Protocols for Verifying the Performance of In Situ Turbidity Sensor



May 3, 2006

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Protocols for the ACT Verification of In Situ Turbidity Sensors

1. Background on ACT Technology Evaluations

Instrument performance verification is necessary to enable effective existing technologies to be recognized and so that promising new technologies can be made available to support coastal science, resource management and the long-term development of an Integrated Ocean Observing System. The Alliance for Coastal Technologies (ACT) has therefore been established to provide an unbiased, third party testbed for evaluating new and developing coastal sensors and sensor platforms for use in coastal environments.

The following protocols describe how ACT will verify the environmental performance characteristics of commercial-ready, in situ turbidity sensors through the evaluation of objective and quality assured data. The goal of this evaluation program is to provide technology users with an independent and credible assessment of instrument performance in a variety of environments. Therefore, the data and information on performance characteristics will cover legitimate information that users need. ACT will not simply verify vendor claims, but instead looks to the broader community to define the data and operational parameters that are valuable in guiding instrument purchase and deployment decisions.

It is important to note that <u>ACT does not certify technologies</u> or guarantee that a technology will always, or under circumstances other than those used in testing, operate at the levels verified. ACT does not seek to determine regulatory compliance; does not rank technologies or compare their performance; does not label or list technologies as acceptable or unacceptable; and does not seek to determine "best available technology" in any form. ACT will avoid all potential pathways to picking "winners and losers". Therefore, although the following protocols will apply to all instruments evaluated, no direct comparisons will be made between instruments from different manufacturers and instrument-specific Verification Statements will be released to the public for each instrument type as a final report.

2. Introduction to Technology

As part of our service to the coastal community, ACT Partner Institutions and Stakeholder Council have chosen the performance verification of commercially available, in situ turbidity sensors as the third ACT Technology Evaluation. Turbidity is a property commonly used to describe water clarity in both marine and freshwater environments, providing a gross assessment of light attenuation due to suspended material. However, turbidity is often not a direct measure of the quantity of interest, such as suspended sediment, living particles, and nonliving organic matter, but rather a measure of the effect of the desired quantity on the optical properties of the water. At present, there are numerous methods for quantifying turbidity (e.g., light attenuation, surface scatter, side scatter, laser diffraction, acoustic back-scatter, etc.). Differences in methods of measurement and their individual responses to varying types of suspended material have made the measurement of turbidity difficult to perform in a consistent and standardized way. This has necessitated many public-service agencies (e.g., USGS, US EPA, ISO, ASTM, etc.) to define turbidity in very specific terms based on optical methods of measurement, since optically-based approaches have been the most conventionally used. Although such standards and definitions were created to be both technically and legally specific (thereby minimizing the ambiguity in interpreting what turbidity is and how it is measured), they still suffer from fundamental deficiencies in their ability create an absolute standard between different natural water types and different instrument designs employing the exact same principles of measurement. Despite these limitations, a variety of in situ instruments that provide some measure of turbidity are commonly and successfully used in many researcher and monitoring settings as at least a relative measure of water clarity.

This ACT Technology Evaluation will examine individual sensor performance both in the laboratory and across different field conditions. We will focus specifically on commonly used back- and side-scattering optical instruments that provide values for turbidity in Nephelometric Turbidity Units (NTU). This unit of measurement pertains to a specific concentration of a given standard medium.

3. Objectives and Focus of the In Situ Turbidity Sensor Performance Verification

ACT has performed a customer needs and use assessment for in situ turbidity sensors (www.act-us.info/customer_needs.php). Scientists, resource managers, and other users of these technologies were asked to respond to a questionnaire regarding their current use or application of these instruments, limitations or problems with their current in situ turbidity sensors, and the important parameters they use when selecting an instrument. The results of this assessment were used to identify the main applications and key parameters that ACT will evaluate in this Performance Verification.

Most users of turbidity sensors surveyed deploy instruments on remote platforms in estuarine and near shore environments, in relativity shallow waters. Therefore, the performance verification will focus on this application. It was also clear from the survey that reliability, accuracy, range/detection limits, precision, and calibration life are the most important parameters guiding instrument selection decisions. The verifications will therefore also focus on these areas of performance.

Finally, based on recommendations from the September 2005 ACT Workshop on "Measures of Turbidity in Coastal Waters" (www.act-us.info/workshops_reports.php), it was decided that while laboratory tests can evaluate instrument performance against known standards (see below), there is no true standard (or single quantifiable variable) to measure instrument performance against when tested in the field. Therefore, while an appropriate suite of environmental parameters will be monitored to characterize the field test conditions that may influence turbidity measurements (see below), instrument performance will not be compared to any particular parameter.

3.1. Parameters to be Verified

Because of the inherent limitations of in situ turbidity sensors and the inability to control various factors that can impact the data during field tests, accuracy, response linearity, precision and range will be determined in the laboratory only. Field tests will focus on reliability/stability and the ability of the instrument to track natural changes in the environment.

• Accuracy – combination of bias and precision of an analytical procedure, which reflects the closeness of the measured value to the true value. Accuracy will be determined in the laboratory by allowing test instruments to record 10 replicate values when exposed to five distinct concentrations of formazin and five distinct concentrations of submicron styrene divinyl benzene polymer beads (SDVB; GFS Chemicals, Inc.) suspensions.

- Response Linearity Stability of a predetermineSd response or calibration factor, computed as: (NTU measured in sample solution NTU measurement in blank solution) / [reference standard] over a range of reference standard concentrations. Response factors will be quantified in the laboratory for each formazin and SDVB concentration series and under varying ambient light conditions.
- **Precision** Precision is a measure of the repeatability of a measurement. Instrument precision will be determined by calculating the coefficient of variation (STD/Mean x 100) of 30 replicate turbidity sensor measurements separately at three different formazin and SDVB concentrations under total darkness.
- **Range** Range is a measure of the minimum (or detection limit) and maximum concentration of specific formazin and SDVB concentration the instrument can accurately (see definition above) measure under total darkness.
- **Reliability** Reliability is the ability to maintain integrity or stability of the instrument and data collections over time. Reliability of instruments will be determined in two ways. In both laboratory and field tests, comparisons will be made of the percent of data recovered versus percent of data expected. In field tests, instrument stability will be determined by pre- and post-measures of blanks and reference standards (formazin and SDVB) to quantify drift during deployment periods. Comments on the physical condition of the instruments (e.g., physical damage, flooding, corrosion, battery failure, etc.) will also be recorded.

