

Battelle

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Environmental Technology Verification Program Advanced Monitoring Systems Center

Generic Verification Protocol
for Verification of
Online Turbidimeters

ETV ✓ ETV ✓ ETV ✓

**GENERIC
VERIFICATION PROTOCOL**

for

Verification of
Online Turbidimeters

Version 1.0

June 4, 2012

Prepared by

**Battelle
505 King Avenue
Columbus, OH 43201-2693**

SECTION A
PROJECT MANAGEMENT

A1 VENDOR APPROVAL PAGE

ETV Advanced Monitoring Systems Center

Generic Verification Protocol
for Verification of
Online Turbidimeters

Version 1.0
June 4, 2012

APPROVAL:

Name _____

Company _____

Date _____

The U.S. Environmental Protection Agency, through its Office of Research and Development, funded and managed, or partially funded and collaborated in, the research described herein. It has been subjected to the Agency's peer and administrative review. Any opinions expressed in this report are those of the author(s) and do not necessarily reflect the views of the Agency, therefore, no official endorsement should be inferred. Any mention of trade names or commercial products does not constitute endorsement or recommendation for use.

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A3 LIST OF ACRONYMS AND ABBREVIATIONS

%RSD	percent relative standard deviation
ADQ	audit of data quality
AMS	Advanced Monitoring Systems
ATP	Alternate Test Procedure
COA	certificates of analysis
DI	deionized
DQIs	data quality indicators
EPA	U.S. Environmental Protection Agency
ETV	Environmental Technology Verification
L	liter
LRB	laboratory record book
NTU	nephelometric turbidity unit
MQO	measurement quality objective
PEA	performance evaluation audit
QA	quality assurance
QAPP	Quality Assurance Project Plan
QM	Quality Manager
QC	quality control
QMP	Quality Management Plan
RMO	Records Management Office
RSD	relative standard deviation
SDVB	styrene divinylbenzene
TSA	technical systems audit
VTC	Verification Test Coordinator
IR	infrared
PC	personal computer

A4 DISTRIBUTION LIST*

U.S. Environmental Protection Agency (EPA)

EPA Advanced Monitoring Systems (AMS) Center Project Officer
EPA AMS Center Quality Manager (QM)

Battelle

Battelle AMS Center Manager
Battelle Verification Test Coordinator (VTC)
Battelle AMS Center QM
Battelle Technical Staff

* Once vendors agree to participate in a verification test in this technology category, this generic protocol will be modified to be specific for the technology(ies) to be verified and then reviewed, finalized, and distributed to the following:

Vendor(s)

Peer Reviewers, at least one EPA Office of Water reviewer and one non-EPA reviewer

Reference Laboratory, if applicable

Test Collaborators (e.g., water utilities), if applicable

A5 VERIFICATION TEST ORGANIZATION

This protocol provides generic procedures for implementing a verification test for the performance of online turbidimeters. The verification tests described in this document will be conducted under the Environmental Technology Verification (ETV) Program. Verification tests will be performed by Battelle, which is managing the ETV Advanced Monitoring Systems (AMS) Center through a cooperative agreement with the EPA. The scope of the AMS Center covers verification of monitoring technologies for contaminants and natural species in air, water, and soil.

Quality Assurance (QA) oversight will be provided by the Battelle AMS Center QM and by the EPA AMS Center QM at their discretion. Based on the procedures outlined in this document, it is anticipated that verifications performed based on this generic protocol will be EPA Category III verification tests. The final determination will be made by the EPA AMS Center QM once the generic protocol is modified to be specific to the technology(ies) being verified. The organization chart in Figure 1 identifies the responsibilities of the organizations and individuals associated with these verification tests. Roles and responsibilities are defined further below.

A5.1 Battelle

Battelle's AMS Center VTC. Battelle's AMS Center VTC will have overall responsibility for ensuring that the technical, schedule, and cost goals established for the verification tests are met. Specifically, the VTC will:

- Assemble a team of qualified technical staff to conduct the verification tests,
- Hold a kick-off meeting approximately one week prior to the start of the verification tests to review the critical logistical, technical, and administrative aspects of the verification tests and confirm responsibility for each aspect of the verification test,
- Direct the team (e.g., Battelle testing staff and vendor) in performing the verification tests in accordance with the Quality Assurance Project Plan (QAPP),
- Ensure that all quality procedures specified in the QAPP and in the AMS Center Quality Management Plan¹ (QMP) are followed,

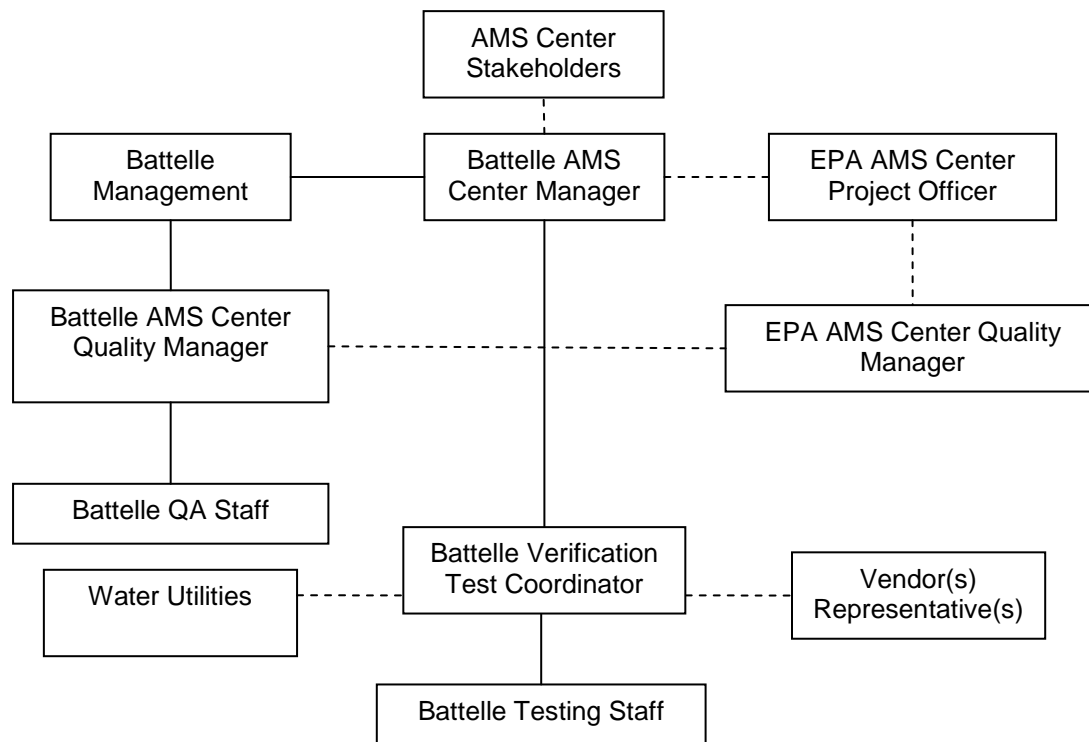


Figure 1. Organizational Chart

- Maintain real-time communication with the Battelle AMS Center Manager, Battelle AMS Center QM, EPA AMS Center Project Officer, and EPA AMS Center QM on any potential or actual deviations from the QAPP,
- Prepare the draft and final QAPP, verification report, and verification statements,
- Provide test data, including data from the first day of testing, to the Battelle AMS Center Manager, Battelle AMS Center QM, EPA AMS Center Project Officer, and EPA AMS Center QM,
- Conduct a technical review of all test data. Designate an appropriate Battelle technical staff member to review data generated by the VTC,
- Revise the draft QAPP, verification report, and verification statements in response to reviewers' comments,
- Document and prepare any deviations to the QAPP that may occur during testing,
- Address any comments from reviewers regarding testing or the deviations,

- Respond to any issues raised in assessment reports and audits, including instituting corrective action as necessary,
- Serve as the primary point of contact for vendor(s) representative(s),
- Coordinate distribution of the final QAPP, verification report(s), and statement(s), and,
- Establish a budget for the verification tests and manage staff to ensure the budget is not exceeded.

