlled (72 °F and 50% RH). The length of conditioning time is determined by the sample type. Laboratory measurements will be collected on tear-off samples collected for each of the five runs for each paper type. Once all on-line data collection is completed in the production facility, a THz sensor will be moved to the TAPPI room to allow static off-line THz measurements.

Off-line Data Collection

Once laboratory basis weight analyses are complete for a given run, the five physical paper samples will be repeatedly measured with a static THz sensor in the TAPPI room to further establish measurement accuracy and precision. The steps to prepare the five paper samples follow.

1) For each sample, there are four sheets, all 12.5” by 9.5”. These sheets will be cut into two inch by two inch pieces resulting in 96 pieces.
2) The suggested best method to establish a calibration factor, and thus the accuracy, is to stack a number (e.g., 12 pieces yielding eight stacks) of individual sheet sections in a sample holder and measure the total ToF. This method improves the measurement confidence in two ways:
    i. By stacking the sheets, any changes in the material’s formation (short distances variations in sample mass / density / consistency) will be averaged away with a single measurement. Note: it is not expected that the eight measurements will yield the same result, even with this spatial averaging procedure. A coated paper product will have appreciable spatial variation in basis weight over fractions of the dimensions of the total sheet sample.
    ii. The increase of total ToF will reduce the contribution of any fixed noise in the measurement.
3) Each of the eight sample stacks will be measured for an integration period of about 30 seconds.

4) All individual sample stack measurement results will then be averaged and used to calculate a THz ToF to basis weight conversion factor.
5) One randomly selected sample stack will be installed in the sample holder and measured twice. This will be used to determine the reproducibility of the measurement.
6) Unfortunately, static off-line nuclear gauge measurements are not possible during this trial. Thus, accuracy and precision comparisons to the nuclear gauge will not be made.
7) Alternatively, a large number of one second average measurements over a number of different positions on a single sheet can be collected. This method would be preferred if it is demonstrated that the formation of the product does not adversely affect the measurement.

The THz “raw” data will consist of a .csv data file of Time-of-Flight values for every measurement (100 per second). The calibration factor to convert these measurements to basis weight will be temporarily determined from the laboratory results from the first data run for a new product. That calibration factor will then be used for subsequent data runs to provide real-time basis weight values and logged as the measurement results. Because the ToF values will be saved for every run, it will be possible to recalibrate the THz measurements to align with the method of choice; on-line THz measurements versus laboratory measurements, off-line static THz measurements versus laboratory measurements or on-line THz versus on-line nuclear gauge.

The data format of the nuclear gauge will be determined by Appleton. There exist possible proprietary limitations on the use of data streams of the nuclear system when in parked mode. Appleton will determine how to best log and report the data to eliminate these issues.

As every measurement of each gauge will be logged, for some analyses the averaging interval for both sets of data can be altered in post analysis, filtered and smoothed. The majority of the processed THz data sets will be made available by the end of that testing day. The availability of the nuclear gauge data will be determined at the start of testing.

B1.1.3 Testing Parameters

A total of 10 data sets each will be taken by the THz technology and the nuclear gauge technology on the Appleton production line. Quantitative assessments will be made. The following sections describe in more detail the evaluation of the testing parameters.

B1.1.3.1 Accuracy

Accuracy will be assessed by evaluating the results of the THz gauge against the results from the standard laboratory method for the determination of basis weight. The comparison of accuracy may be limited for this production measurement trial. The limitation is the collection of laboratory samples (i.e., the standard measurement method) in that the exact overlap between the on-line and physical paper sample cannot be guaranteed. In order to address this possible complication, off-line static THz measurements are planned on the paper samples. By making a large number of measurements over the spatial area of the lab sample, the accuracy of the THz measurement can be assessed.

B1.1.3.2 Precision

The precision of the THz system will be assessed with static off-line measurements of paper samples used for laboratory basis weight determination. With repeated measurements on a static sample, the measurement precision can be assessed.