4. Summary of Basic Verification Approach

The protocols are based on an amalgamation of methods for sensor calibration and testing provided by the manufacturers participating in this ACT Performance Verification, on ISO 7027:1999, and on suggestions from the Technical Advisory Committee. Initial generic protocols were further refined through direct discussions during an ACT Turbidity Sensor Performance Verification Workshop held on 6-7 February, 2006. Participants of this workshop included ACT Headquarters Staff, ACT Partner Institution Technical Coordinators, ACT Quality Assurance Manager, a Turbidity Technical Advisory Committee, and representatives from the participating manufacturers. It was decided that the protocols will follow a format that:

- employs formazin and SDVB as the standard for determining instrument performance characteristics in controlled laboratory tests, and
- includes field tests to evaluate performance under a variety of environmental conditions.

Qualified personnel affiliated with ACT will conduct all tests. All personnel involved in this verification exercise will be properly trained on use of instruments by manufacturer representatives and on a standardized water sampling, storage, analysis and shipping method.

Laboratory results will be presented as:

- means, standard deviations (SD), and number of replicates (n) of instrument measurements in NTU (raw voltage will also be presented when possible); and
- corresponding reference standard concentration or known NTU value.

Field data will be presented as:

- means, standard deviations (SD), and number of replicates (n) of instrument measurements in NTU (raw voltage will also be presented when possible), over time;
- associated physical conditions (e.g., temperature, salinity, TSS) over time.

When required, raw values from test instruments will be converted to produce derived NTU values using the specific calibration or correction factors that individual manufacturers would suggest to any of their customers. We also acknowledge that the range of field environments is constrained due to testing and time limitations, but locations are widely geographically distributed and spring/summer field conditions will have significant biofouling pressure.

The goal of this Performance Verification is to test the same model instruments in the laboratory and in a moored field application. It is also preferred to evaluate instruments incorporated in stand-alone packages, which include features such as data logging, data transformation/conversion equations, independent power, and biofouling prevention. However, in some cases, certain test instruments will be incorporated into other associated equipment (e.g., datalogger) owned and operated by ACT Partner Institutions. Instruments will be set up by the manufactures (prior to any testing) to measure turbidity that will be typically in the range of 10 to 0 NTU and to provide values at 1-minute (lab tests) or 15-minute (field tests) intervals. How each 1-minute or 15-minute value is calculated by the individual instruments (e.g., integration of multiple readings over several seconds) will be left to the manufacturer to decide and program.

A total of four sensors of each particular model will be evaluated during this verification. For the laboratory exercise, one instrument of each model will be randomly selected for testing. The moored field tests will use all four instruments, conducted over the course of two separate evaluation periods. The first will include the test instruments deployed simultaneously at four ACT Partner sites followed by a second set of evaluations at the remaining ACT Partner sites (seven total moored deployments).

4.1. Laboratory Tests

Laboratory tests of accuracy, response linearity, precision, range, and reliability will be conducted at Moss Landing Marine Laboratories, an ACT Partner Institution. The various conditions below will be produced in well-mixed (submersible circulating pumps), temperature controlled (monitored at two locations in each bath) water baths where instruments will be submerged for testing. Instrument output will be calibrated as suggested by each manufacturer.

Although field tests will include instrument deployments under varying salinity conditions, it was decided that salinity would not be tested as a variable in the laboratory evaluations. Therefore, all laboratory tests will be conducted in filtered, bubble-free, deionized water.

Instrument Setup - Prior to deployment, all instruments will be setup and calibrated as suggested in individual manufacturer manuals. The turbidity sensors will then be programmed to record data every 1 minute during the laboratory tests and their internal clocks set to GMT using www.time.gov as the time standard.

Accuracy and Response Linearity – For the accuracy and linearity/stability tests, a mean and standard deviation of 10 instrument readings at 1-minute intervals for each test condition

will be collected after the instruments are allowed at least 30 minutes to equilibrate (since response time is not a parameter being tested as part of this evaluation). This instrument mean and SD will be compared to known NTU values of the test standards. Test baths will be filled with filtered, bubble-free DI water (to provide a baseline) and held in the dark. In separate trials, instruments will then be exposed to formazin and SDVB in a 15 °C bath, at the following concentrations: 0.2, 0.5, 1, 2, 4, 6, 8, 10, 50, 100, 250, and 500 NTU. A range of concentrations from 0.2 to 100 NTU will also be examined at 4 and 32 °C to produce a matrix with approximately 80 independent conditions for evaluating response linearity.

Precision – Precision tests will be conducted simultaneously by monitoring the variance of instrument signal over 30 consecutive measurements at 1 minute intervals in a selected subset of controlled bath conditions in the test matrix described above. Minimally instrument precision will be determined at three formazin and SDVB concentrations tested with the reference water temperature at 15 °C.

Detection Range – The experimental matrix above will enable determination of the linear detection ranges at each test temperature. Limits of detection will be computed as: (Mean + 3 S.D. of blank readings) and upper detection range will be determined as either the dye concentration causing saturation of instrument output or a greater than 50% decline in response factor. Only tests conducted in dark conditions will be used to determine detection limits of the instruments. These range estimates will then be independently tested at a reference temperature of 15°C by monitoring instrument output over a low range of formazin and SDVB concentrations (e.g., 0.2 NTU) and very high levels (e.g., 500 NTU). As described above, instruments will be set to measure turbidity typically in the range of 10 - 0 NTU for all tests.

Impacts of Light – Sensitivity to ambient light will also be assessed by exposing the test instruments to high light (>200 umol/m²/s PAR) using 500W halogen worklights. Instruments will be placed in a bath at 15°C with 0.2, 0.5, 1, 2, 4, 6, 8, 10, 50, 100, 250 and 500 NTU of SDVB (in separate trials) and response measured (as described above) obtained in darkened conditions and under high irradiance (ca 200 μ mol quanta /m² / s, near sensor heads) conditions will be compared.

Reliability – Finally, instrument reliability in the laboratory will be determined by comparing percent of data recovered versus percent of data expected. Comments on problems or instrument failures will also be recorded.