Battelle's AMS Center Manager. Battelle's manager for the AMS Center will:

- Review the draft and final QAPP,
- Review the draft and final verification report and verification statements,
- Ensure that necessary Battelle resources, including staff and facilities, are committed to the verification tests,
- Ensure that confidentiality of sensitive vendor information is maintained,
- Ensure that testing staff respond to QAPP deviations and any issues raised in assessment reports, audits, or from test staff observations, and that any necessary corrective actions have been implemented,
- Maintain communication with EPA's AMS Center Project Officer and QM, and
- Facilitate a stop work order if Battelle or EPA QA staff discover adverse findings that will compromise data quality or test results.

Battelle Testing Staff. Battelle Testing Staff will support the VTC in conducting these verification tests. Battelle Testing Staff will:

- Assist in planning for the tests, and making arrangements for the receipt of and training on the technologies,
- Attend the verification test kick-off meeting, as requested,
- Assist vendor staff as needed during technology receipt and training,
- Participate in training provided by the vendor(s), as requested,
- Conduct verification testing following all aspects of the ETV AMS Center QMP as well as this QAPP,

- Record qualitative observations about the maintenance and operation of the technology(ies) during testing,
- Ensure that the data from the technology(ies) are immediately reviewed for quality, and compiled, recorded, and transmitted to the VTC, on the first day of testing and thereafter on at least a weekly basis,
- Notify the VTC of any QAPP deviations and institute corrective action as necessary,
- Support the VTC in the preparation of the QAPP, report, and verification statements, as necessary, and
- Support the VTC in responding to any issues raised in assessment reports and audits related to technical performance, statistics, or data reduction as needed.

Battelle's AMS Center Quality Manager. The Battelle QM or a designated QA Officer will:

- Review the draft and final QAPP,
- Attend the verification test kick-off meeting and lead the discussion of the QA elements of the kick-off meeting checklist,
- Prior to the start of verification testing, verify the presence of applicable training records, including any vendor training on test equipment,
- Prepare audit checklists,
- Conduct a technical systems audit at least once near the beginning of each verification test,
- Conduct audits to verify data quality,
- Prepare and distribute an audit report for each audit,
- Verify that audit responses for each audit finding and observation are appropriate and that corrective action has been implemented effectively,
- Communicate to the VTC and/or technical staff the need for immediate corrective action if an audit identifies QAPP deviations or practices that threaten data quality,
- Provide a summary of the QA/quality control (QC) activities and results for the verification reports,
- Review the draft and final verification report and verification statements,

- Maintain real-time communication with the VTC on QA activities, audit results, and concerns,
- Recommend a stop work order if audits indicate that data quality or safety is being compromised,
- Work with the VTC and Battelle's AMS Center Manager to resolve data quality concerns and disputes,
- Delegate QA activities to other Battelle quality staff as needed to meet project schedules, and
- Review and approve QAPP amendments, deviations and audit reports.

A5.2 Vendor(s)

The vendor's responsibilities are as follows:

- Review and provide comments on the draft QAPP,
- Approve the final QAPP prior to test initiation,
- Provide the technology to be tested for evaluation during the verification tests,
- Provide all equipment/supplies/reagents/consumables needed to operate their technology for the duration of the verification tests,
- Supply a representative to train Battelle staff in operation of their technology and provide written consent for Battelle staff to operate their technology during verification testing,
- Provide written instructions for routine calibration, operation, and maintenance of their technology, and
- Review and provide comments on the draft verification report and statement for their technology.

A5.3 EPA

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

EPA AMS Center Project Officer. The EPA AMS Center Project Officer will:

- Review the draft QAPP,
- Approve the final QAPP,
- Review and approve deviations to the approved final QAPP,
- Appoint a delegate to review and approve deviations to the approved final QAPP in his absence, so that testing progress will not be delayed,
- Review the first day of data from the verification tests and provide immediate comments if concerns are identified,
- Review the draft verification report and statements,
- Oversee the EPA review process for the verification report and statements, and
- Coordinate the submission of verification report(s) and statement(s) for final EPA approval.

EPA AMS Center Quality Manager. The EPA AMS Center QM will:

- Review the draft QAPP,
- Review deviations to the approved final QAPP,
- Review the first day of data from the verification tests and provide immediate comments if concerns are identified,
- Perform at the EPA AMS Center QM's option one external technical systems audit and/or audit of data quality during the verification tests,
- Notify the EPA AMS Center Manager of the need for a stop work order if the external audit indicates that data quality or safety is being compromised,
- Prepare and distribute an assessment report summarizing results of any external audits, and
- Review the draft verification report(s) and statement(s).

A5.4 Test Facilities

Portions of this verification test will be conducted at the facilities of different water utilities. The roles of specific water utilities participating in this verification test are as follows:

- Allow facility access to vendor(s), Battelle, and EPA representatives during the scheduled verification test including set-up and tear-down operations,
- Define facility health and safety requirements to Battelle, EPA, and vendor staff who may visit the testing facility,
- Provide adequate working space during verification test,
- Provide access to adequate water flow, and
- Provide sufficient power for the simultaneous operation of all test equipment and technology(ies) being verified.

A5.5. Verification Test Stakeholders

Stakeholders for the generic protocol included:

- Rick Sakaji, East Bay Municipal Water District
- Steve Wendelken and Derek Losh, EPA Office of Water.

A QAPP will developed based on this generic protocol. The responsibilities of verification test stakeholders who will contribute to the QAPP include:

- Participate in technical panel discussions (when available) and/or review an outline of the verification tests to provide input to the test design,
- Review and provide input to the QAPP, and
- Review and provide input to the verification report and verification statements.

The names and affiliations of the verification test stakeholders will be listed in the final QAPP. One of the verification test stakeholders will be from EPA's Office of Water. If the vendor will be utilizing ETV data to have the turbidimeter recognized under the Alternative Test Procedure (ATP) program, the Office of Water representative will be one that is involved with the ATP program.

A6 BACKGROUND

A6.1 Technology Need

The ETV Program's AMS Center conducts third-party performance testing of commercially-available technologies that detect or monitor natural species or contaminants in air, water, and soil. Stakeholder committees of buyers and users of such technologies recommend technology categories, and technologies within those categories, as priorities for testing. Among the technology categories recommended for testing are turbidimeters. An ETV AMS Center test/QA plan for online turbidimeters was originally published in 1999 (<http://www.epa.gov/nrmrl/std/etv/vt-ams.html#Turbidimeters>)³. Four technologies were verified under this test/QA plan. This generic protocol builds on the original test/QA plan for turbidimeters plus adds elements of testing consistent with current approvals of online turbidimeters under the EPA ATP program.

The technologies tested under this plan are commercial turbidimeters capable of real-time monitoring of the low-level turbidity necessary to reliably assess compliance with current drinking water regulations. In such applications these turbidimeters can provide real-time continuous monitoring of water quality and allow early warning of potential non-compliance conditions, whereas grab sample analysis by standard methods is both time-consuming and non-continuous.

In order for turbidimeters to be used for compliance monitoring, the technology and method must gain acceptance under the EPA ATP program (<http://water.epa.gov/scitech/methods/cwa/atp/>). This acceptance is based on the performance of the vendor's turbidimeter against an EPA Method 180.1⁴ compliant turbidimeter. This protocol describes generic testing procedures to evaluate the performance of a turbidimeter that would be submitted by a vendor for ETV testing. The verification test will involve comparison to an online turbidimeter which is compliant with EPA Method 180.1 (Appendix A).

A.6.2 Technology Description

This section will describe the specific technology(ies) identified for ETV testing. This section will be updated for the final version of the QAPP based on the participating technology(ies).

What follows is an example of what might be included in this section; the text should be accompanied by figures, as appropriate, that illustrate the principles of technology operation.

The online turbidimeter technologies to be verified rely upon 90° light scattering (i.e. nephelometry), or forward scattering, as a means of water quality characterization. These technologies are capable of continuous monitoring and can be designed either for use directly in-line by immersion in the sample stream, or alternatively, in a by-pass mode of operation. In the case of by-pass turbidimeters, the sample stream is drawn from a larger source stream and is directed through the nephelometer for subsequent analysis, whereas the immersion turbidimeters are designed for operation through direct submersion in the source water stream. Although the overall design requirements are significantly different, the basic components of these technologies are similar.

In general, these technologies contain at least the following components:

- Light source
- Optics
- Detector.

Typically the light sources for these technologies belong to one of two distinct groups. Historically, a filtered, broadband source has been used for turbidity measurement. This type of source consists of a tungsten lamp operated at a color temperature between 2200 - 3000 °C. More recently, narrow wavelength sources, including light emitting diodes (LEDs) and lasers, with intensity maxima in the IR wavelength range have been introduced as an alternative light source for these measurements. The technologies to be tested employ one or more light sources which fit into these categories and can be configured in single or multiple beam arrangements.