B1.1.3.3 Comparability

This test will assess the performance of the THz technology based on the measurement of whole sample basis weight. The measurement of that whole sample basis weight accounts for the vast majority of nuclear gauge use. If a nuclear gauge is extended to measure an additional parameter (e.g., thickness), the additional measurement is dependent on the same results to obtain basis weight. Thus, comparing the basic measurement of basis weight should allow the best comparison between the two technologies. There are, however, some limitations in the comparison of the nuclear and THz gauge measurement of basis weight that should be noted.

One limitation in the comparison between the techniques is the operational need to remain static for this testing. That is, cross sheet scanning will not be conducted. Though cross sheet scanning is typically how the nuclear gauge would be operated in the Appleton production environment, due to practical limitations, cross sheet scanning is beyond the scope of this verification test. An additional factor limiting comparison is the difference in measurement spot size between the two technologies. A typical nuclear gauge inspection spot is 25 millimeter (mm), while a typical THz sensor inspection spot is 2 mm. At a production line speed of 4000 feet/minute, these "spots" become spread to 25 mm x 1041 mm for the nuclear gauge's 50 millisecond measurement integration time and 2 mm x 125 mm with an 80 mm gap between THz measurements. Clearly, these two systems will be inspecting the product in different ways. The nuclear gauge covers a larger area and thus could provide an improved result for the average basis weight value. The higher measurement rate of the THz system somewhat compensates for this difference. In addition, the smaller inspection spot allows for better streak detection and could possibly provide information on the formation (uniformity of basis weight) of the sample.

B1.1.3.4 Operational Factors

Operational factors such as maintenance needs, power needs, calibration frequency, data output, consumables used, ease of use, repair requirements, training and certification requirements, safety requirements and image throughput will be evaluated based on Battelle staff testing observations and input provided from the vendor. Input will either be provided by the vendor on-site during the verification test and be recorded by Battelle staff or will be provided in documentation to the VTC after completion of the verification test. To the extent possible, Battelle technical staff will also observe and record their own observations of these operational factors. Examples of information to be recorded include the daily status of diagnostic indicators for the technology, use or replacement of any consumables, use and nature of power supply needed to operate the technology, the effort or cost associated with maintenance or repair, vendor effort (e.g., time on site) for repair or maintenance, the duration and causes of any technology down time or data acquisition failure, observations about technology startup, ease of use, clarity of the user's instruction manual, user-friendliness of any needed software, overall convenience of the technologies and accessories/consumables, the safety hazard associated with the use of the technology, or the number of images that could be taken and processed per hour or per day. These observations will be summarized to aid in describing the technology performance in the verification report.

B1.2 Statistical Analysis

The statistical methods and calculations used for evaluating quantitative performance parameters are described in the following sections. Continuous measurement data will be collected and logged by both the nuclear gauge and THz sensor for at least a five minute period during each paper roll test. This will be done over at least five separate runs (Runs 1 – 5 for each paper type). A five minute period will result in a log of 60 nuclear gauge measurements and 30,000 measurements from the THz technology.

As noted in Section B1.1, cross sheet scanning is typically performed by the nuclear gauge during production at the Appleton facility. The scanning is required to monitor the uniformity of the coating process. As the nuclear gauge scans across the sheet, the product is divided into a number of cross direction "bins". The nuclear gauge will scan across the web at approximately 6 inches per second, producing 96 bin measurements for each single 15 second scan across the product. Then, a number of repeated scans are averaged before a measurement result for that bin is reported. This means for routine operation of a nuclear gauge, a single bin measurement is an average of a number of sets of measurements with each individual measurement covering a certain amount of product. These values are running averages. These average results are then reported through the system to help monitor the production process. The averaging of both across the sheet (cross direction) measurements and along the production direction (machine direction) measurements is used to maintain controlled measurement feedback to the coating devices.

However, for this test, the gauges must remain in "parked" mode. Thus, the cross web averaging cannot be accomplished. However, the data acquired during this test is the closest approximation to the typical operation of the nuclear and THz gauges in a controlled setting.

