

**Region 4**  
**U.S. Environmental Protection Agency**  
**Science and Ecosystem Support Division**  
**Athens, Georgia**

**OPERATING PROCEDURE**

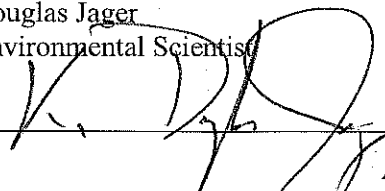
**Title: Field Measurement Uncertainty**

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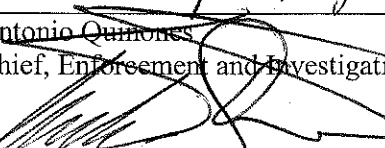
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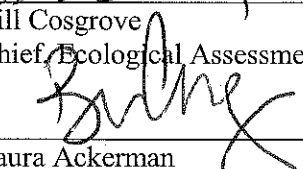
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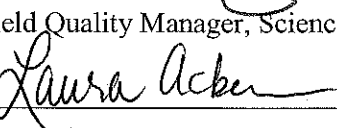
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## Revision History

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This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

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SESDPROC-014-R0, Estimating Field Measurement Uncertainty, Original Issue	February 11, 2008

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# 1 General Information

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## 1.1 Purpose

The purpose of this procedure is to provide direction regarding reporting uncertainty of field measurements.

## 1.2 Scope/Application

Environmental field measurements pose unique challenges for estimating measurement uncertainty. This procedure addresses these uncertainty issues related to field measurements and field instrumentation and provides a methodology for estimating uncertainty for environmental measurements conducted in the field by SESD investigators. See Section 3 of this procedure for more detail on the issues associated with estimating uncertainty for environmental field measurements and the methodologies employed by SESD for estimating field measurement uncertainty. Uncertainty statements can only be made for data collected following the initial effective date of this operating procedure.

## 1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities. The official copy of this procedure resides on the H: drive of the SESD local area network. The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the H: drive and for maintaining records of review conducted prior to its issuance.

## 1.4 Definitions

### *1.4.1 Accuracy of measurement (Accuracy)*

Closeness of the agreement between the result of a measurement and the true value of the analyte being measured; where the true value is often theoretical or unknowable. Accuracy is a function of both precision and bias.

### *1.4.2 Precision*

With respect to a single device, put into operation repeatedly without adjustments, *precision* is the ability to produce the same value or result, given the same input conditions and operating in the same environment. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

### *1.4.3 Bias*

Consistent deviation of measured values from the true value, caused by a systematic error, or by two or more such errors operating cumulatively.

#### **1.4.4 Qualitative Measurement**

Detection techniques used to identify the compounds or physical properties present in a sample.

#### **1.4.5 Quantitative Measurement**

Measurement techniques used to determine the amount of each compound or the amount of the physical property in the sample.

#### **1.4.6 Detection Limits**

The lowest concentration or amount of target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive.

#### **1.4.7 Standard Deviation**

A measure of how much the data in a sample population are scattered around its mean value. It is usually denoted with the letter  $\sigma$  (lower case sigma). A standard deviation is defined as the square root of the variance.

#### **1.4.8 Significant**

Having an outcome unlikely to be caused by chance, and therefore indicating a systematic relationship, between measurements and conditions.

### **1.5 References**

International Organization for Standardization (ISO). (1993, corrected and reprinted 1995). *Guide to the Expression of Uncertainty in Measurement (GUM)*.

International Organization for Standardization (ISO). (1993, second edition). *International Vocabulary of Basic and General Terms in Metrology*.

National Institute for Standards and Technology (NIST). (1994). *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results (TN-1297)*.

National Institute for Standards and Technology (NIST). (October 2000). *Essentials of Expressing Measurement Uncertainty*. Retrieved February 28, 2007 from <http://www.physics.nist.gov/cuu/Uncertainty/index.html>

William J. Tilstone, "Uncertainty of Measurement," *The FQS Update*, vol. 2, issue 2 (June 2007), pages 1-3.

Analytical Support Branch Laboratory Operations and Quality Assurance Manual, January 2007.

National Enforcement Investigations Center Operating Procedure for Estimation of measurement Uncertainty (NEICPROC/07-004), November 9, 2007.

SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011), most recent version.

SESD Table of Field Measurement Uncertainties (SESDFORM-034), most recent version.

Field Data for Measurement Uncertainty Form (SESDFORM-035), most recent version.

## **2 Methodology**

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### **2.1 General**

ISO/IEC 17025:2005 requires reporting of measurement uncertainty for quantitative analytical results under three conditions:

- 1) when uncertainty is relevant to the validity or application of a measurement method,
- 2) when a customer's instruction so requires,
- 3) when the uncertainty affects compliance to a regulatory limit.