4.2. Field Tests

In situ evaluations of instrument performance in a moored application will be conducted at each of the seven ACT Partner Institution sites (site descriptions below). One turbidity sensor from each manufacturer will be deployed continuous for four weeks (CBL, CILER and SkIO), six weeks (MLML, UHawaii and UMaine), or eight weeks (offshore USF) based on our understanding of biofouling growth that will likely occur during the testing period. Detailed site descriptions are provided below. Test instruments will only be removed from the water after the test period is complete or in the event of a problem such as a weather event that could jeopardize the safety of the instruments. Because each manufacturer will provide only four total instruments, two sets of consecutive field tests will be run. Instrument packages will, however, be returned to manufacturers for a maximum of 3 weeks for reconditioning and calibration in between the two sets of field tests.

Instrument Setup - Prior to deployment, all instruments will be setup and calibrated at the field sites as suggested in individual manufacturer manuals. The turbidity sensors will then be programmed to record data every 15 minutes during the entire field deployment and their internal clocks set to GMT using www.time.gov as the time standard.

As an estimate of instrument drift over time, all turbidity sensors will be placed in a container of a turbidity blank (fixed, bubbled-free DI water) and then a second container of 5 NTU SDVB (as reference standards) both before and after deployment. Randomly select blank and 5 NTU solutions will be selected and sent back to GFS Chemicals after post deployment readings for analysis to assure that the properties of the solution are unchanged (i.e., were not contaminated). The turbidity sensors will be allowed to make three readings with each pre and post standards, with post reference standard measurements taken only after biofouling has been removed from the optical surfaces and instrument cleaned and prepared as prescribed by individual manufacturers. Test staff will be trained by instrument manufactures on proper cleaning techniques.

Biofouling - A photograph of each individual sensor and the entire instrument rack will also be taken just prior to deployment, just after recovery, and after cleaning to provide a qualitative estimate of biofouling during the field tests. As an additional estimate of biofouling, a series of three growth substrates (glass plates, smooth PVC plates and sanded PVC plates) will be deployed at the same depth and as close as possible to the test instruments. Replicate substrates will be used to allow a non-disruptive photograph of biofouling growth after each week of deployment, at each site. One of each substrate type will be removed from the water each week, photographed, and then discarded.

Deployment Rack - All instrument packages will be deployed on a single, box-shaped rack that allows all sensor heads to be at the same depth, and with each manufacturer's instrument side by side. Instrument sensor heads will be deployed at the closest proximity that their designs will allow but far enough apart to prevent cross interference (as separation distance of at least 1 instrument diameter). The rack will be deployed so that all of the turbidity sensors remain at a fixed depth of 1 m below the water surface (using a float system or on a floating dock). The sensor rack design will be standardized as much as possible from site to site. However, physical conditions at particular sites may require specialized modifications.

Sampling Schedule - At the initiation of deployment (during the first three hours), three consecutive water samples (see below) will be taken at one-hour intervals. During the remainder of the moored deployments, each test site will take two water samples every weekday (M-F), each timed to again correspond to the instrument sampling time. The timing of routine water sampling will be left up to the individual site with the goal of capturing natural variations in turbidity levels. A minimum of two intensive sample sets will be collected sometime during the first two weeks of deployment. Timing of the two intensive sampling set will again be left up to the individual sites but are meant to correspond to high turbidity events (e.g., large storms, tidal flushing, or algal blooms) but each will include a minimum of six consecutive samples taken at no greater than one hour apart. This basic sampling schedule will be amended as needed for the USF offshore test site because of limited access. However, the total number of samples collected will be approximately the same as other test sites. Finally, any specific water samples will only

be taken when all test instruments are exposed to the same basic light environment (i.e., all uniformly shaded or uniformly lit).

Water Samples – A standard Van Dorn bottle will be used at each field test site to collect water samples for TSS and POC as ancillary data (see below). However the size of the Van Dorn bottle used by any specific site will vary from 2-l to 8-l depending on the volumes required. The bottles will be lowered into either the center or just upstream of the sensor rack, at the same depth and as close as physically and safely possible to the test instruments. The bottle will be triggered to close at the same time as instrument sampling, to ensure that the same water mass is being compared to in situ turbidity measurements. The entire water sample will be emptied through the bottom of the Van Dorn bottle into a light protected carboy (using a large funnel) and transported to the laboratory at ambient in situ water temperature for further subsampling and processing. Prior to subsampling the whole water sample is homogenized by carefully inverting the carboy 3 times. Each subsample (volume to be determined for each site) will be filtered and processed for TSS (n=3), POC (n=3), as described below. The Van Dorn bottle will be wiped with a clean cloth and rinsed with copious DI water (>5 rinses) between uses.

Filtering - All TSS and POC samples from each ACT Partner test site will be filtered on Whatman GF/F membranes immediately after collection (<30 min). Sufficient volumes of sample water will be filtered to obtain visible color on filter surface and the precise volume filtered for each sample recorded. All filtrations will be done with low vacuum pressure (<5 in Hg).

Cleaning - Filtration apparatus and sample storage vessels will be cleaned between use by wiping with a clean cloth and copious rinses (>5) with DI water. Once per week (typically Fridays after sampling) filtration apparatus will be wiped, rinsed and soaked overnight in a 2% solution of Micro-90 or equivalent detergent, followed by a second round of copious rinsing with DI water.

Reliability - Instrument reliability during moored tests will be determined by comparing percent of data recovered versus percent of data expected. Comments on problems or instrument failures will also be recorded, including internal clock drift.

4.3. Ancillary Environmental Data

A series of ancillary data sets will also be collected during field deployments to both fully characterize the different field conditions during testing and to provide qualitative insight into if any particular environmental parameters are correlated to instrument turbidity values. A calibrated CTD package will be attached to the test rack at each test locale and programmed to provide an independent record of conductivity and temperature at the same depth and the same 15-minute intervals as the test instruments. Each site will also maintain a PAR cosine sensor to continuously monitor surface irradiance history at the deployment site (< 50 meters of the test rack).

A calibrated in situ fluorometer and transmissometer will be connected to a datalogger and placed into the water (at the same depth and as close as possible to the test instruments with cross interference) to collect ancillary data on relative fluorescence and beam-c 660, respectively, every 15 minutes (again corresponding to the timing of test instrument readings). However, because these optical instruments are sensitive to biofouling, they will be cleaned daily just prior to a sampling event. After the daily cleaning, one value for both the fluorometer and transmissometer will be taken in air prior to returning to the water. The cleaned/in air values will either be recorded directly on a datasheet as they are collected or the times record on the datasheet for analysis after the field deployments are competed and all logged values reviewed.