B1.2.1 Accuracy

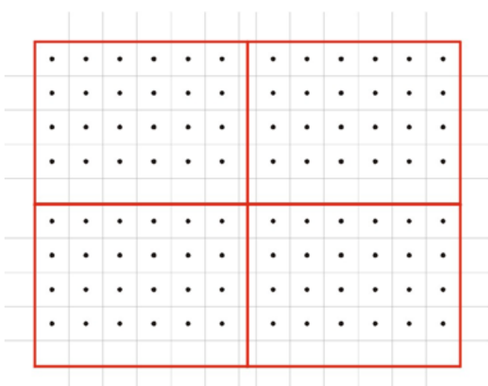
The accuracy of the results will be assessed by calculating the percent error between the laboratory measurements and the results from the nuclear gauge and THz technologies. Percent error will be calculated using the following:

$$\%Error = \frac{|Technology - Lab|}{Lab} \times 100 \tag{1}$$

The first sample collection run (Run 1) will be used to establish the calibration factor used to calculate basis weight. The remaining runs (Run 2-5) end of roll sample collection can be used for an accuracy comparison. However, as previously mentioned, there are identified issues with using end of roll samples to compare to on-line measurements.

In an attempt to better qualify accuracy, off-line static THz measurement of samples collected for laboratory analysis is proposed. By measuring the laboratory samples used for the basis weight measurement, at least the overlap issue is addressed. The remaining consideration is how measurements over what percentage of the 475 square inch samples should be measured with THz. In order to integrate a larger sample area and to reduce the effect of formation (variability in the sample's basis weight), the off-line tests will be made with a large number (e.g., 12) of stacked sections cut from the large laboratory sample sheet. If the sample sheet was completely cut into two inch by two inch square pieces, then eight stacks of 12 sheets per stack could be used for static measurements.

It is important to note that the product under study, coated paper, is inherently non-uniform. Thus, the ToF delay measurements for each "stack" are expected to have different values because they are taken from different positions of the laboratory test sheets. The need to average out this non-uniformity is the logic for selection sample form all positions of the four lab test sheets. The goal is to measure the whole area of laboratory samples, as illustrated in Figure 4.



Red squares represent 12.5 by 9.5 inch sheets cut from the tear off. The four sheets combine for 475 square inches of sample. The black dots represent the position of a THz measurement when the cut sections are stacked into eight groups of 12 sections.

Figure 4 - Illustration of Laboratory Sample Sectioning to Achieve Spatial Sampling of Test Sheets

B1.2.3 Precision

The cut samples used in the above testing will also be used to determine measurement precision. The precision will calculated as the standard deviation of repeated measurements.

$$StdDev = \sqrt{\frac{\sum_{i=1}^{N}\left(Xi - X\right)^2}{\mathrm{N-1}}} \quad (1)$$

B1.2.2 Comparability

One simple measure of comparability between the technologies will be assessed by calculating the percent difference between the measurements made by the THz technology and the nuclear gauge while in the on-line production mode. This evaluation will help in assessing the performance of the THz technology in relation to that of the instrument typically used for production control processes (i.e., the nuclear gauge). Percent difference will be calculated using the following:

$$\%Difference = \frac{\left(Terahertz\ Technology\ Result - Nuclear\ Gauge\ Result\right)}{Nuclear\ Gauge\ Result} \times 100 \quad (2)$$

Comparability results will be calculated for each separate run evaluated during the verification test. Average percent difference will also be determined across the five runs.

Some exploratory data analysis procedures will also be examined to evaluate the comparability of the two technologies. In particular, the raw data from each technology will be plotted together. These plots will allow for examination of how well the measurements from the two gauges track each other. These plots might also be used to explore variations in basis weight measurements over time. The raw data may also be smoothed for the exploratory analysis, such as using locally weighted scatterplot smoothing. The use of histograms, or other techniques such as Grubbs, Ferguson, Dixon, and Hofer-Rickard methods, will be utilized to study the data variability (i.e., outliers).

More advanced statistical methods of comparing the variance between the two data sets will also be considered. A t-test and/or an ANOVA will be considered to evaluate the agreement and variance between the data from the nuclear gauge and the THz sensor.

