# THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM







TECHNOLOGY TYPE: ALTERNATIVE TECHNOLOGY FOR SEALED

RADIOACTIVE SOURCE NUCLEAR GAUGES

APPLICATION: PRODUCTION QUALITY CONTROL

TECHNOLOGY NAME: T-Ray 4000® Time-Domain Terahertz System

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## **ETV Joint Verification Statement**

The U.S. Environmental Protection Agency (EPA) has established the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by accelerating the acceptance and use of improved and cost-effective technologies. ETV seeks to achieve this goal by providing high-quality, peer-reviewed data on technology performance to those involved in the design, distribution, financing, permitting, purchase, and use of environmental technologies. Information and ETV documents are available at www.epa.gov/etv.

ETV works in partnership with recognized standards and testing organizations, with stakeholder groups (consisting of buyers, vendor organizations, and permitters), and with individual technology developers. The program evaluates the performance of innovative technologies by developing test plans that are responsive to the needs of stakeholders, conducting field and laboratory tests (as appropriate), collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted according to rigorous quality assurance (QA) protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

The Advanced Monitoring Systems (AMS) Center, one of six verification centers under ETV, is operated by Battelle in cooperation with EPA's National Risk Management Research Laboratory. The AMS Center evaluated the performance of an alternative technology for sealed radioactive source nuclear gauges. This verification statement provides a summary of the test results for Picometrix T-Ray  $4000^{\circ}$  Time-Domain Terahertz (TD-THz) System.

### VERIFICATION TEST DESCRIPTION

This verification test evaluated the ability of a technology with a non-radioactive source, the Picometrix T-Ray  $4000^{\circ}$  Time-Domain Terahertz (TD-THz) System, to provide production-line quality control measurements. Testing was designed to monitor basis weight measurements at a paper production facility. Test results were compared to results from a reference technology, the commonly used Measurex MXOpen system nuclear gauge. The test design consisted of a production process phase and a laboratory phase. Testing was conducted on February 16, 17, and 23, 2011 at a commercial paper company in Appleton, WI.

The performance of the technology was tested using two different thicknesses of paper –mid- and heavy weight. For each paper type, five separate rolls (treated as lots at the site) were tested, resulting in ten sets of synchronized nuclear gauge and THz sensor data collected during the actual production process. Data collected within a single lot run were generated within a single roll of product. The emitter for the nuclear gauge system was "parked" at the edge of the paper and the THz sensor aligned with the nuclear gauge emitter. Both the THz sensor and nuclear gauge remained stationary during testing. The data collected throughout each run was time stamped by each technology: the nuclear gauge to 5 second increments and THz results to 0.01 second increments. The nuclear gauge technology was a Measurex MXOpen system that existed at the facility, and was operated by qualified facility staff according to standard operating procedures for that facility. The Picometrix THz technology was operated by the vendor.

A paper sample was collected from the end of each roll used in the production line testing phase. The 10 samples collected from these runs were used for testing during the laboratory phase. Laboratory testing was performed in a Technical Association of the Pulp and Paper Industry (TAPPI) room with tightly controlled temperature and humidity. The paper sample basis weight from each lot was measured using standard TAPPI gravimetric protocols, cut into small squares, stacked, and the time of flight (ToF) measured for each stack using the THz technology. A qualified technician conducted all reference measurements for this verification test according to procedures described in the facility's Standard Test Method 10001.00 *Basis Weight – Laboratory Determination of Coated and Uncoated Paper* as modified in the quality assurance project plan (QAPP) for this test.

The Picometrix TD-THz System was verified by evaluating the following parameters:

- Accuracy Accuracy was assessed by evaluating the offline basis weight determined by the Picometrix THz sensor on cut, stacked, tear-off paper samples against basis weight measured using the standard laboratory method. The accuracy of the results was assessed by calculating the percent error.
- *Precision* Precision was assessed by triplicate THz sensor measurements of one stack of the heavy paper used for laboratory basis weight determination. Precision was calculated as the standard deviation of repeated measurements.
- Comparability Comparability was assessed by evaluating the online results from the nuclear gauge and THz sensor directly against each other on a roll by roll basis. Comparability between the technologies was assessed by calculating the percent difference. A paired t-test was used to determine if the percent difference between technology readings for a given roll of paper was significantly different from zero.
- *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 were also evaluated.