In conjunction with each water sample collection, each deployment site will also record other basic site-specific conditions. At the time of water sample collection, each site will logged on standardized datasheets: date and time (GMT), weather conditions (e.g., haze, % cloud cover, rain, wind speed/direction), air temperature, recent large weather event or other potential natural or anthropogenic disturbances, tidal state and distance from bottom of sensor rack, and any obvious problems or failures with instruments. Datasheets will be transmitted on a weekly basis to the ACT Chief Scientist, for data archiving and ACT personnel performance QA/QC.

4.3.1. Supplementary Data

As an additional characterization of the optical properties of the water at two selected field sites, LISST laser diffraction particle analyzers will also be deployed. During only the first week of field tests, one calibrated LISST will be deployed at the CILER site and one at the UMaine site to record continuously at 15-minute intervals. Like other instruments, they will be placed at the same depth and as close as possible to the test instruments with cross interference. The LISST will be set up, run, and data analyzed by members of the ACT Turbidity Technical Advisory Committee (Emmanuel Boss and Nathan Hawley). After data analysis, it will be decided (in consultation with the participating manufacturers) whether LISST provides any meaningful insight into turbidity sensor performance and if the data should be included in the final Verification Statements.

4.3.2. Total Suspended Solids (TSS)

Each test site will be responsible for collecting and processing TSS samples for the field tests and TSS will be determined for every sampling event. TSS is the retained material on a standard glass filter pad after the filtration and drying of a well-mixed sample of water, with the results expressed in mg/L. The methods to be used are based on APHA Method 208D (1975), USEPA Method 160.2 (1979), and Etcheber, H. 1981, *Journal de recherche oceanographique*, 6:37-42.

Personnel from each test site will be trained on this standardized method and will conduct a minimum of five test TSS analyses with water collected at their moored deployment site prior to field tests to help determine the appropriate sample size.

Pre-collection Preparation -

1. Sequentially number Whatman 47 mm GF/F filters (0.7 μ m pore size) along the outside edge, where the sample will not pass through, using a fine-tipped permanent marker.

2. Pre-rinse both sides of filters prior to use with DI water.

3. Dry filters at 60 - 65°C overnight (> 24 hours).

4. Weigh filters (in grams) to 4 decimal places to obtain pre-weight. Pre-weighed filters are stored in sealed containers until ready for use.

Sample Collection and Handling -

1. Pre-rinse the filter with 0.5 M ammonium formate to wet the entire filter pad and then put

filter cone in place. Pre-rinsing will help prevent the formation of a salt "halo" that tends to creep along toward the outer edge of the filter, as a dry filter is wetted at its center.

2. Filter a known volume of water through the filter pad and rinse filter with 0.5 M ammonium formate to remove salts (both the ammonium formate rinses can be excluded for freshwater samples).

3. Fold pad in half, sample inside, and place in a sealed container or aluminum foil pouch.

4. Freeze filter pads for storage.

Analytical Procedure –

1. Dry filters at 60 - 65°C overnight (> 24 hours). Allow samples to cool to room temperature in dessicator.

2. Weigh filters and record weights (in grams).

3. Subsample a portion of the filters and replace them in the drying oven for a minimum of 1 hour.

4. Re-weigh subsampled filters. If there is >0.5 mg weight loss between the first and second weight of the subsampled filter pads, then all filter pads should be re-dried and re-weighed. 5. Repeat steps 3 and 4 as necessary.

Calculation of TSS -

TSS concentration is calculated using the following equation:

$$mgTSS/L = \frac{(W_{post} - W_{pre})x1000}{V}$$
(4)

where:

 W_{post} = dry weight of filter pad after filtering (g), W_{pre} = dry weight of filter pad before filtering (g), and V = volume of water filtered (L).

4.3.3. Particulate Organic Carbon

POC samples will be sent off for analysis to an outside laboratory facility (see below) and will be collected for all intensive sampling events but only once per day during normal sampling. POC samples will be collected on pre-combusted 25 mm Whatman GF/F filters (450 °C, 5 h) and the precise volume filtered for each sample recorded. Prior to use, all pre-combusted filters should be stored in a dry place (e.g. in a desiccator). As soon as fluid completely runs through the filter, rinse the inside of the filtration tower with GF/F filtered seawater. The filter sample should be removed immediately (with the vacuum still on), placed in a small petri dish (one filter per dish) and left in a drying oven (65 °C) overnight (> 24 hours). Prior to shipping, all the dried POC samples will be folded in half and packed in pre-combusted aluminum foil. If the POC samples are packed in advance, they can be stored in a desiccator until the day of the shipment.

Each week a number of dry and wet blanks (3 each) should be collected and stored as separate samples. A dry blank is a pre-combusted filter that has not been used in a filtration. A wet blank takes into account the amount of dissolve organic carbon (DOC) that is in the residual water left in the filter matrix after filtration of a sample (Gardner et al. 2003, Deep-Sea Research II 50:655-674). A wet blank is made by "dunking" a pre-combusted filter in seawater filtrate

(either from that days TSS samples or the POC collection) and excess water is removed by placing the filter on a filtration-manifold (with the vacuum on). Further processing and storage is the same as for an ordinary POC sample. One set of dry and one set of wet blanks will also be taken just prior to instrument deployment and set off for analysis to help establish a baseline that can be used to identify contamination, if it occurs during the field tests.

The dried samples will be shipped by express courier (and with appropriate COC forms) to the University of California at Santa Barbara, Marine Science Institute's Analytical Laboratory for analysis. Determination of elemental composition of organic material in solid samples (weight percent of carbon, hydrogen, and nitrogen) will be made using an Automated Organic Elemental Analyzer (Dumas combustion method) with the following sensitivity, range and precision:

С	2 - 4000 ug, 0.01 - 100%	2 ug or 0.3%
Η	2 - 1000 ug, 0.02 - 20%	2 ug or 0.3%
Ν	1 - 2000 ug, 0.01 - 60%	1 ug or 0.3%

The UCSB Marine Science Institute Analytical Laboratory was created in 1977 to provide analytical chemistry support, services, and consultation to the marine science community. A variety of researchers rely on the data produced by the Analytical Laboratory to accurately represent the nature of the material submitted. The laboratory analyzes thousands of samples per year, submitted by individual investigators and large research programs both nationally and internationally. In addition to the widely accepted methodologies and protocols used, the Analytical Laboratory has a rigorous Quality Assurance Plan that provides a means of assuring the output of reliable and valid analytical data (available from ACT and UCSB Marine Science Institute upon request).