B1.3 Reporting

Following completion of the data evaluations, a draft verification report and verification statement will be prepared, stating the verification test procedures and documenting the performance observed. For example, descriptions of the data acquisition procedures, consumables used, repairs and maintenance needed, and the nature of any problems will be presented in the draft report. The report will briefly describe the ETV Program, the AMS Center, and the procedures used in verification testing. The results of the verification test will be stated quantitatively, without comment on the acceptability of the technology's performance. The draft verification report will be submitted for review by the technology vendor, by EPA, and other peer reviewers. Comments on the draft report will be addressed in revisions of the report. The reporting and review process will be conducted according to the requirements of the AMS Center QMP.[1]

B2 SAMPLING REQUIREMENTS

Laboratory samples will be collected at the end of each roll for which a set of nuclear and THz gauge measurements were collected. Thus, five laboratory samples will be collected from the mid-weight paper stock and five from the heavyweight stock. The sample will be an approximately 400 square inch piece of material. After a sample is cut from the end of a roll, it is immediately bagged per the pertinent Appleton protocol and/or TAPPI standard. The samples will be transported and stored in a specialty TAPPI room (i.e., tightly controlled environment) within the Appleton production facility. The samples will then condition for a set time in the TAPPI room before measurements are taken. Conditions for the conditioning room will be followed per established Appleton and/or TAPPI protocols.

B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

The paper samples collected for laboratory measurements will be handled per the appropriate Appleton paper protocol.[3] Each paper sample will be labeled and assigned a unique sample ID, known as the "Reference Roll Number", generated by Appleton. This will identify the Roll No., Run No., Paper Type, Date/Time of Collection, and other pertinent processing parameters. The unique sample ID will also be documented in the test LRB or associated data collection sheet(s). Sample custody information that documents collection, possession, and transfer of samples will be documented directly on the sample collection forms.

Once conditioned for the appropriate amount of time in the TAPPI room, the measurements of interest will be collected using the Appleton laboratory basis weight measurement protocol[3]. Once measurements are complete, unless otherwise specified, the samples will be available for further study by the THz gauge. Samples will not be archived for this test or removed from the Appleton facility.

B4 REFERENCE METHOD

Laboratory analysis of basis weight at the Appleton laboratory will serve as the reference method for this test. Laboratory procedures are described in Appleton Standard Test Method 10001.00 *Basis Weight – Laboratory Determination of Coated and Uncoated Paper*.[3] This method is based on TAPPI Method T 410. Each sample will be approximately 800 square inches and must be tested, cut and weighed at the TAPPI Room conditions (defined in Table 2). The laboratory measurements will be performed by Appleton staff. Laboratory measurement results will be provided to Battelle. The QA/QC requirements for the performance of the analytical method are described as data quality indicators (DQI) in Section A8.

B5 QUALITY CONTROL REQUIREMENTS

Table 2 presents the DQIs and criteria for the laboratory reference method measurements. The reference method measurement quality will be assured by adherence to these measurement quality and DQI criteria. All laboratory equipment will be maintained, calibrated, and operated according to Appleton internal requirements and TAPPI standards.

B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

The equipment for the reference method for this test includes balance, pre-scribed cutting device (guillotine cutter) (indicating the cut dimensions of the type of paper under study), temperature measuring device, and hygrometer. This equipment will be tested, inspected, and maintained by Appleton staff according to the manufacturer's recommendations and any appropriate TAPPI standards to ensure that the performance requirements established in this document can be achieved. Battelle staff will observe that the balance, temperature measuring device, and hydrometer have been tested and verified to be operating with the Table 2 criteria prior to use. Operation of the guillotine cutter will be observed during laboratory tests but cutting accuracy is not a DQI. The TSA will confirm compliance with the QAPP. A Hobo continuous-recording monitor that has been calibrated at Battelle's ISO 17025-certified instrument laboratory will collect temperature and relative humidity measurements during production test runs and while measurements are made in the TAPPI room.

B7 INSTRUMENT CALIBRATION AND FREQUENCY

The balance, temperature measuring device, and hygrometer, will be calibrated according to the manufacturer's specifications and Appleton procedures prior to testing. Table 2 defines calibration frequency. Calibration of the TAPPI room equipment will be verified during the TSA. Certified analytical balance weights will be used to verify balance calibration and a Hobo continuous-recording monitor will be used to verify the accuracy of the TAPPI room temperature and relative humidity equipment. The nuclear gauges, MeasureX, MX Open system, will have been calibrated by the Appleton plant operator. The THz technology will be calibrated by the vendor according to the technology's specified procedures. This calibration will be performed onsite prior to testing and confirmed once every mid-day.