Optics in these technologies are used for focusing of the incident source beam and collection of the scattered light. The detectors used are generally either photomultiplier tubes or photodiode assemblies and are chosen to match the spectral output of the light source with the peak detector

response. The technologies generally provide a digital output which can be processed remotely to allow continuous, in-situ monitoring capabilities.

A7 VERIFICATION TEST DESCRIPTION AND SCHEDULE

A final QAPP derived from this generic protocol will provide a plan for generating performance data for online turbidimeters. The data generated are intended to provide organizations and users interested in turbidimeter performance with information on the tested technology(ies) in comparison to turbidimeters compliant with EPA Method 180.1.

The overall objective of the verification test is to provide quantitative verification of the performance of online turbidimeters under realistic operational conditions. These technologies are commonly used for water quality monitoring in water treatment facilities and to help ensure compliance with drinking water regulations. For these applications, the turbidimeters must be accurate ($\pm 10\%$) relative to the reference measurement (in this case, a Method 180.1 compliant turbidimeter) used for reporting, and must be precise ($\pm 10\%$). Since these technologies are intended for use online for compliance purposes, they should be reliable and exhibit stability to avoid frequent or unscheduled offline maintenance. The verification test is designed to address and quantify these performance characteristics.

A7.1 Verification Test Description

Since turbidity is a measurement of light scattering, a number of factors can influence the responses of these technologies to a given sample solution. Instrumental design, including light source selection and geometric differences, may result in significant differences between the responses of the technology(ies) being verified and the reference measurements. Further differences may result from the variable nature of both the size and composition of particles typically found in water streams, relative to those in standard formazin or SDVB solutions. These issues will be evaluated in this verification test by utilizing a variety of samples in the test design.

Additionally, to assess the response of these technologies to both prepared solutions and to real world water samples, verification will involve both offline and online tests. The offline test will

include challenging the technologies with a series of prepared standards or other test solutions to verify performance under well controlled conditions. The online test will assess performance under realistic operating conditions by monitoring a sample stream in at least three municipal treatment facilities under typical normal operation.

Testing will consist of analyzing surface water, ground water, and fortified deionized (DI) water samples using both the turbidimeter undergoing verification as well as an online reference turbidimeter which is EPA Method 180.1 compliant. The reference turbidimeter must be specified in the final QAPP. The turbidimeters will be tested online in at least three water utilities and “offline” in a laboratory. Side-by-side measurements of turbidity using both the turbidimeter undergoing verification and a Method 180.1 compliant reference turbidimeter using the respective plant effluent will be conducted at each water utility. Offline testing of the turbidimeters will require the use of a sample recirculation system, similar to that used in the 1999 ETV test of online turbidimeters conducted at Battelle³ with the exception that grab sampling ports will not be required. In the final QAPP, Appendix B should describe and illustrate the sample recirculation system to be used in testing.

Turbidimeters will be verified for the following performance parameters (attributes):

- Accuracy,
- Precision,
- Data completeness, and
- Operational and sustainability factors.

A7.2 Proposed Testing Schedule

Table 1 shows an estimated schedule of testing and data analysis/reporting activities to be conducted in a verification test designed using this generic protocol. Data from the verification testing should be immediately checked by the testing staff. For each technology, data should be compiled, recorded, and transmitted to the VTC on the first day of testing and on a weekly basis thereafter so that any data quality issues can be rapidly identified. The VTC should post the first day of testing data for QA and EPA review within five days of test initiation and the remaining

data every two weeks thereafter. Unaudited data should include the disclaimer “has not been reviewed by Battelle QM.”

Table 1. Estimated Verification Testing Schedule

Task	Month 1	Month 2	Month 3	Month 4	Month 5	Month 6
Finalize QAPP						
Test Preparation						
Testing at Battelle						
Testing at Water Utility 1						
Testing at Water Utility 2						
Testing at Water Utility 3						
Draft report						
Final report						
Technical Systems Audits		X	X	X		
Audit of Data Quality		X		X		

A7.3 Testing Facilities

At least three water utilities will participate in the online testing of turbidimeters for verifications. Online testing must include at least one surface water source and one ground water source. Offline testing is anticipated to be conducted at Battelle’s Columbus, OH facility, although another laboratory could be utilized if the accommodations for sample recirculation system are available to support testing.

The vendor must train Battelle staff and participating staff at each utility in the operation of their turbidimeter. Battelle staff trained in the operation of the turbidimeter to be verified will set up the turbidimeter for online operation at the testing site. It is anticipated that the same operator from each participating laboratory will operate both the turbidimeter being verified and the reference turbidimeter during testing.

A8 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

The objective of these verification tests is to verify the performance of online turbidimeters against an EPA Method 180.1 compliant reference turbidimeter. The verification tests will also rely upon operator observations to assess other performance characteristics of the turbidimeters including data completeness, ease of use, and maintenance requirements.

Data quality indicators (DQIs) ensure that these verification tests provide suitable data for a robust evaluation of performance. DQIs have been established for flow meter accuracy and reference turbidimeter accuracy vs. an independent standard. The DQIs were established to ensure that data used to support the quantitative performance evaluations of turbidimeters are of sufficient quality. The DQI and quantitative acceptance criteria for these supporting measurements are defined in Table 2. Quantitative performance parameters for vendor technology performance are discussed in Section B.

Additionally, the verification tests rely in part on observations of the Battelle testing staff for assessment of the performance of the turbidimeters being tested. The requirements for these observations are described in the discussion of documentation requirements and data review, verification, and validation requirements for these verification tests.

The Battelle QM or designee will perform a technical systems audit (TSA) of laboratory testing activities to augment these QA/QC requirements. A TSA will be performed at Battelle during offline testing and at one participating utility during online testing and will occur within the first week of each testing phase. The EPA QM also may conduct an independent TSA at the EPA QM's discretion.

A9 SPECIAL TRAINING/CERTIFICATION

Documentation of training related to technology testing, data analysis, and reporting is maintained for all Battelle technical staff in training files at their respective locations. The Battelle QM may verify the presence of appropriate training records prior to the start of testing. The vendors will be required to train technical staff from Battelle and each participating utility prior to the start of testing. Battelle will document this training with a consent form, signed by the vendor, which states which staff have been trained to use the vendor's turbidimeter. In the event that other staff members are required to use the technologies, they will be trained by the Battelle staff that were trained by the vendors. Battelle technical staff supporting these verification tests have a minimum of a Bachelor's degree in a scientific field or equivalent work experience.

Table 2. DQI and Criteria for Critical Supporting Measurements

Technology	DQI	Method of Assessment	Frequency	Measurement Quality Objectives (MQO)	Corrective Action
Flow Meter	Flow Meter Accuracy	Stop watch and graduated cylinder	Once	±10%	Recalibrate or replace
Reference Turbidimeter	Reference Method Accuracy	Formazin or SDVB standard	Daily prior to testing	±10%	Recalibrate

A10 DOCUMENTATION AND RECORDS

The documents for these verification tests will include the final QAPP, vendor instructions, reference methods, the verification report, verification statement, and audit reports. The project records will include certificates of analysis (COA), chain-of-custody forms, laboratory record books (LRB), data collection forms, electronic files (both raw data and spreadsheets), and QA audit files. The final QAPP should include the forms to be used for online and offline data collection. All of these documents and records will be maintained at the laboratory, with the participating utilities, or in the VTC's office during the tests. At the conclusion of testing, all raw data and test records will be provided to the VTC. All test records and copies of supporting records from the participating utilities (and laboratory(ies), if not Battelle) will be transferred to permanent storage at Battelle's Records Management Office (RMO) at the conclusion of the verification tests. Electronic documents and records will also be uploaded to a SharePoint site designated for these tests and will be provided to EPA upon request. All Battelle LRBs are stored indefinitely by Battelle's RMO; other project-related data are stored for 10 years. EPA will be notified before disposal of any files. Section B10 further details the data recording practices and responsibilities.

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 the Battelle QM for review prior to use so that appropriate changes, if any, can be made.

SECTION B

MEASUREMENT AND DATA ACQUISITION

B1 EXPERIMENTAL DESIGN

The verification tests described in this generic protocol address verification of turbidimeters by evaluating the following performance factors:

- Accuracy,
- Precision,
- Data completeness, and
- Operational and sustainability factors.