5.0. Verification Schedule (planned dates but may vary).

- The Final Verification Protocols and ACT Verification Contract will be sent to Manufacturers on March 10, 2006
- Signed contracts are due back to ACT Headquarters by April 7, 2006
- All instruments to be tested will be delivered to MLML by April 21, 2006
- ACT Chief Scientist, Technical Coordinators, Quality Manager, and Manufacturer Representatives will meet at MLML for instrument use/operation/deployment, sample collection, storage and shipping training on April 26 28, 2006
- Selected ACT staff will conduct the laboratory verification tests on May 1-5, 2006
- All instruments will be delivered to the first four ACT test sites by May 5, 2006
- The first four moored deployment tests will begin sometime between May 8 and 18 (UHawaii 6 weeks, MLML 6 weeks, UMaine 6 weeks, and USF offshore 8 weeks)
- Three sets of instruments (from UHawaii, MLML and UMaine) will be sent back to individual Manufacturers for reconditioning and calibration by June 23, 2006
- Instruments will be sent back from Manufacturers to the second set of three ACT test sites and received by June 23, 2006
- The second set of three moored deployment tests (CBL 4 weeks, CILER 4 weeks and SkIO 4 weeks) will begin sometime between July 12 and 21, 2006
- All instruments will be sent back to individual Manufacturers by August 25, 2006
- ACT Chief Scientist, Technical Coordinators, Technical Advisory Committee, and Quality Manager, will meet for 2 days to analyze results and evaluate the Verification

processes in October 2006

- ACT Verification Statements for each individual instrument will be drafted and sent out for review by, Technical Advisory Committee, Technical Coordinators, Quality Manager, Partners, and Stakeholders in early December 2006
- Final Verification Statements will be sent to Manufacturers the first week of January, 2007
- One page comment letters from Manufacturers are due by the end of January, 2007
- Final Verification Statements will be released to the public by the end of January, 2007

6. Data Recording, Processing and Storage

This section describes methods employed during data recording, processing, and storage to minimize errors and assure high quality analyses in the Verification Statements.

6.1. Documentation and Records

A variety of data will be acquired and recorded electronically and manually by ACT staff in the laboratory verification test and field study. Operational information and results from ancillary data collection methods will generally be documented in field/laboratory record books and on the data sheet/chain-of-custody forms (see below). An electronic copy of these raw data will be transferred to the ACT Chief Scientist weekly, who will store it permanently along with the rest of the study data.

The results from the test turbidity sensors will also be recorded electronically. Test instrument data will be logged by individual sensor packages and will only be downloaded and analyzed upon completion of the four-week field deployments. Once collected, one copy of these data will reside at the corresponding ACT test facility and a second copy at ACT Headquarters until the entire verification is finished. The table below summarizes the types of data to be recorded and the process for recording data.

Data to be	Responsible	Where Recorded	How Often	Purpose of Data
Recorded	Party		Recorded	
Dates, times of sampling events	Each ACT Partner	Field/laboratory record books/data sheets	Each laboratory and field sample collection	Used to organize/check test results; manually incorporate data into spreadsheets - stored in study binder
Environmental parameters (site conditions), including water temperature, salinity, PAR, weather, and sea state	Each ACT Partner	Field/laboratory record books/data sheets	Each field sample collection	Used to define site characteristics; manually incorporate data into spreadsheets - stored in study binder
Water sample test parameters (ancillary data), including TSS and POC	Each ACT Partner; Certified lab (TBD) for POC	Laboratory record book/data sheets	At the conclusion of each analytical sample batch.	Used to define site characteristics; manually or electronically incorporate data into spreadsheets - stored in study binder

Test parameters	Each ACT	Internal datalogger	Each field sample	Used to define site
(ancillary data),	Partner;		collection	characteristics; manually
including	(CILER &			or electronically
fluorescence, %	UMaine for			incorporate data into
transmission,	LISST)			spreadsheets - stored in
				study binder
Ancillary	Each ACT	Laboratory record	Start/end of test	Document correct
parameter	Partner;	book/data sheets		performance of ancillary
instrument	(CILER &			parameter instrument
calibration data	UMaine for			
	LISST)			
Test instrument	Each ACT	Laboratory record	Start/end of test	Document correct
calibration data	Partner	book/data sheets		performance of test
				instrument
Test instrument	Each ACT	- Data sheets	After completion of	Used as part of test
data	Partner	- Instrument data	the laboratory	results; incorporate data
- digital display		acquisition system	verification and field	into electronic
- electronic		(data logger)	deployments	spreadsheets - stored in
output				study binder
Water sample	ACT Partner	Laboratory record	At the conclusion of	Used to check
analytical	Certified lab	book/data sheets	each analytical	test results; manually
results			sample batch.	incorporate data into
				spreadsheets - stored in
				study binder

6.2. Data Review

All data are to be recorded directly in the field/laboratory record book as soon as they are available. Records are to be written in water-proof ink, written legibly, and have any corrections initialed by the person performing the correction. Any corrections will be crossed out with a line (not blackened or white-out), and the correction made, with initials and date of correction. These data will include electronic data, entries in field/laboratory record books, operating data from the ACT Partner test facility, and equipment calibration records. Records will be spot-checked within two weeks of the measurement to ensure that the data are recorded correctly. The checker shall not be the individual who originally entered the data. Data entries shall be checked in general for obvious errors and a minimum of 10 percent of all records shall be checked in detail. Errors detected in this manner shall be corrected immediately. The person performing the review will add his/her initials and the date to a hard copy of the record being reviewed. The ACT Technical Coordinator (TC) will place this hard copy in the files for this verification test. In addition, data generated by each ACT Partner test site will be provided to the ACT Chief Scientist and reviewed before they are used to calculate, evaluate, or report verification results.

7. Quality Assurance/Quality Control

Technology performance verifications are implemented according to the test/QA plans and technical documents (e, g. Standard Operating Procedures) prepared during planning of the verification test. Prescribed procedures and a sequence for the work are defined during the planning stages, and work performed shall follow those procedures and sequence. Technical procedures shall include methods to assure proper handling and care of test instruments. All implementation activities are documented and are traceable to the test/QA plan and SOPs and to test personnel.