Uncertainty statements are typically requested by the customer in the planning phase for a project. If a customer requests an uncertainty statement following completion of the project, SESD management will determine whether it is feasible to report the uncertainty. For field data appearing in reports, uncertainty should be calculated and reported for data that directly affects compliance to a regulatory limit. For data collected as indicator parameters (e.g. measurements to verify stabilization of a monitoring well or to develop strata for sample collection), uncertainty statements will not be reported, unless the customer requests it or Project Leader deems it necessary.

The overall goal of documenting measurement uncertainty is to provide information that can be used for decision making. To achieve this goal, it is important to know if measurement uncertainty related to detection limits, field analytical bias, lack of precision, and susceptibility to interferences is significant.

### **2.2 Uncertainty Contributors**

Both qualitative and quantitative factors contribute to field measurement uncertainty. For field measurements, there are several factors that can affect qualitative and quantitative measurements. Qualitative uncertainty factors include but are not limited to interferences, environmental conditions, sample handling, and instrument maintenance and operation. The SESD field branches reduce the impact of qualitative uncertainty factors through standard operating procedures that address the previously mentioned factors when possible. To date, a uniform approach to estimating the uncertainty associated with qualitative uncertainty contributors has not been developed by the scientific community. Qualitative uncertainty factors that are not addressed through standard operating procedures (e.g., weather extremes and any potential impact they may have had on the data) are discussed in the final report.

The amount of uncertainty contributed by quantitative uncertainty factors can typically be determined. However, it is important to note that qualitative uncertainty factors can also impact quantitative uncertainty factors. Quantitative uncertainty factors include but are not limited to calibration, sample matrix, environmental conditions, sample handling, and equipment maintenance and operation. Factors that are known to affect quantitative uncertainty are instrument precision and accuracy, and the accuracy of calibration

standards. Field investigators should be aware of inherent instrument limitations, such as potential interferences, detection limits, and accuracy, and should ensure that the instrument is capable of collecting data that satisfies the data quality objectives of the study, particularly for studies that involve regulatory limits.

### **2.3 Reporting Uncertainty Statements**

Uncertainty statements will be reported in accordance with the guidance presented in the International Organization for Standardization (ISO) *Guide to the Expression of Uncertainty in Measurement* (GUM) or other adapted references based on the GUM. The National Institute for Standards and Technology (NIST) *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results* (NIST Technical Note 1297) is a helpful aid for understanding the GUM. For a complete listing of references for this procedure see Section 1.5 of this procedure.

In addition to the procedures defined here, if an uncertainty statement is reported, SESD investigators will consult the GUM, NIST or other adapted references in order to specifically evaluate how to use quoted uncertainties to derive and report standard uncertainty for a particular field measurement. See section 2.1 of this procedure for a list of when uncertainty statements are reported.

### **2.4 Estimating Quantitative Uncertainty**

When estimating field measurement uncertainty, field investigators will attempt to identify all the major uncertainty contributors and make a reasonable estimation. When an uncertainty statement is required as discussed previously in Section 2.1, the uncertainty statement will be prepared taking into consideration the instrument uncertainty, calibration standard uncertainty, the data quality objectives and any other applicable factors.

### **2.5 Uncertainty Types**

The GUM and NIST documents discuss the evaluation of uncertainty according to a “Type A” or “Type B” method of evaluation. The Type A method evaluates standard uncertainty by the statistical analysis of a series of repeated observations. The Type B method evaluates uncertainty based on scientific judgment using all of the relevant information available, which may include:

- previous measurement data
- experience with or general knowledge of the behavior and properties of relevant materials and instruments
- manufacturer’s specifications
- data provided in calibration certificates and other certificates.

Broadly speaking, Type B uncertainty is either obtained from an outside source, or obtained from an assumed distribution based on judgment using all information available.

## 3 Implementation

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### 3.1 General

Environmental field measurements pose unique challenges for estimating measurement uncertainty. Unlike like conducting a Type A uncertainty analysis in a laboratory setting, many times during field measurement activities it is impossible to perform replicate analyses. Supplying a known spike and attempting to determine percent recoveries for many field measurements is also often not possible. Qualitative uncertainty factors as cited in Section 2.2 Uncertainty Contributors, such as sample handling and constantly changing environmental conditions (temperature as one example) complicate and at times directly impede the ability to conduct a Type A uncertainty estimation of the environment measurements performed in the field. Because of these qualitative uncertainty contributors making a Type A uncertainty estimation of the instruments response to the environmental sample problematic, SESD will estimate its field measurement uncertainty using a hybrid approach of combining a Type B assessment along with a Type A assessment of the instruments response to known calibration standards. Typically, a Type A uncertainty estimation would evaluate the instruments variability to the environmental sample and not the instruments variability to known calibration standards. This is because an instruments performance against a known calibration standard will often be more precise than against an environmental sample. However, by combining a Type A uncertainty estimation based on a field instrument performance against known calibration standards along with a Type B estimation of the environmental sample, a total field measurement uncertainty will be estimated. For more detail on the SESD hybrid field measurement uncertainty estimation see Section 3.3 of this procedure. For more detail on Type A uncertainty estimations computed solely on the measurements of the environmental sample see Section 3.4 of this procedure.