To assess the response of these technologies to both prepared solutions and to real world water samples, verification will involve both online and offline tests. The online test will assess long-term performance under realistic operating conditions by monitoring a sample stream in a municipal treatment facility under typical normal operation. The offline test will include challenging the technologies with a series of prepared test solutions to verify performance under well controlled conditions. Comparisons will be made to an approved EPA Method 180.1 alternative online turbidimeter to assess performance relative to this standard reference method.

Both online and offline testing will involve continuous monitoring of turbidity by multiple technologies. Throughout the testing period, a PC-based data acquisition system or data logger will be used to collect measurements from the online turbidimeters at preset intervals, as needed. If a turbidimeter has its own data logging capability, then that capability will be used to record the data.

Data will be evaluated in terms of accuracy and precision for the turbidimeter undergoing testing. Accuracy will be determined as the degree of agreement of the turbidimeter undergoing testing with a reference turbidimeter; and precision will be determined as the degree of repeatability between successive measurements of the same sample. Any seemingly large differences between the turbidimeter undergoing testing and the reference turbidimeter (i.e., an approved EPA Method 180.1 alternative online turbidimeter) noted during testing will be reported immediately to the vendor so that corrective action can be taken, as necessary. Table 3

presents a summary of the tests to be performed. The verification test will be conducted during an approximate 6 month timeframe. Throughout the verification tests, each turbidimeter will be operated by Battelle staff or water utility staff that have been trained by the vendor.

Table 3. Summary of Tests and Testing Frequency

Phase	Performance Parameter	Objective	Comparison Based On	Testing Frequency	Number of Data Points
Online	Accuracy (Comparability)	Determine the degree of agreement between trends in the data from the EPA reference method	EPA 180.1 accepted alternative online turbidimeter results	3 utilities	Total of 3 surface and groundwater runs
Offline	Accuracy (Percent Error)	Determine the degree of agreement with the EPA reference method using formazin or SDVB solutions	EPA 180.1 accepted alternative online turbidimeter results	10 replicates low NTU solution and 10 replicates high NTU solution	20 replicates
Offline	Precision (RSD)	Determine the degree of repeatability between successive measurements of the same sample	Technology results	10 replicates low NTU solution and 10 replicates high NTU solution	20 replicates
Both phases	Data Completeness	Overall Amount of data returned by each technology	Technology results	Once. Based on overall data return achieved	Variable, depending on frequency of data collection by technology

B1.1 Online Testing

The online test phase will focus on assessing the accuracy and performance of the turbidimeters under realistic operating conditions, through the monitoring of typical sample streams. It is expected that natural meteorological occurrences will contribute to the variability of water quality in the treatment facility, and therefore provide a natural range of turbidity over which technology performance can be characterized.

The online phase will involve monitoring a sample stream of variable turbidity within the treatment facilities. Testing will be conducted at least three water utility facilities and at least one surface water sample and one ground water sample will be evaluated. Thus, at least three technology data sets will be collected during the online testing phase. One water utility may provide both a surface water and a groundwater sample, but from separate plants in the city. The specific location and number of water utilities to be included in the verification test will be defined in the final QAPP. The online turbidimeters will operate in parallel from the same source, not in series.

An online Method 180.1-compliant reference turbidimeter installed in parallel to the technologies being verified will be used to assess accuracy (comparability). Both the turbidimeter undergoing verification and the reference turbidimeter will be connected to the same water effluent line for analysis. The turbidimeters will either be directly connected to the effluent line within 5 feet of each other or connected to the same port in an effluent line using a y-connector to split the effluent evenly into both turbidimeters, depending on the configuration at the water utility. Both turbidimeters will operate for 24 hours, collecting data once per minute, or at the output rate of the turbidimeter. After 24 hours, the turbidimeter undergoing verification will be turned off to preserve the data for downloading. Reference turbidimeter readings collected by the water utility during this time period will be provided to Battelle within 5 days of collection. One aim of the verification test is to assess the real-world variability of the technologies being tested. To that end, measurements which may appear to be anomalous with comparable data will be retained in the data set. If an assignable cause can be identified, this cause will be described in the verification report.

B1.2 Offline Testing

The offline phase of the test will be aimed at assessing the accuracy and precision of the turbidimeters relative to the standard methods under controlled conditions. These parameters will be tested in an offline recirculation system that will enable testing of known formazin or styrene divinylbenzene (SDVB) solutions by the technologies being verified and a method 180.1-compliant online reference turbidimeter. Offline testing will be conducted at Battelle's

laboratory or a laboratory with an appropriate recirculation system as shown in Appendix B [to be included in the final QAPP].

Ten separate solutions of formazin in DI water will be prepared in individual 10 liter (L) containers using formazin or SDVB primary stock solution(s) purchased from a commercial supplier. These formazin or SDVB stock solution(s) will be diluted to the appropriate concentration in DI water. A high nephelometric turbidity unit (NTU) and a low NTU fortified DI water sample will be prepared and evaluated on each turbidimeter during offline analysis. Concentrations to be tested will be approximately 100 ‘milli’ (1/1,000 units) nephelometric turbidity units (NTU). Milli-NTU will be expressed as mNTU, or simply ‘low’ NTU. In contrast, a concentration of 800 mNTU will be set as the ‘high’ NTU. Ten replicates of each fortified DI water sample (low NTU and high NTU) will be tested simultaneously on both the technology being verified and a reference turbidimeter. Because online turbidimeters require that a continuous flow be maintained for proper operation, a sample recirculation system will be used to introduce the fortified samples to the turbidimeters.

B1.2.1 Recirculating Test System for Offline testing

The recirculation system used for this verification test will be designed and built at Battelle or the selected laboratory or location used for offline analysis. Any laboratory or location used for offline analysis will need to be able to supply and build a sufficient recirculation system. The selected location of offline testing will be defined in the final QAPP. The recirculation system will be designed and built to minimize the number of flow obstructions and potential sources of turbulence. Any valves used in the recirculation system will be either two or three way, full bore, ball valves. The recirculation pump will be a standard centrifugal pump and will have sufficient flow and pressure capabilities to meet the requirements of all the turbidimeters being verified. A flow meter will be installed downstream of each turbidimeter, and if needed, a pressure gauge will be installed downstream of the turbidimeters. In general, the tubing will be a flexible plastic material appropriate for high purity applications, and the diameter will be at least ½" to allow adequate flow for all the turbidimeters. Similarly, most connections will be made using hard plastic compression fittings, although in some cases, tubing of smaller diameter or different material, or other fittings may be used for certain portions of the system. Each turbidimeter will

be installed per the recommendations of the respective vendor. Before the test, the recirculation system will be checked by laboratory staff to ensure system integrity, including proper flow through the system, and adequate pumping capacity for recirculation. The flow rate requirements will be defined in the final QAPP.

B1.2.2 Detailed Procedure for Offline Testing

The turbidimeters will operate in parallel from the same source, not in series so that the turbidimeter(s) being verified and the EPA-approved Method 180.1 compliant reference turbidimeter are testing the same solution simultaneously. Fortified samples will be prepared as described in Section B1.2. Each test solution will be introduced individually to each turbidimeter at approximately the same flow rate ($\pm 10\%$). Flow meters will be used to monitor the flow rates of the circulation system. Testing will proceed according to the following steps:

1. Unspiked DI water will be pumped to each of the turbidimeters.
2. Once continuous flow is established through each of the turbidimeters and the readings have stabilized, a reading will be recorded from each turbidimeter onto datasheets. This reading will represent the baseline turbidity before the formazin or SDVB spike solution is added.
3. A formazin or SDVB stock solution will be added to the recirculation system at the desired concentration (low NTU or high NTU) .
4. The fortified water will be pumped to each of the turbidimeters. Once the turbidimeter readings have stabilized, a reading will be recorded from each turbidimeter onto datasheets. This reading will represent the measured turbidity after the formazin or SDVB spike.
5. Readings will not be recorded from either turbidimeter before or after the spike until both turbidimeter readings have stabilized. Stable is defined as changes in instrument read-outs of $< 10\%$ for 5 minutes.
6. Once a fortified replicate sample has been measured, both turbidimeters will be flushed clean using a container filled with clean, unspiked DI water.
7. As part of the flushing procedure, the turbidimeter sample chamber will be emptied at least 4 times to ensure that no residual fortified sample remains. Flush is sufficient when the readings for each unit return to within 5% of background (DI only) levels.