7.1. Analytical Laboratory Quality Control for TSS and POC

The analyses for TSS and POC shall have the following Quality Controls:

a. <u>Blanks</u>

Weekly analysis of blanks. These blanks will be collected weekly during sampling and should include Field Blanks (see Section 7.4.2).

b. <u>Control Charts.</u> Two types of control charts are used in laboratories: a mean chart for **blanks** and a range chart for **replicate** analyses.

7.2. Quality Control for Instrument Calibration

The test instrumentation to be used in the laboratory verification test and field studies will be calibrated by the ACT TCs at MLML according to the SOPs for the instrumentation prior to the laboratory tests. A calibration log will be created for each instrument. The logs shall include at least the following information: name of instrument, serial number and/or identification number of instrument, date of calibration, and calibration results. These logs shall be provided to the ACT Chief Scientist and maintained in a master calibration file as part of the QA/QC records.

7.3 Laboratory Test Quality Control

All analytical measurements are performed using materials and/or processes that are traceable to a Standard Reference Material. Standard Operating Procedures are utilized to trace all quantitative and qualitative determinations to certified reference materials. All metrology equipment (analytical balances, thermometers, etc.) is calibrated using materials traceable to the National Institute of Standards and Technology (NIST) and maintained on a schedule to ensure accuracy.

All volumetric glassware must be calibrated as conforming to Class A. A valid certificate of calibration or compliance must be available for each item. If the item has been calibrated in-house, the laboratory shall have a documented record of the calibration data showing traceability to national standards. Since the capacity of volumetric glassware may change with use, the calibration should be verified at regular intervals. Volumetric capacity is normally determined gravimetrically, using water conforming to the ACT glassware calibration Standard Operating Procedure (SOP) [see attached file]. Before starting, scrupulous care must be taken to ensure that the glassware is clean and, in particular, grease free. The amount of water that the vessel contains, or delivers at a measured temperature, is accurately weighed, and the volume calculated in milliliters at standard temperature and pressure.

7.4. Field Quality Control – Mooring Deployments

Field quality control represents the total integrated program for assuring the reliability of measurement data. It consists of the daily field logs, quality control samples, and sample custody procedures.

7.4.1. Field Logs

Standard, uniform field logs should be maintained for all fieldwork. These logs should report name of staff conducting fieldwork, date (month, day, and year), operating status of all equipment, and manual readings of environmental conditions.

7.4.2. Field Quality Control Samples

Field quality control samples provide information on the potential for bias due to contamination of analytical results by sample collection, processing, shipping, and analysis. To ensure that the field sample collection and analysis procedures are properly controlled, field blanks and replicate samples will be taken once a week during the field period. These will be analyzed in the same manner as the collected samples for TSS and POC. Based on analysis of previous trip blanks, sample device / equipment blanks, filtration blanks, and field blanks for TSS samples collected and analyzed by the ACT field sites, only the all-inclusive field blanks will be employed for this evaluation. Field blanks are generated under actual field conditions. These blanks account for all of the above sources of contamination that might be introduced to a sample plus other incidental or accidental sample contamination during the entire process of sampling, transport, sample preparation, and processing. While field blanks mimic sample collection and processing, they do not come in contact with ambient water. The protocol is:

- a) Fill a carboy with 2-1 of deionized water in the laboratory and transport to the field site.
- b) Pour deionized water into the Van Dorn bottle sampler, then empty the water from the sampler into a second carboy.
- c) Transport blank to laboratory, filter the sample as per the protocol, and ship to the lab with the remaining samples.

7.4.3 Sample Custody

All TSS and POC samples will be accompanied by the sample collection sheet and for the POC only, a Chain-of-Custody (COC) form.

The COC specifies time, date, sample location, unique sample number, requested analyses, sampler name, required turnaround time, time and date of transaction between field and laboratory staff, and name of receiving party at the laboratory. Proper labeling of sample bottles is critical. The COC is a mechanism by which a sample can be tracked through the various phases of the process: collection, shipping, receiving, logging, sample prep/extraction, analysis and final data QA/QC review.

When transferring the possession of the samples, the transferee must sign and record the date and time on the chain-of-custody record. Custody transfers, if made to a sample custodian in the field, should account for each individual sample, although samples may be transferred as a group. Every person who takes custody must fill in the appropriate section of the chain-of-custody record. The TC is responsible for properly packaging and dispatching samples to the laboratory for analysis. This responsibility includes filling out, dating, and signing the appropriate portion of the chain-of-custody record. The original and one copy of the chain-of-custody record form should be placed in a plastic bag inside the secured shipping container with the samples. One copy of the chain-of-custody record form should be placed in a plastic bag inside the secured shipping container with the samples. The transportation case should then be sealed and labeled. All records should be filled out legibly in waterproof pen. When shipping samples via common carrier, the "Relinquished By" line should be filled in by the TC; however, the TC should leave the

"Received By" box blank. The laboratory sample custodian is responsible for receiving custody of the samples and will fill in the "Received By" section of the Chain-of-Custody Record.

7.4.4 Sample Handling

All collected TSS and POC samples at each test site will be handled in the same manner. Each sample should be dated and coded according to site and sample sequence. The actual sample container should be labeled with a number for identification. Samples stored for any period of time shall be routinely inspected by the TC to assure proper preservation and label integrity. The storage containers and storage devices (i.e. freezers and refrigerators) must be inspected routinely for proper operation and integrity. Results of all inspections shall be included in the sample records. All logs shall be duplicated weekly. The original shall be retained at the ACT Partner site and a copy shall be sent to the ACT Chief Scientist.

7.5. Audits

Independent of each Partner test facility QA activities, the ACT Chief Scientist will be responsible for ensuring that the following audits are conducted as part of this verification test at a minimum of three ACT Partner test sites. Audits shall be performed by Quality Assurance Specialists, who shall be independent of direct responsibility for performance of the verification test.

7.5.1. Technical Systems Audits

ACT's Quality Assurance Specialists will perform a TSA at least once during the laboratory verification test and field studies. The purpose of this audit is to ensure that the verification test and field studies are being performed in accordance with the ACT Protocols, published reference methods, and any SOPs used by the Partner test facility. In this audit, the ACT Quality Assurance Specialists may review the reference methods used, compare actual test procedures to those specified or referenced in the Protocols, and review data acquisition and handling procedures. A TSA report will be prepared, including a statement of findings and the actions taken to address any adverse findings.