SESD is in the process of designing the necessary forms and data information management systems to gather the instrument performance data that will enable instrument uncertainty to be computed using a hybrid of a statistical Type A approach and the professional judgment (Type B) of SESD staff. Until adequate data is collected, all uncertainty will be assumed to be produced from random effects and will be treated as residing in a normal distribution. The Type A portion of this hybrid uncertainty assessment will be computed and tracked on a quarterly basis for those instruments used frequently enough for a quarterly uncertainty assessment, or will be computed on an annual basis for those instruments that are used less frequently. Until these forms and data management systems are completed and sufficient data has been compiled to determine uncertainty using the Type A portion of this hybrid methodology, this operating procedure will assess uncertainty solely through a Type B evaluation.

### **3.2 Initial Implementation (Type B)**

Through a review of the logbooks of historical environmental sampling activities, manufacturer's instruction manuals and the best professional judgment of the Subject Matter Experts (SME)s for their respective field instruments, the SESD Table of Field Measurement Uncertainties (Type B) (SESDFORM-034) has been developed to estimate the Type B uncertainty associated with each type of accredited field instrument utilized by SESD. The uncertainty values found in this table will be used as the field instrument uncertainty findings until the hybrid approach is finalized. The SESD Table of Field Measurement Uncertainties (Type B) will be maintained on the H: drive of the SESD local area network. Once the hybrid approach is finalized, the uncertainty procedure will be revised.

### **3.3 Implementation of Hybrid Type A and B uncertainty methodology**

After the necessary forms and data management systems are completed and sufficient data has been compiled, uncertainty will be determined using a hybrid of both Type A and Type B methodology. The Type B portion of this hybrid methodology is intended to account for the uncertainty associated with the environmental sample. The Type A portion of the hybrid uncertainty assessment will be limited to evaluating the instrument performance against known and traceable calibration standards. The Type A portion of this hybrid uncertainty methodology is only intended to determine instrument variability with respect to known and traceable calibration standards. As such, the instrument response will be compared to the traceable calibration standards and not the environmental sample itself. If the Data Quality Objectives (DQO)s of an environmental investigation require that the Type A uncertainty be determined by comparing the instrument's response to the environmental sample itself, field investigators will follow Section 3.4 of this procedure.

The assessment of uncertainty through this hybrid methodology will start with a field instrument's operating manual and the quoted uncertainties obtained from a calibration standard's certificate. In addition to the daily calibrations being performed in accordance with the instrument's operating manual, project leaders will perform a post-operation instrument verification check (aka unadjusted calibrations). This verification check will be performed using the appropriate standards at the end of the day or after all measurements have been completed for a particular period of operation. These measurements must be recorded in both the field log book and also entered into the Field Data for Measurement Uncertainty Form (SESDFORM-035).

The Field Data for Measurement Uncertainty Form will be submitted electronically via email to the SESD Enforcement and Investigation Branch (EIB) Quality Assurance Officer (QAO) at the completion of the study. The EIB QAO will compile all instrument verification checks obtained from EIB study investigations and compute the uncertainty statistics on a quarterly basis. The EIB QAO will also be responsible for compiling and

computing the uncertainty statistics for the Ecology Assessment Branch (EAB) as well, but the uncertainty statistics will be computed annually.

Type A uncertainty statistics will be computed for each instrument's performance based on these verification checks. At a minimum, these summary statistics will include mean instrument performance as defined by Equation 1, bounded by 95% confidence limits of the mean defined by Equation 4. The Hybrid Uncertainty will be computed as the sum of the Type A uncertainty (computed through verification checks), the Type B uncertainty found from SESDFORM-034, and the uncertainty associated with the calibration standards. Equation 5 will be used to compute the estimated hybrid uncertainty. These instrument uncertainties will be calculated and reported for each piece of measurement equipment subject to the scope of field accreditation. In some cases, measurement uncertainties are provided in percent as opposed to standard deviations. For these cases, equation 6 will be used to convert all uncertainties into standard deviations so that they may be used in equation 5.