B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

It is not anticipated that any materials, supplies, or consumables will be ordered by the VTC for this test. Supplies used by Appleton and the vendor will be received, inspected, and documented according to their internal procedures. For this test, additional documentation may be required. This includes: source, certification of analysis or verification, and date of receipt for supplies that could impact data quality. If this documentation is required, Appleton staff and vendor representatives will be asked to supply the appropriate paperwork.

B9 NON-DIRECT MEASUREMENTS

No non-direct measurements will be used during this verification test.

B10 DATA MANAGEMENT

Various types of data will be acquired and recorded electronically or manually by Battelle during the verification test. Electronic data, including the JOHN NEW COMMENT: THz sensor, nuclear gauge, and Hobo monitoring data, will be saved with unique file names that are directly traceable to the test records for each test run date/time and activity. Table 3 summarizes the types of data to be recorded. All maintenance activities, repairs, and calibrations relevant to the technology operation will be requested from Appleton staff and the technology vendor. All observations relevant to the technology operation during the course of verification testing will be documented in LRBs or on data sheets by Battelle technical staff present during testing. Results from the reference method, including raw data, analyses, and final results, will be documented by the Appleton laboratory and compiled by Battelle. Battelle will obtain copies of all results (hard copy and electronic) in real time to ensure the integrity of the data.

Records received by or generated by any technical staff during the verification test will be reviewed by a Battelle staff member within two weeks of generation or receipt, before the records are used to calculate, evaluate, or report verification results. If a Battelle staff member generated the record, this review will be performed by a Battelle technical staff member involved in the verification test, but not the staff member who originally generated the record. The review will be documented by the person performing the review by adding his/her initials and date to the hard copy of the record being reviewed. A technical review of 100% of the test and reference data produced will be conducted. In addition, any calculations performed by technical staff will be spot-checked by Battelle QA and/or technical staff to ensure that calculations are performed correctly. Calculations to be checked include any statistical calculations described in this QAPP.

Data obtained during the verification test will be maintained confidentially at Battelle, and used only for purposes of the technology evaluation. Data reporting in the final report will consist of tabular results of the calculations in Section B.

It is anticipated that testing of the THz technology will take place over 2-3 days during the week of February 14th, 2011. As such, Battelle will provide technology test data and associated reference data (including records; data sheets; notebook records) within 2 weeks of receipt to EPA. The goal of this data delivery schedule is prompt identification and resolution of any data collection or recording issues.

SECTION C
ASSESSMENT AND OVERSIGHT

C1 ASSESSMENTS AND RESPONSE ACTIONS

Every effort will be made in this verification test to anticipate and resolve potential problems before the quality of performance is compromised. One of the major objectives of this QAPP is to establish mechanisms necessary to ensure this. The procedures described in this QAPP, which is peer reviewed by a panel of outside experts, implemented by the technical staff and monitored by the VTC, will give information on data quality on a day-to-day basis. The responsibility for interpreting the results of these checks and resolving any potential problems resides with the VTC, who will contact the Battelle AMS Center Manager, Battelle AMS Center QM, EPA AMS Center Project Officer, and EPA AMS Center QM if any deviations from the QAPP are observed. The VTC will describe the deviation in a teleconference or by email, and once a path forward is determined and agreed upon with EPA, the deviation form will be completed. Technical staff have the responsibility to identify problems that could affect data quality or the ability to use the data. Any problems that are identified will be reported to the VTC, who will work with the Battelle Quality Manager to resolve any issues. Action will be taken to control the problem, identify a solution to the problem, and minimize losses and correct data, where possible. Independent of any EPA QA activities, Battelle will be responsible for ensuring that the following audits are conducted as part of this verification test.

C1.1 Performance Evaluation Audits

No independent standard of basis weight is available to provide an independent, certified standard against which laboratory results can be compared.