7.5.2. Data Quality Audits

ACT's Assurance Specialists will audit approximately 10% of the verification data acquired in the verification test to determine if data have been collected in accordance to the Protocols with respect to compliance, correctness, consistency, and completeness. The ACT Quality Assurance Specialists will trace the data from initial acquisition to final reporting.

7.5.3. Assessment Reports

Each assessment and audit will be documented, and assessment reports will include the following:

- a. Identification of any adverse findings or potential problems,
- b. Response to adverse findings or potential problems,
- c. Possible recommendations for resolving problems,
- d. Citation of any noteworthy practices that may be of use to others, and
- e. Confirmation that solutions have been implemented and are effective.

7.6. Corrective Action

The ACT Chief Scientist, during the course of any assessment or audit, will identify to the ACT Technical Coordinators performing experimental activities any immediate corrective action that should be taken. If serious quality problems exist, the ACT Chief Scientist is authorized to stop work. Once the assessment report has been prepared, the ACT Chief Scientist will ensure that a response is provided for each adverse finding or potential problem and will implement any necessary follow-up corrective action. The ACT Quality Assurance Specialists will ensure that follow-up corrective action has been taken.

7.7. QA/QC Document Control

It is the responsibility of the ACT Chief Scientist to maintain QA/QC records, which shall include the following:

- 1) records of the disposition of samples and data.
- 2) records of calibration of instruments.
- 3) records of QA/QC activities, including audits and corrective actions.

8. Roles and Responsibilities

The verification test is coordinated and supervised by the ACT Chief Scientist and ACT Partner institution personnel. Staffs from the Partner institutions participate in this test by installing, maintaining, and operating the respective technologies throughout the test; operating the reference equipment, collecting the water samples, downloading the data from the instrument package, and informing the ACT Chief Scientist staff of any problems encountered. Manufacturer representatives shall train ACT Partner staffs in the use of their respective technologies and, at their discretion, observe the calibration, installation, maintenance, and operation of their respective technologies throughout the test. QA oversight is provided by the ACT Quality Managers. In addition to aiding the development of these protocols, the ACT Turbidity Technical Advisory Committee will be consulted during the evaluation in the event problems occur, will assist in the analyses of results, and will review the final Verification Statement prior to release. Specific responsibilities are detailed below.

The ACT Chief Scientist has the overall responsibility for ensuring that the technical goals and schedule established for the verification test are met. The ACT Chief Scientist shall:

- Prepare the draft Test Protocols/QA Plan and Verification Statements.
- Revise the draft Test Protocols/QA Plan and Verification Statements in response to reviewers' comments.
- Coordinate distribution of the final Test Protocols/QA Plan and Verification Statements.
- Coordinate testing, measurement parameters, and schedules at each ACT Partner institution testing site.
- Ensure that all quality procedures specified in the test/QA plan are followed.
- Respond to any issues raised in assessment reports and audits, including instituting corrective action as necessary.
- Serve as the primary point of contact for manufacturers and ACT Partner Technical Coordinators.
- Ensure that confidentiality of proprietary manufacturer technology and information is maintained.

ACT Quality Managers for the verification test shall:

- Review the draft Test Protocols/QA Plan and Verification Statements.
- Conduct a technical systems audit (TSA) once during the verification test.
- Audit at least 10% of the verification data.
- Prepare and distribute an assessment report for each audit.
- Verify implementation of any necessary corrective action.
- Notify the ACT Chief Scientist if a stop work order should be issued if audits indicate that data quality is being compromised or if proper safety practices are not followed.
- Provide a summary of the audit activities and results for the verification reports.
- Review the draft verification reports and statements.
- Have overall responsibility for ensuring that the test/QA plan and ACT QMP are followed.
- Ensure that confidentiality of proprietary manufacturer technology and information is maintained.

ACT Technical Coordinators at each ACT Partner institution shall:

- Assist in developing the Test Protocols/QA Plan.
- Allow facility access to the manufacturers and ACT Headquarters representatives during the field test periods.
- Select a secure location for the tests.
- Install, maintain, and operate the turbidity test systems at the test location at their respective institution.
- Perform sample collections and analyses as detailed in the test procedures section of the test/QA plan.
- One member of TC team will conduct 10% data audit as described in QA procedures. This will be done for all data logs and electronically entered data.
- Provide all test data to the ACT Chief Scientist electronically, in mutually agreed upon format.
- Remove sensor systems and other related equipment from the test facility upon completing the verification test.
- Provide the ACT Chief Scientist and Quality Managers access to and /or copies of appropriate QA documentation of test equipment and procedures (e.g., SOPs, calibration data).
- Provide information regarding education and experience of each staff member involved in the verification.
- Assist in ACT's reporting of their respective test facility's QA/quality control results.
- Review portions of the draft Verification Statements to assure accurate descriptions of their respective test facility operations and to provide technical insight on verification results.

ACT West Coast Partner Institution, Moss Landing Marine Laboratories, shall:

- Perform reference measurements.
- Perform sample collections and analyses as detailed in the test procedures section of the Test Protocols.
- Provide the ACT Chief Scientist and Quality Managers access to and /or copies of appropriate QA documentation of test equipment and procedures (e.g., SOPs, calibration data).
- Provide information regarding education and experience of each staff member involved in the verification.

- Assist in ACT's reporting of their respective test facility's QA/quality control results.
- Review portions of the draft Verification Statements to assure accurate descriptions of their respective test facility operations and to provide technical insight on verification results.

Manufacturers shall:

- Review the draft test/QA plan and provide comments and recommendations.
- Approve the revised test/QA plan.
- Work with ACT to commit to a specific schedule for the verification test.
- Provide four commercial-ready sensor systems for testing, in individual shipping containers/boxes, to the laboratory test facility by agreed upon date.
- Provide an on-site operator(s) to train ACT staff in the installation, calibration, operation, maintenance and cleaning of the sensor systems.
- Review and comment upon their respective draft Verification Statements.

Note: ACT reserves the right to dismiss any manufacturer from the Performance Verification if it does not comply with agreed upon schedules or requirements.