8. After both turbidimeters have been flushed clean, another replicate sample will be introduced to both turbidimeters following the same procedure previously described (Steps 3 – 6).
9. Ten replicates of each standard concentration level will be evaluated by the turbidimeters.

Results from the turbidimeters being evaluated will be recorded by the operator on data sheets supplied by Battelle or automatically by any data-logging system supplied with the turbidimeter or used by the participating water utilities.

B1.3 Data Completeness

No additional test procedures will be carried out specifically to address data completeness. This parameter will be assessed based on the overall data return achieved by each technology (Section B.1.5.3).

B1.4 Operational and Sustainability Factors

Operational and sustainability factors such as waste generated, maintenance needs, calibration frequency, data output, consumables used, power requirements, hazardous components, ease of use, repair requirements, and sample throughput will be evaluated based on operator observations. Battelle testing staff and testing staff from any participating utilities will document observations in a LRB or data sheets. Examples of information to be recorded include the daily status of diagnostic indicators for the technology, use or replacement of any consumables, the duration and causes of any technology down time or data acquisition failure, operator observations about technology startup, ease of use, clarity of the vendor's instruction manual, user-friendliness of any needed software, overall convenience of the technologies and accessories/consumables, or the number of samples that could be processed per hour or per day. Battelle will summarize these observations to aid in describing the technology performance in the verification report on each technology.

B1.5 Statistical Evaluation

The statistical methods and calculations used for evaluation of the quantitative performance parameters are described in the following sections.

B1.5.1 Accuracy

For offline testing, the relative accuracy of the results of the turbidimeter undergoing verification with respect to the Method 180.1 compliant turbidimeter results will be assessed. Relative accuracy will be determined for the standard formazin or SDVB solution using a percent error calculation, where the absolute difference between the average reference turbidimeter results and average turbidimeter undergoing verification results is divided by the average reference turbidimeter results. Accuracy results will be evaluated in regard to any criteria defined by EPA as part of the verification test to determine the turbidimeters acceptance against Method 180.1.

$$\text{Relative Percent Error} = \frac{|\text{Average Reference Turbidimeter Results} - \text{Average Technology Results}|}{\text{Average Reference Turbidimeter Results}} \quad (1)$$

The purpose of the online portion of the test will not be to determine if the Method 180.1 compliant reference turbidimeter and the turbidimeter undergoing verification provide the same turbidity readings at each interval, but to determine if the turbidimeter undergoing verification is tracking the same changes that the reference turbidimeter is reporting across the measurement period. Therefore, for the ground and surface water data from the public utilities, accuracy will be assessed by plotting the raw data for both turbidimeters on the same graph to determine how well the measurements track each other. Averages and standard deviations of the data for each turbidimeter will be reported. Based on calculations performed in the Hach FT660 protocol⁵, comparisons between the reference turbidimeter and turbidimeter undergoing verification will be conducted using non-parametric tests as appropriate.

B1.5.2 Precision

Precision will be evaluated using the replicate results for the fortified DI water samples. Precision will be reported in terms of the percent relative standard deviation (%RSD) of a group of measurement replicates. Readings from the spiked replicate samples will be blank (i.e., background)-corrected using the initial, before-spike measurements made on each replicate. The average, standard deviation, and %RSD will be calculated using these blank-corrected values for each turbidimeter at each spike level. Equations 2 and 3 will be used to calculate precision:

$$S = \left[\frac{1}{n-1} \sum_{k=1}^n (M_k - \overline{M})^2 \right]^{1/2} \quad (2)$$

where S is the standard deviation, n is the number of replicate samples, M_k is the technology measurement for the k^{th} sample, and \overline{M} is the average technology measurement of the replicate samples.

$$RSD(\%) = \left| \frac{S}{\overline{M}} \right| \times 100 \quad (3)$$

B1.5.3 Data Completeness

Data completeness will be assessed based on the overall data return achieved by the technology during the testing period. For each technology, this calculation will use the total number of apparently valid data points divided by the total number of data points potentially available from all testing. The causes of any incompleteness of data return will be established from operator observations, and noted in the discussion of data completeness results. The goal for data completeness is 100%. Any problems with the data will be brought to the attention of the VTC. The VTC will first work with the vendor to resolve any data issues. Data issues which remain will be discussed with the Battelle QM and AMS Center Manager, and EPA Project Officer and QM, as necessary.

B1.6 Reporting

The statistical comparisons described above will be conducted separately for each technology, and information on the operational performance will be compiled and reported. One verification report and one verification statement will be prepared for each technology. The verification report will present the test procedures and test data, as well as the results of the statistical evaluation of those data.

Operational aspects of the technologies will be recorded by testing staff during and immediately following testing and will be summarized in the verification report. For example, descriptions of the data acquisition procedures, use of vendor-supplied proprietary software, consumables used, repairs and maintenance needed, and the nature of any problems will be presented in the report.

The verification report will briefly describe the ETV program, the AMS Center, and the procedures used in verification testing. The results of the verification tests regarding technology performance will be stated quantitatively. The draft verification report will be reviewed by the vendor, EPA, and other peer reviewers. The resulting review comments will be addressed in a subsequent revision of the report, and the peer review comments and responses will be tabulated to document the peer review process and submitted to EPA. The reporting and review process will be conducted according to the requirements of the ETV/AMS Center QMP.¹

B2 SAMPLING METHOD REQUIREMENTS

No discrete grab samples will be collected for this test and therefore the use of traditional sample collection and handling methods are not applicable. All samples generated and analyzed for this test will be in situ samples and tested by in-line technologies. Water effluent will be tested as-is from each participating water utility.

Formazin or SDVB solutions, standards for use as calibration standards for the reference turbidimeters, and the material used for the performance evaluation audit (PEA) will be purchased from a commercial supplier (i.e., Hach Company, Loveland, CO). When available, stock solutions of the correct turbidity needed for calibration will be purchased. When not available, the standard solution will be prepared through the dilution of a purchased formazin or SDVB solution using distilled, deionized water. Preparation of diluted standard solutions will be performed within 24 hours of their use and stored at $25\pm3^{\circ}\text{C}$. For long term storage, the purchased standards will be stored as recommended by the vendor. Excess and waste solutions will be disposed of in accordance with the site procedures. When not in use, the glassware used for preparation and storage of these solutions will be kept scrupulously clean.

B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

No discrete grab samples will be collected for this test and therefore the use of traditional sample handling and custody procedures are not applicable. All solutions used in offline testing will be prepared at Battelle. The receipt of standards used for testing will be documented.

B4 ANALYTICAL METHOD REQUIREMENTS

ANPA Method 180.1-compliant online turbidimeter will be used as the reference technology for this verification test. If not available otherwise, this turbidimeter will be rented for testing and operated by Battelle (or other laboratory) for the offline tests and will be supplied and operated by the participating water utilities for the online testing. Testing using the reference turbidimeter will follow manufacturer's recommendations and EPA Method 180.1. Once the specific turbidimeter(s) is identified, detailed operational requirements will be defined in the final QAPP.

Appropriate data logging instruments will be used during offline testing to record results from the reference turbidimeter. Results will be recorded by the individual water utilities and supplied to Battelle for the online testing.

B5 QUALITY CONTROL REQUIREMENTS

Quality control procedures will follow the requirements described in this protocol, the final QAPP, EPA Method 180.1, the ETV QMP², and any vendor specified requirements for analysis using their turbidimeters. All standard values and equipment calibrations for these technologies will be documented in the study records. DQIs are defined in Table 2. Potential QC samples and measurement quality objectives (MQOs) are defined in Table 5.

B6 INSTRUMENT/ EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

Battelle staff will operate and maintain the turbidimeters as directed by the vendor during staff training and as noted in the technology operating manuals. The vendor will be consulted if issues with the technologies arise. The reference turbidimeter will be operated and maintained per the manufacturer's instructions or applicable testing facility SOPs by Battelle and the water utility staff. Critical measurements and MQOs related to operating the turbidimeters are included in Table 4.