QA oversight of verification testing was provided by Battelle and EPA. Battelle QA staff conducted technical systems audits of the testing, and Battelle QA staff conducted a data quality audit of at least 25% of the test data. This verification statement, the full report on which it is based, and the QAPP for this verification test are available at <a href="https://www.epa.gov/etv/centers/center1.html">www.epa.gov/etv/centers/center1.html</a>.

#### TECHNOLOGY DESCRIPTION

The following is a description of the Picometrix TD-THz System, based on information provided by the vendor. The information provided was not verified in this test.

The TD-THz system emits and detects a narrow (<1 picosecond [ps]) electromagnetic (EM) pulse that forms photons in the THz frequency range. The THz pulse is low energy and can be focused and reflected. After this pulse has interacted with matter (transmission, reflection, scatter), the changes in the pulse can be analyzed by measuring changes in the Time-of-Flight (ToF). Alteration of the ToF (readings will be less than the speed of light) is a result of the index of refraction for the material of interest. This ToF value is calibrated against accepted values of the material's basis weight. Analysis of the ToF for THz pulses can be used to determine the basis weight (mass per unit area) of manufactured products. The THz method is a time-based measurement, as compared to the amplitude attenuation-based measurement method implemented by nuclear gauges. The amplitude of the transmitted THz pulse may be configured to provide simultaneous complementary information related to the chemical and physical properties of the sample, including moisture content.

#### VERIFICATION RESULTS

**Accuracy.** The percent error for the THz sensor basis weight, as compared to the laboratory gravimetric reference method values, ranged from 0.1-2.8% for the mid-weight rolls and 0.3-2.9% for the heavy weight rolls. The percent error for the offline THz sensor readings, based on the averages across all five rolls for a particular weight paper, was 0.02%.

**Precision.** The standard deviation for triplicate THz sensor measurements was 0.0115 ps with a coefficient of variation of <0.005%.

Comparability. Plots of the online results for the THz sensor and the nuclear gauge showed that the THz sensor data tracked the nuclear gauge basis weight trends for most rolls across both paper weights. In four instances, the THz sensor basis weight results diverged at some point within the production of a roll of paper. The offset in the two data sets at these points was 0.2 lbs/ream for most rolls and 1-2 lbs/ream for one roll where there was a consistent offset between the THz sensor and nuclear gauge data. In the other three instances, the data divergence was only over the course of seconds at certain points in the production of a particular roll.

The average percent difference between the mean basis weight values for the THz sensor and nuclear gauge results ranged from -0.65% to 0.14%. All but one of the mean basis weight measurements of the THz sensor and nuclear gauge technologies was within  $\pm 0.20\%$ . To evaluate whether the mean percent difference was significantly different from zero, a paired t-test was used. For the mid-weight samples, three of five production runs had p-values >0.05, indicating no statistically significant difference between the means of the two samples. Similar statistical testing for two of five production runs for the heavy-weight samples indicated that there was no statistically significant difference between sample means.

The nuclear gauge data were processed to provide a piece-wise linear fit response that is useful for production control. The THz sensor data were lightly smoothed using an 11-point adjacent average filter to mimic this fit to some degree, but the THz data were still more variable.

Operational Factors. Picometrix states the THz sensor is approximately 50 times smaller and lighter than the nuclear gauge enclosure. The Picometrix LLC THz sensor operates on the principal of EM reflection and refraction which makes system setup, use, and maintenance easier than for the nuclear gauge. Also, the THz system does not rely on any radiological sources and thus does not present any special safety concerns or any special procurement, use, or disposal concerns (e.g., extra disposal costs or potential exposure hazards). The laser must be replaced every 5-10 years; the laser window must be cleaned weekly, but no factory or operator actions are required after the initial vendor setup. The instrument is automatically calibrated during each scan; a NIST standard can be placed in-line monthly at the user's discretion. The THz system has diagnostics to indicate to the user if and when recalibration is required. The instrument should be adequately grounded when installed, as some occasional interruptions in operation were observed due to static electricity. The instrument requires 90-246 volt power supply and uses < 100 watts of power. Data were output digitally in real-time. The unit cost is \$220,000.

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