Turbidity Technical Advisory Committee shall:

- Assist in developing the Test Protocols/QA Plan.
- Approve the final Test Protocols/QA Plan.
- Provide specific advice during testing.
- Review and comment upon draft Verification Statements.
- Approve final Verification Statements.

9. Turbidity Technical Advisory Committee

- Emmanuel Boss, University of Maine
- Earle Buckley, North Carolina State University and ACT Advisor/QA Manager
- June Harrigan, ACT Stakeholder
- Nathan Hawley, NOAA Great lakes Environmental Research Laboratory
- Mark Luther, ACT Partner and University of South Florida
- Scott McLean, ACT Stakeholder and Satlantic
- W. Scott Pegau, Kachemak Bay Research Reserve
- Paul Pennington, NOAA Center for Coastal Environmental Health and Biomolecular Research and ACT QA Specialist

10. Field Test Site Descriptions

Chesapeake Biological Laboratory Field Test Site -

The ACT Partner at Chesapeake Biological Laboratory (CBL), University of Maryland Center for Environmental Science, has established a Technology Verification Field Test Site on a fixed pier (Lat: 38°19.039 N, Lon: 76°27.065 W, with an average depth of 7 ft) at the mouth of the Patuxent River, a tributary of the Chesapeake Bay. The Chesapeake is a nutrient rich estuary with a watershed that encompasses portions of six states and the District of Columbia. Water temperatures at the testing location range from 0° to 35°C and salinities range from 5ppt to 20ppt depending on season, rainfall, wind, and other external factors.

Cooperative Institute of Limnology and Ecosystem Research Field Test Site –

The ACT Partner at the Cooperative Institute for Limnology and Ecosystems Research, University of Michigan, has established a Technology Verification Field Test Site on a fixed pier at the Water Studies Institute of Northwestern Michigan College in Traverse City, Michigan (44-46-00 N x 85-37-00W). The site provides direct access to Traverse Bay, Lake Michigan with water depth at the end of the pier averaging 3m. Lake temperatures range from 2 to 24°C on an annual basis. The Institute operates a continuous real-time meteorological station and fully equipped laboratories to process field samples.

Gulf of Maine Ocean Observing System Test Site -

The ACT Partner at the Gulf of Maine Ocean Observing System (GoMOOS) has established a Technology Verification Field Test Site at the University of Maine's, Darling Marine Center in Walpole, Maine. The Center occupies 170 acres of largely wooded property bordering 2 km of pristine water frontage on the Damariscotta River Estuary, and offers a secure and easy access to the estuary and maintains a pier and boating facility on site. Water sample analysis can be conducted during sensor evaluations in the laboratory facility near the pier. The Damariscotta River estuary is a tide dominated embayment approximately 5 km from the open waters of the Gulf of Maine. The site experiences a predominantly semi-diurnal tide with an approximate amplitude of 3m. Local marine environments include rocky shores, sandy beaches, mud flats, sea grass beds, and expansive sponge communities. The complexity of the Maine coastline allows for a wide range of exposure to waves and ice, further adding to the diversity of habitats. Sea temperatures range from 2 to 15 C° in the open ocean and from -2 to 20 C° in the upper reaches of the estuary. Salinity at the Center's dock ranges from 28 to 32 ppt.

Moss Landing Marine Laboratories Field Test Site -

The ACT Partner at Moss Landing Marine Laboratories (MLML) has established its Technology Verification Field Test Site at the MLML Small Boat Facilities (36.8041N, 121.7862W). This secure deployment site is located in Moss Landing Harbor on the junction of northern tributary of the Salinas River and Elkhorn Slough National Estuarine Reserve on the central coast of California. Instrumentation is deployed off a secure floating dock in waters with a tidal range of 2 meters and a maximum depth below the floating dock of 4 meters. It is an estuarine environment with a mean temperature of 12.858 °C (range: 11.287 to 15.767°C) and a mean conductivity/salinity of 3.615 S m⁻¹ / 30.577 PSU (range: 1.358 to 4.036 S m⁻¹ and 10.851 to 32.942 PSU) at 1 meter depth.

Skidaway Institute of Oceanography Field Test Site -

The ACT Partner at the Skidaway Institute of Oceanography (SkIO) has established a Technology Verification Field Test Site on a floating dock adjacent to fixed wooden dock located on the western shore of Skidaway Island (Lat: 31° 59.442' N; Lon: 81° 01.298 W). Skidaway Island is sheltered from the Atlantic Ocean by a chain of barrier islands. The site experiences a semi-diurnal tide with a 2 m amplitude. The SkIO site is located within a typical subtropical estuary dominated by *Spartina alterniflora*. The fixed dock is a wooden structure that juts westerly into the north/south running Skidaway River. The minimum depth at test site is 10.5 ft. or 3.2m at MLW. Water temperature ranges from 10 - 32°C, and salinity from 10 - 35 ppt.

University of Hawaii Field Test Site -

The University of Hawaii field site will be on the Kaneohe Bay Barrier Reef flat ($157^{\circ}48'W$, $21^{\circ}28.5'$) in waters ~2 m deep. Kaneohe Bay sits on the northeast, or windward, side of Oahu. The barrier reef acts as a physical divider separating coastal waters from the Kaneohe Bay lagoon and coastal ocean, as well as impeding the passage of surface wave energy into the bay interior. Significant wave heights at the study site are typically < 1 m with mean cross-reef currents only on the order of a few cm s-1. Both wave heights and cross-reef currents appear to be heavily modulated by the tides. Water temperatures at this site vary between 21 and 29°C with highest values in summer. Tidal variations are typically less than 0.5 m and salinities are between 34.5 and 35.5 psu.

University of South Florida Field Test Site –

The ACT Partner at the University of South Florida (USF) has established its Technology Verification Field Site this year at the start of the Tampa Bay main shipping channel, approximately five miles offshore at the Palatine Shoal. Tampa Bay is the largest Florida estuary and the second largest estuary in the eastern US. The deployment site (27° 35.722 N and 82° 51.516 W) is a piling structure constructed of H-beam extending five meters above the surface. These waters have a May-June temperature range from 23.7.5°C to 31.0°C with a mean of 28.6°C. The salinity during this time varies from 20 psu to about 32 psu and is strongly dependent on rainfall amount. The site has a mean depth of 6.0 m and a mixed tidal range of about 1m. The seabed consists of fine to medium siliclastic quartz and shell hash.