B7 INSTRUMENT CALIBRATION AND FREQUENCY

Each reference turbidimeter used in testing will be calibrated before any testing begins in accordance with the procedures described in the manufacturer's instrument manual to ensure that the instrument is working properly. The calibration will be verified daily using a check standard in the mid-range of the initial calibration prior to testing. Calibration standards must bracket the NTU range being tested. Standard solutions necessary for calibration of the reference turbidimeter will be purchased from a commercial vendor. When available, the standards used in the calibration, or calibration check, will be purchased with the appropriate turbidity value for direct evaluation. Otherwise, the standard solution will be prepared through subsequent dilution of stock formazin or SDVB solution with DI water using Class A volumetric glassware.

Calibration for each participating technology will be performed according to the vendor's instructions.

Each flow meter will be factory calibrated and will be checked once during the verification test by measuring the time required to pass a known volume of liquid through the individual meters for a specific time period. If the calibration check indicates an error in excess of 10%, the meter will be recalibrated, when feasible, or replaced.

Table 4. Acceptance Criteria for Quality Control Samples and Turbidimeter Calibration

Test	Method of Assessment	Frequency	Measurement Quality Objective (MQO)	Corrective Action
Reference turbidimeter calibration (if needed)	Initial Calibration Linearity test or as specified by manufacturer	Initially prior to testing	$0.90 < \text{slope} < 1.10$, $r^2 < 0.98$ Or Per manufacturer's instructions	Recalibrate
Reference turbidimeter calibration check ¹	Formazin or SDVB check standard	Daily prior to use	$\pm 10\%$	Recalibrate; repeat measurements if calibration not met after tests
Reference turbidimeter Reagent Blank	Deionized water	Prior to each test and between offline replicate test solutions	≤ 5 times the reference turbidimeter detection limit ²	Continue flushing Replace deionized water
Reference turbidimeter Quality Control Sample	AMCO-AEPA-1 standard solution or independent formazin or SDVB solution	Once prior and quarterly	$\pm 10\%$ of stated value	Performance maintenance, re-calibrate, and repeat measurement
Tested turbidimeter	Per vendor	Once or per vendor's instructions	Per vendor	Recalibrate
Reagent Blank	Deionized water	Prior to each test and between offline replicate test solutions	$\pm 5\%$ of initial DI water reading	Continue flushing Replace deionized water

¹This check confirms the DQI listed in Table 2.

²To be determined once the reference turbidimeter model is identified.

B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

Upon receipt of any supplies or consumables used for testing, Battelle will visually inspect and ensure that the materials received are those that were ordered and that there are no visual signs of damage that could compromise the suitability of the materials. If damaged or inappropriate goods are received they will be returned or disposed of and arrangements will be made to receive replacement materials. COA or other documentation provided with all reagents and standards will be checked to ensure suitability for these verification tests. Unsuitable materials will be returned or disposed of and arrangements for the receipt of replacement materials will be made.

B9 NON-DIRECT MEASUREMENTS

Non-direct measurements will not be used during these verification tests.

B10 DATA MANAGEMENT

Various types of data will be acquired and recorded electronically or manually by Battelle staff and staff from participating utilities during these verification tests. All manually-recorded data, such as solution preparation records and results from supporting analyses, will be recorded according to Section B10. Table 5 summarizes the types of data to be recorded. All maintenance activities, repairs, calibrations, and operator observations relevant to the operation of the monitoring systems being tested will be documented by Battelle staff or staff from participating utilities in an LRB or on data sheets. Report formats will include all necessary data to allow traceability from the raw data to final results.

Records received by or generated by any Battelle staff or staff from participating utilities during testing will be reviewed by a Battelle staff member within five days of receipt or generation, respectively, 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 received or 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. Some of the checks that will be performed include:

- QC samples and calibration standards were analyzed according to the QAPP and the acceptance criteria were met. Corrective action for exceedances was taken,
- 100% hand-entered and/or manually calculated data were checked for accuracy,
- Calculations performed by software are verified at a frequency sufficient to ensure that the formulas are correct, appropriate, and consistent,
- For each cut and paste function, the first and last data value was verified vs. the source data,
- Data are reported in the units specified in the QAPP, and

- Results of QC samples are reported.

Calculations to be checked include any statistical and concentration calculations described in the QAPP. A dedicated shared folder within the ETV AMS Center SharePoint site will be established for all project records.

Battelle will provide technology test data (including records, data sheets, and notebook records) from the first day of testing within five days of generation to EPA for simultaneous review. Thereafter, the data will be provided to EPA every two weeks. The goal of this data delivery schedule is prompt identification and resolution of any data collection or recording issues. These data will be labeled as preliminary and may not have had a QA review before their release.

Table 5. Summary of Data Recording Process

Data to Be Recorded	Where Recorded	How Often Recorded	By Whom	Disposition of Data
Dates and details of test events	ETV LRBs or data forms	Start/end of test event	Battelle staff Staff from participating utilities	Used to organize/check test results; manually incorporated in data spreadsheets as necessary
Technology operator/analyst, data collection and analysis dates, sample volume and/or time, sample description	ETV LRBs or electronically	When performed	Battelle staff Staff from participating utilities Battelle staff	Incorporated in verification report as necessary
Technology and reference test calibration information, reagent and test solution information,	ETV LRBs or electronically	When performed	Battelle staff Staff from participating utilities Battelle staff	Incorporated in verification report as necessary
Turbidimeter readings	ETV LRBs or electronically	Each measurement initiated by testing staff	Battelle staff Staff from participating utilities Battelle staff	Converted to spreadsheet for statistical analysis and comparisons

SECTION C

ASSESSMENT AND OVERSIGHT

C1 ASSESSMENTS AND RESPONSE ACTIONS

Every effort will be made in these verification tests to anticipate and resolve potential problems before the quality of performance is compromised. One of the major objectives of the QAPP is to establish mechanisms necessary to ensure this. Internal quality control measures described in the final 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. Technical staff and the VTC will work with the Battelle QM to resolve any issues. Action will be taken by the VTC and Battelle testing staff to identify and appropriately address the issue, 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 these verification tests.

C1.1 Performance Evaluation Audit

A PEA will be conducted to verify the accuracy of reference turbidimeter readings which will be the basis of determining technology accuracy. A separate PEA will be conducted for each reference turbidimeter used in testing.

The PEA sample will be analyzed after routine maintenance and calibration of the reference turbidimeters by analyzing a standard formazin or SDVB solution and comparing the results to a reference that is independent of standards used during the test (i.e., AMCO-AEPA-1 standard

solution). Agreement between the formazin or SDVB solution turbidity readings and AMCO-AEPA-1 must be within 10% for each reference turbidimeter. If this criterion is not met, the reference turbidimeter must be recalibrated.

C1.2 Technical Systems Audits

The Battelle QM or designee will perform a TSA at Battelle during offline testing and at one participating utility during online testing. The purpose of these audits is to ensure that the verification tests are being performed in accordance with the AMS Center QMP¹ and the QAPP. The Battelle QM will compare actual test procedures to those specified or referenced in this plan, and review data acquisition and handling procedures. The Battelle QM or designee will prepare a project-specific checklist based on the QAPP requirements to guide the TSA, which will include a review of the test location and general testing conditions; observe the testing activities; and review test documentation. The Battelle QM will also check data acquisition procedures, and confer with testing staff. The Battelle QM 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 QM 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 tests. The TSA findings will be communicated to technical staff at the time of the audit and documented in the TSA reports.

C1.3 Data Quality Audits

As an EPA QA Category III test, the Battelle QM, or designee, will audit at least 10% of the sample results data acquired in the verification tests and 100% of the calibration and QC data versus the QAPP requirements. Two Audits of Data Quality (ADQs) will be conducted for this project: Data collected on the first day of testing for each technology will be audited within 10 business days of receipt and assessed using a project-specific checklist. The remaining data will be audited at the conclusion of testing and will be completed within 10 business days of receipt of all test data. During these audits, the Battelle QM, or designee, will trace the data from initial acquisition (as received from the vendor's technology), through reduction and statistical comparisons, to final reporting. All calculations performed on the data undergoing the ADQ will

be checked. Data must undergo a 100% validation and verification by technical staff (i.e., VTC or designee) before it is 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 QM 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. A final ADQ that assesses overall data quality, including accuracy and completeness of the technical report, will be prepared as a narrative and distributed to the VTC and EPA within 10 business days of completion of the audit.