C1.2 Technical Systems Audits

The Battelle Quality Manager will perform a technical systems audit (TSA) during this verification test. The purpose of this audit is to ensure that the verification test is being performed in accordance with the AMS Center QMP,[1] this QAPP, and any Standard Operating Procedures (SOPs) used by Appleton. Prior to the TSA, a project-specific checklist based on the

QAPP, Appleton SOP, and TAPPI requirements will be prepared to guide the TSA. The TSA will include a review of the test location and general testing conditions; observe the testing activities; and review test documentation at both the Appleton plant and laboratory. During the TSA, the Battelle Quality Manager or a designee will specifically review the reference method procedures, compare actual test procedures to those specified or referenced in this plan, and review data acquisition and handling procedures.

The Battelle Quality Manager will prepare an initial TSA report and will submit the report to the EPA QA Manager (with no corrective actions documented) and VTC within 10 business days after completion of the audit. A copy of each final TSA report (with corrective actions documented) will be provided to the EPA AMS Center Project Officer and Quality Manager within 20 business days after completion of the audit. At EPA's discretion, EPA QA staff may also conduct an independent on-site TSA during the verification test. The TSA findings will be communicated to technical staff at the time of the audit and documented in a TSA report.

C1.3 Data Quality Audits

The Battelle Quality Manager, or designee, will audit at least 25% of the sample results data acquired in the verification test and 100% of the calibration and QC data versus the QAPP requirements. Two audit of data quality (ADQ) will be conducted for this project: data collected during the test will be audited within 10 business days of receipt by the Quality Manager or designee and assessed using a project-specific checklist. The synthesized data and verification report will be audited within 10 business days of receipt of all test data and the report. During these audits, the Battelle quality manager, or designee, will trace the data from initial acquisition (as received from the Appleton or the vendor's technology), through reduction and statistical comparisons, to final reporting. Data must undergo a 100% validation and verification by technical staff (i.e., VTC, or designee) before it will be assessed as part of the ADQ. All QC data and all calculations performed on the data undergoing the audit will be checked by the Battelle Quality Manager or designee. Results of each ADQ will be documented using the checklist and reported to the VTC and EPA within 10 business days after completion of the audit. The final ADQ will assess overall data quality, including accuracy and completeness of the technical report.

C1.4 QA/QC Reporting

Each audit will be documented in accordance with Sections 3.3.4 and 3.3.5 of the AMS Center QMP.[1] The results of the audits (both TSA and ADQ) will be submitted to EPA within 10 business days as noted above. Audit reports will include the following:

- Identification of any adverse findings or potential problems
- Response to adverse findings or potential problems
- Recommendations for resolving problems
- Confirmation that solutions have been implemented and are effective
- Citation of any noteworthy practices that may be of use to others.

C2 REPORTS TO MANAGEMENT

During the laboratory evaluation, any QAPP deviations will be reported immediately to EPA. The Battelle Quality Manager and/or VTC, during the course of any assessment or audit, will identify to the technical staff performing experimental activities any immediate corrective action that should be taken. A summary of the required assessments and audits, including a listing of responsibilities and reporting timeframes, is included in Table 4. If serious quality problems exist, the Battelle Quality Manager will notify the AMS Center Manager, who is authorized to stop work. Once the assessment reports have been prepared, the VTC will ensure that a response is provided for each adverse finding or potential problem and will implement any necessary follow-up corrective action. The Battelle Quality Manager will ensure that follow-up corrective action has been taken. The QAPP and final report are reviewed by the EPA AMS Center Quality Manager and the EPA AMS Center Project Officer. Upon final review and approval, both documents will then be posted on the ETV website (www.epa.gov/etv).

Table 4. Summary of Assessment Reports[1]

Assessment	Prepared By	Report Submission Timeframe	Submitted To
TSA	Battelle	TSA response is due to QM within 10 business days TSA responses will be verified by the QM and provided to EPA within 20 business days	EPA ETV AMS Center
ADQ (raw data)	Battelle	ADQ will be completed within 10 business days after receipt of first data set	EPA ETV AMS Center
ADQ (Synthesized data and verification report)	Battelle	ADQ will be completed within 10 business days after completion of the verification report review	EPA ETV AMS Center

[1] Any QA checklists prepared to guide audits will be provided with the audit report.