These uncertainties will be reported in the quarterly EIB QC report issued in accordance with SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011) and made available to the SESD management, the Field Quality Manager (FQM) and the Branch Field Equipment Manager (BFEM). The yearly uncertainties will be included in the appropriate quarterly EIB QC report and distributed to EAB management and personnel.

Summary statistics computed for the field instrument's performance against known traceable calibration standards will be computed using the equations cited below; where Equation 1 is the mean of the verification checks, Equation 2 is the sample standard deviation, Equation 3 is the standard deviation of the mean (aka standard error), and Equation 4 is the 95% confidence limits for the standard deviation of the mean.

$$\bar{x} = \frac{1}{N} \sum_{i=1}^N x_i$$

**Equation 1 Mean**

where  $x_i$  is the measured verification check, N is the total number of samples, and  $\bar{x}$  is the mean of the verification checks

$$s = \sqrt{\frac{\sum_{i=1}^N (x_i - \bar{x})^2}{N - 1}}$$

**Equation 2 Sample Standard Deviation**

where  $x_i$  is the measured verification check, N is the total number of samples, and  $\bar{x}$  is the mean of the verification checks, and s is the sample standard deviation

$$\sigma_a = \sqrt{\frac{\sum_{i=1}^N (x_i - \bar{x})^2}{N(N-1)}} = \frac{s}{\sqrt{N}}$$

**Equation 3 Standard Deviation of the Mean**

where  $x_i$  is the measured verification check, N is the total number of samples, and  $\bar{x}$  is the mean of the verification checks, s is the sample standard deviation, and  $\sigma_a$  is the standard deviation of the mean verification checks

$$\bar{X}_{\pm 95\%} = \bar{x} \pm t_{0.95, n-1} \cdot \sigma_a$$

**Equation 4 95% Confidence Limits of sample mean**

where  $\bar{x}$  is the mean of the verification checks,  $\sigma_a$  is the standard deviation of this mean,  $t_{0.95, n-1}$  is the 95<sup>th</sup> quantile of a t-distribution with n-1 degrees of freedom, and  $\bar{X}_{\pm 95\%}$  is the 95% confidence range of the mean verification checks

$$\sigma_{hybrid} = \sqrt{\sigma_a^2 + \sigma_b^2 + \sigma_c^2}$$

**Equation 5 Hybrid Uncertainty Estimation**

where  $\sigma_a$  is the type a uncertainty found through the standard deviation of this mean verification checks (Equation 3),  $\sigma_b$  is the type b uncertainty for the field instrument of interest (found in SESDFORM-034-R0),  $\sigma_c$  is the uncertainty associated with the calibration standards, and  $\sigma_{hybrid}$  is the estimated hybrid uncertainty

$$\varepsilon_x \equiv \frac{\sigma_x}{x} * 100$$

**Equation 6 Relative Standard Uncertainty**

where  $\sigma_x$  is the standard deviation of the mean, x is the value of interest, and  $\varepsilon_x$  is the relative standard uncertainty expressed as a percent

### 3.4 Environmental Type A Uncertainty Methodology

In rare circumstances the hybrid methodology cited in Section 3.3, where the Type A portion of the hybrid uncertainty is based solely on an instrument's response to a known standard and not the instrument's response to the environmental sample itself, may not be sufficient for documenting the field measurement uncertainty. For field investigations performed under these circumstances, a Type A uncertainty estimation of the field

instrument's response to the actual environmental sample would be required. In such a case, the uncertainty evaluation will be interpreted in the context of a thorough understanding of the measurement methodology. For example, in large Category 1 projects with many mobile laboratory field measurements, statistical measures might be undertaken to define the uncertainty of the measurements. Even then, the statistical tests would only be reported in conjunction with the following important elements:

- Previous measurement data,
- Experience with, or general knowledge of, the behavior and property of relevant materials and instruments,
- Manufacturer's specifications,
- Data provided in calibration and other reports, and
- Uncertainties assigned to reference data taken from handbooks.

When the Data Quality Objectives (DQO)s of an environmental investigation require that the Type A uncertainty determination be computed solely on the environment sample and not based on instruments' agreement to a known calibration standard or utilizing a Type B uncertainty estimation, in addition to performing the required verification checks against the appropriate known standards, SESD field investigators will perform multiple measurements of the environmental sample in question. A minimum of 7 repeat measurements per environmental sample will be performed to allow for a statistically sound sample size. Means of the instrument response to the environmental sample and their associated 95% confidence intervals will be computed. Means will be computed using Equation 1 of this procedure and the 95% confidence interval of this mean will be computed using Equation 4 of this procedure.