C1.4 QA/QC Reporting

Each assessment and audit will be documented in accordance with Section 3.3.4 of the AMS Center QMP.¹ The results of all audits will be submitted to EPA within 10 business days as noted above. Assessment reports will include the following:

- Identification of Findings and Observations,
- Recommendations for resolving problems,
- Response to adverse findings or potential problems,
- Confirmation that solutions have been implemented and are effective, and
- 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 QM 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 6. If serious quality problems exist, the Battelle QM 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 QM will ensure that follow-up corrective action has been taken. The QAPP and final report are reviewed by the EPA AMS Center QM 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 6. Summary of Quality Assessment and Control Reports¹

Assessment	Prepared By	Report Submission Timeframe	Submitted To
Technology Offline Testing TSA (within the first week of testing)	Battelle	10 business days after TSA is complete ² 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
Technology Online Testing TSA (within the first week of testing)	Battelle	10 business days after TSA is complete ² 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 (Day 1 data) each technology	Battelle	ADQ will be completed within 10 business days after receipt of first data set	EPA ETV AMS Center
ADQ (Remaining 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.

² A separate TSA report will be prepared for each technology; the report submission timeframe is the same for each.

SECTION D

DATA VALIDATION AND USABILITY

D1 DATA REVIEW, VERIFICATION, AND VALIDATION REQUIREMENTS

The key data review and data verification requirements for these tests are stated in Section B10 of this protocol. In general, the data review requirements specify that data generated during these tests will be reviewed by a Battelle technical staff member within five days of generation of the data. 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 the data have been recorded, transmitted and processed properly. Furthermore, this process will ensure that the monitoring systems data were collected under appropriate testing.

The data validation requirements for these tests involve an assessment of the quality of the data relative to the DQIs and MQOs for these tests referenced in Tables 2 and 5. 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 protocol. 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, such as “wn” for wrong number, and the initials of the recorder and date of entry). Electronic data from technology, if applicable, and any other analytical equipment used during the test will be inspected to ensure proper transfer from the data logging system. 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 inspection of the equations used for the calculations and verification of selected calculations by handheld calculator. Calculations performed using specialized commercial software (i.e., for

analytical instrumentation) will be reviewed by inspection and, when feasible, verified by handheld calculator, or standard commercial office software.

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

An ADQ will be conducted by the Battelle QM to ensure that data review, verification, and validation procedures were completed, and to assure the overall quality of the data.

D3 RECONCILIATION WITH USER REQUIREMENTS

This purpose of these verification tests is to verify the performance of turbidimeters compared to an online turbidimeter which is compliant with EPA Method 180.1. To meet the requirements of the user community, input on the tests described in the final QAPP will be provided by external experts. Additional performance data regarding operational characteristics of the evaluated turbidimeters will be collected by verification test personnel. To meet the requirements of the user community, these data will include thorough documentation of the performance of the technologies during the verification tests. The data review, verification, and validation procedures described above will assure that data meeting these requirements are accurately presented in the verification reports generated from this test, and will assure that data not meeting these requirements will be appropriately flagged and discussed in the verification reports.

This protocol and the resulting ETV verification report will be reviewed by the vendor, EPA, and expert peer reviewers. The reviews of the QAPP will help to improve the design of the

verification tests and the resulting report such that they better meet the needs of potential users of these technologies.

SECTION E

REFERENCES

1. Battelle, Quality Management Plan for the ETV Advanced Monitoring Systems Center, Version 8.0, U.S. EPA Environmental Technology Verification Program, prepared by Battelle, Columbus, Ohio, April 2011.
2. U.S. EPA, Environmental Technology Verification Program Quality Management Plan, EPA Report No: 600/R-08/009 EPA/600/R-03/021, U.S. Environmental Protection Agency, Cincinnati, Ohio, January 2008.
3. "Test/QA Plan for Verification of Online Turbidimeters," U.S. Environmental Protection Agency Environmental Technology Verification Program, prepared by Battelle, Columbus, OH, June 1999.
4. EPA Method 180.1 Turbidity (Nephelometric), Methods for the Determination of Inorganic Substances in Environmental Samples EPA-600-R-93-100. 1993.
5. Method Validation Study Plan, Hach Method 10133, Determination of Turbidity by Laser Nephelometry Revision 2, as part of the DynCorp Memorandum (Subject: D99-0002:Hach Filter Trak Turbidity Method 10133), Hach Company, January 10, 2000.

APPENDIX A

EPA Method 180.1

METHOD 180.1

DETERMINATION OF TURBIDITY BY NEPHELOMETRY

Edited by James W. O'Dell
Inorganic Chemistry Branch
Chemistry Research Division

Revision 2.0
August 1993

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METHOD 180.1

DETERMINATION OF TURBIDITY BY NEPHELOMETRY

1.0 SCOPE AND APPLICATION

- 1.1 This method covers the determination of turbidity in drinking, ground, surface, and saline waters, domestic and industrial wastes.
- 1.2 The applicable range is 0-40 nephelometric turbidity units (NTU). Higher values may be obtained with dilution of the sample.

2.0 SUMMARY OF METHOD

- 2.1 The method is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the higher the turbidity. Readings, in NTU's, are made in a nephelometer designed according to specifications given in Sections 6.1 and 6.2. A primary standard suspension is used to calibrate the instrument. A secondary standard suspension is used as a daily calibration check and is monitored periodically for deterioration using one of the primary standards.
 - 2.1.1 Formazin polymer is used as a primary turbidity suspension for water because it is more reproducible than other types of standards previously used for turbidity analysis.
 - 2.1.2 A commercially available polymer primary standard is also approved for use for the National Interim Primary Drinking Water Regulations. This standard is identified as AMCO-AEPA-1, available from Advanced Polymer Systems.

3.0 DEFINITIONS

- 3.1 **Calibration Blank (CB)** -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogates analytes.
- 3.2 **Instrument Performance Check Solution (IPC)** -- A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.
- 3.3 **Laboratory Reagent Blank (LRB)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method

analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

- 3.4 **Linear Calibration Range (LCR)** -- The concentration range over which the instrument response is linear.
- 3.5 **Material Safety Data Sheet (MSDS)** -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.
- 3.6 **Primary Calibration Standard (PCAL)** -- A suspension prepared from the primary dilution stock standard suspension. The PCAL suspensions are used to calibrate the instrument response with respect to analyte concentration.
- 3.7 **Quality Control Sample (QCS)** -- A solution of the method analyte of known concentrations that is used to fortify an aliquot of LRB matrix. The QCS is obtained from a source external to the laboratory, and is used to check laboratory performance.
- 3.8 **Secondary Calibration Standards (SCAL)** -- Commercially prepared, stabilized sealed liquid or gel turbidity standards calibrated against properly prepared and diluted formazin or styrene divinylbenzene polymers.
- 3.9 **Stock Standard Suspension (SSS)** -- A concentrated suspension containing the analyte prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source. Stock standard suspension is used to prepare calibration suspensions and other needed suspensions.

4.0 **INTERFERENCES**

- 4.1 The presence of floating debris and coarse sediments which settle out rapidly will give low readings. Finely divided air bubbles can cause high readings.
- 4.2 The presence of true color, that is the color of water which is due to dissolved substances that absorb light, will cause turbidities to be low, although this effect is generally not significant with drinking waters.
- 4.3 Light absorbing materials such as activated carbon in significant concentrations can cause low readings.

5.0 **SAFETY**

- 5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable.
- 5.2 Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in

this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

- 5.3 Hydrazine Sulfate (Section 7.2.1) is a carcinogen. It is highly toxic and may be fatal if inhaled, swallowed, or absorbed through the skin. Formazin can contain residual hydrazine sulfate. Proper protection should be employed.