SECTION D

DATA VALIDATION AND USABILITY

D1 DATA REVIEW, VERIFICATION, AND VALIDATION REQUIREMENTS

The key data review requirements for the verification test are stated in Section B10 of this QAPP. In general, the data review requirements specify that the data generated during this test will be reviewed by a Battelle technical staff member within two weeks of data generation. The reviewer will be familiar with the technical aspects of the verification test, but will not be the person who generated the data. This process will serve both as the data review and the data verification, and will ensure that data have been recorded, transmitted, and processed properly.

The data validation requirements for this test involve an assessment of the quality of the data relative to the DQIs and Measurement Quality Objectives for this test referenced in Table 2. Any deficiencies in these data will be flagged and excluded from any statistical comparisons, unless these deviations are accompanied by descriptions of their potential impacts on the data quality.

D2 VERIFICATION AND VALIDATION METHODS

Data verification is conducted as part of the data review, as described in Section B10 of this QAPP. A visual inspection of handwritten data will be conducted to ensure that all entries were properly recorded or transcribed and that any erroneous entries were properly noted (i.e., single line through the entry with an error code and the initials of the recorder and date of entry). Electronic data from the technologies and other instruments used during the test will be inspected to ensure proper transfer from the data logging system. Data manually incorporated into spreadsheets for use in calculations will be checked against handwritten data to ensure that transcription errors have not occurred. All calculations used to transform the data will be reviewed to ensure the accuracy and the appropriateness of the calculations. Calculations performed manually will be reviewed and repeated using a handheld calculator or commercial software (e.g., Excel). Calculations performed using standard commercial office software (e.g., Excel) will be reviewed by inspecting the equations used in calculations and verifying selected calculations by handheld calculator. Calculations performed using specialized commercial software (i.e., for the nuclear and THz gauges) will be reviewed by inspection and, when feasible, verified by handheld calculator, or standard commercial office software.

To ensure that the data generated from this test meet the goals of the test, a number of data validation procedures will be performed. Sections B and C of this QAPP provide a description of the validation safeguards employed for this verification test. Data validation and verification efforts include the completion of QC activities and the performance of TSA as described in Section C. The data from this test will be evaluated relative to the measurement DQIs described in Section A4 and B5 of this QAPP. Data failing to meet these criteria will be flagged in the data set and not used for evaluation of the technologies, unless these deviations are accompanied by descriptions of their potential impacts on the data quality.

A data quality audit will be conducted by the Battelle Quality Manager to ensure that data review, verification, and validation procedures were completed, and to assure the overall data quality.

D3 RECONCILIATION WITH USER REQUIREMENTS

The purpose of this verification test is to evaluate the performance of a commercial technology that measures basis weight (mass per unit area). This test evaluates the non-radioactive source THz technology capabilities. This evaluation will include comparisons of the results from the technology to results from the standard laboratory technique, which is being used as the reference method for this test and the results of a nuclear gauge. To meet the requirements of the user community, the data obtained in such a verification test will include thorough documentation of the technology’s performance during the verification test. The data review, verification, and validation procedures described above will assure that verification test data meet these requirements, are accurately presented in the verification reports generated from the test, and that data not meeting these requirements are appropriately flagged and discussed in the verification reports. Additionally, all data generated using the reference method, which are used to evaluate technology results during the verification test, should meet the QA requirements of any applicable standard operating procedures or instrumentation instruction manuals.

This QAPP and any resulting ETV verification report generated following procedures described in this QAPP will be subjected to review by the participating technology vendor, ETV AMS Center staff, test collaborators, EPA, and external expert peer reviewers. These reviews will assure that this QAPP, verification test, and the resulting report meet the needs of potential users and regulators. The final report will be submitted to EPA in 508 compliant Adobe Portable Document Format (pdf) and subsequently posted on the ETV website.

SECTION E
REFERENCES

E1 REFERENCES

1. Quality Management Plan for the ETV Advanced Monitoring Systems Center, Version 7.0, U.S. EPA Environmental Technology Verification Program, Battelle, Columbus, Ohio, November 2008.

2. Environmental Technology Verification Program Quality Management Plan, EPA/600/R-08/009, U.S. Environmental Protection Agency, Cincinnati, Ohio, January 2008.

3. Appleton Standard Test Method, “Basis Weight – Lab Determination of Coated and Uncoated Paper,” Spec. No. 10001.00, Revision 1, July 1993.