6.0 EQUIPMENT AND SUPPLIES

- 6.1 The turbidimeter shall consist of a nephelometer, with light source for illuminating the sample, and one or more photo-electric detectors with a readout device to indicate the intensity of light scattered at right angles to the path of the incident light. The turbidimeter should be designed so that little stray light reaches the detector in the absence of turbidity and should be free from significant drift after a short warm-up period.
- 6.2 Differences in physical design of turbidimeters will cause differences in measured values for turbidity, even though the same suspension is used for calibration. To minimize such differences, the following design criteria should be observed:
- 6.2.1 Light source: Tungsten lamp operated at a color temperature between 2200-3000°K.
- 6.2.2 Distance traversed by incident light and scattered light within the sample tube: Total not to exceed 10 cm.
- 6.2.3 Detector: Centered at 90° to the incident light path and not to exceed $\pm 30^\circ$ from 90°. The detector, and filter system if used, shall have a spectral peak response between 400 nm and 600 nm.
- 6.3 The sensitivity of the instrument should permit detection of a turbidity difference of 0.02 NTU or less in waters having turbidities less than 1 unit. The instrument should measure from 0-40 units turbidity. Several ranges may be necessary to obtain both adequate coverage and sufficient sensitivity for low turbidities.
- 6.4 The sample tubes to be used with the available instrument must be of clear, colorless glass or plastic. They should be kept scrupulously clean, both inside and out, and discarded when they become scratched or etched. A light coating of silicon oil may be used to mask minor imperfections in glass tubes. They must not be handled at all where the light strikes them, but should be provided with sufficient extra length, or with a protective case, so that they may be handled. Tubes should be checked, indexed and read at the orientation that produces the lowest background blank value.
- 6.5 Balance -- Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.6 Glassware -- Class A volumetric flasks and pipets as required.

7.0 REAGENTS AND STANDARDS

7.1 Reagent water, turbidity-free: Pass deionized distilled water through a 0.45µ pore size membrane filter, if such filtered water shows a lower turbidity than unfiltered distilled water.

7.2 Stock standard suspension (Formazin):

7.2.1 Dissolve 1.00 g hydrazine sulfate, $(\text{NH}_2)_2\text{H}_2\text{SO}_4$ (CASRN 10034-93-2) in reagent water and dilute to 100 mL in a volumetric flask. **CAUTION--** carcinogen.

7.2.2 Dissolve 10.00 g hexamethylenetetramine (CASRN 100-97-0) in reagent water and dilute to 100 mL in a volumetric flask. In a 100 mL volumetric flask, mix 5.0 mL of each solution (Sections 7.2.1 and 7.2.2). Allow to stand 24 hours at $25 \pm 3^\circ\text{C}$, then dilute to the mark with reagent water.

7.3 Primary calibration standards: Mix and dilute 10.00 mL of stock standard suspension (Section 7.2) to 100 mL with reagent water. The turbidity of this suspension is defined as 40 NTU. For other values, mix and dilute portions of this suspension as required.

7.3.1 A new stock standard suspension (Section 7.2) should be prepared each month. Primary calibration standards (Section 7.3) should be prepared daily by dilution of the stock standard suspension.

7.4 Formazin in commercially prepared primary concentrated stock standard suspension (SSS) may be diluted and used as required. Dilute turbidity standards should be prepared daily.

7.5 AMCO-AEPA-1 Styrene Divinylbenzene polymer primary standards are available for specific instruments and require no preparation or dilution prior to use.

7.6 Secondary standards may be acceptable as a daily calibration check, but must be monitored on a routine basis for deterioration and replaced as required.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with turbidity free water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

8.2 No chemical preservation is required. Cool sample to 4°C .

- 8.3 Samples should be analyzed as soon as possible after collection. If storage is required, samples maintained at 4°C may be held for up to 48 hours.

9.0 QUALITY CONTROL

- 9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and analysis of laboratory reagent blanks and other solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of data generated.

9.2 INITIAL DEMONSTRATION OF PERFORMANCE.

- 9.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS).
- 9.2.2 Linear Calibration Range (LCR) -- The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by $\pm 10\%$, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.
- 9.2.3 Quality Control Sample (QCS) -- When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analysis of a QCS. If the determined concentrations are not within $\pm 10\%$ of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before continuing with on-going analyses.

9.3 ASSESSING LABORATORY PERFORMANCE

- 9.3.1 Laboratory Reagent Blank (LRB) -- The laboratory must analyze at least one LRB with each batch of samples. Data produced are used to assess contamination from the laboratory environment.
- 9.3.2 Instrument Performance Check Solution (IPC) -- For all determinations, the laboratory must analyze the IPC (a mid-range check standard) and a calibration blank immediately following daily calibration, after every tenth sample (or more frequently, if required) and at the end of the sample run. Analysis of the IPC solution and calibration blank immediately following calibration must verify that the instrument is

within $\pm 10\%$ of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within $\pm 10\%$. If the calibration cannot be verified within the specified limits, reanalyze the IPC solution. If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift the instrument recalibrated. All samples following the last acceptable IPC solution must be reanalyzed. The analysis data of the calibration blank and IPC solution must be kept on file with the sample analyses data. NOTE: Secondary calibration standards (SS) may also be used as the IPC.

- 9.3.3 Where additional reference materials such as Performance Evaluation samples are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Turbidimeter calibration: The manufacturer's operating instructions should be followed. Measure standards on the turbidimeter covering the range of interest. If the instrument is already calibrated in standard turbidity units, this procedure will check the accuracy of the calibration scales. At least one standard should be run in each instrument range to be used. Some instruments permit adjustments of sensitivity so that scale values will correspond to turbidities. Solid standards, such as those made of lucite blocks, should never be used due to potential calibration changes caused by surface scratches. If a pre-calibrated scale is not supplied, calibration curves should be prepared for each range of the instrument.

11.0 PROCEDURE

- 11.1 Turbidities less than 40 units: If possible, allow samples to come to room temperature before analysis. Mix the sample to thoroughly disperse the solids. Wait until air bubbles disappear then pour the sample into the turbidimeter tube. Read the turbidity directly from the instrument scale or from the appropriate calibration curve.
- 11.2 Turbidities exceeding 40 units: Dilute the sample with one or more volumes of turbidity-free water until the turbidity falls below 40 units. The turbidity of the original sample is then computed from the turbidity of the diluted sample and the dilution factor. For example, if 5 volumes of turbidity-free water were added to 1 volume of sample, and the diluted sample showed a turbidity of 30 units, then the turbidity of the original sample was 180 units.
- 11.2.1 Some turbidimeters are equipped with several separate scales. The higher scales are to be used only as indicators of required dilution volumes to reduce readings to less than 40 NTU.

Note: Comparative work performed in the Environmental Monitoring Systems Laboratory - Cincinnati (EMSL-Cincinnati) indicates a progressive error on sample turbidities in excess of 40 units.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Multiply sample readings by appropriate dilution to obtain final reading.

12.2 Report results as follows:

<u>NTU</u>	<u>Record to Nearest:</u>
0.0 - 1.0	0.05
1 - 10	0.1
10 - 40	1
40 - 100	5
100 - 400	10
400 - 1000	50
>1000	100

13.0 METHOD PERFORMANCE

13.1 In a single laboratory (EMSL-Cincinnati), using surface water samples at levels of 26, 41, 75, and 180 NTU, the standard deviations were ± 0.60 , ± 0.94 , ± 1.2 , and ± 4.7 units, respectively.

13.2 The interlaboratory precision and accuracy data in Table 1 were developed using a reagent water matrix. Values are in NTU.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American

15.0 WASTE MANAGEMENT

- 15.1 The U.S. Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water and land by minimizing and controlling all releases from hoods, and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Section 14.3.

16.0 REFERENCES

1. Annual Book of ASTM Standards, Volume 11.01 Water (1), Standard D1889-88A, p. 359, (1993).
2. Standard Methods for the Examination of Water and Wastewater, 18th Edition, pp. 2-9, Method 2130B, (1992).

17.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

TABLE 1. INTERLABORATORY PRECISION AND ACCURACY DATA

Number of Values Reported	True Value (T)	Mean (X)	Residual for X	Standard Deviation (S)	Residual for S
373	0.450	0.4864	0.0027	0.1071	-0.0078
374	0.600	0.6026	-0.0244	0.1048	-0.0211
289	0.65	0.6931	0.0183	0.1301	0.0005
482	0.910	0.9244	0.0013	0.2512	0.1024
484	0.910	0.9919	0.0688	0.1486	-0.0002
489	1.00	0.9405	-0.0686	0.1318	-0.0236
640	1.36	1.3456	-0.0074	0.1894	0.0075
487	3.40	3.2616	-0.0401	0.3219	-0.0103
288	4.8	4.5684	-0.0706	0.3776	-0.0577
714	5.60	5.6984	0.2952	0.4411	-0.0531
641	5.95	5.6026	-0.1350	0.4122	-0.1078

REGRESSIONS: $X = 0.955T + 0.54$, $S = 0.074T + 0.082$

APPENDIX B

Recirculation System Schematic

[to be completed for final QAPP]

APPENDIX C

Example Data Sheets

[to be completed for final QAPP]