

MEASUREMENT UNCERTAINTY

PRINCIPLES AND RELEVANCE

All types of measurement have some inaccuracy due to bias and imprecision and therefore measurement results can be only estimates of the values of the quantities being measured. To properly use such results environmental laboratories and their users need some knowledge of the accuracy of such estimates. Traditionally, this has been by using the concept of error, but the difficulty with this approach is that the term 'error' implies that the difference between the true value and a test result can be determined and the result corrected which is rarely the case. In contrast, the more recent concept of measurement uncertainty (MU) assumes that significant measurement bias is either eliminated, corrected or ignored, evaluates the random effects on a measurement result, and estimates an interval within which the value of the quantity being measured is believed to lie with a stated level of confidence.

Estimates of MU provide a quantitative indication of the level of confidence that a laboratory has in each measurement and are therefore a key element of an analytical quality system for environmental laboratories. The principles of measurement uncertainty contribute to ensuring test results are fit-for-purpose by:

- defining the quantity intended to be measured (measurand)
- indicating the level of confidence a laboratory has in a given measurement
- providing information essential for the meaningful interpretation of measurement results and their comparison over space and time
- identifying significant sources of MU and opportunities for their reduction.

Outlined in ISO/IEC 17025:2017(E) 3rd Edition: **General requirements for the competence of testing and calibration laboratories** Section 7.6 Evaluation of measurement uncertainty requires the following:

7.6.1 Laboratories shall identify the contributions to measurement uncertainty. When evaluating measurement uncertainty, all contributions that are of significance, including those arising from sampling, shall be taken into account using appropriate methods of analysis.

7.6.2 A laboratory performing calibrations, including of its own equipment, shall evaluate the measurement uncertainty for all calibrations.

7.6.3 A laboratory performing testing shall evaluate measurement uncertainty. Where the test method precludes rigorous evaluation of measurement uncertainty, an estimation shall be made based on an understanding of the theoretical principles or practical experience of the performance of the method.

NOTE: Unless Eurofins are directly involved in sampling this has not been considered in the below values.

Reporting Measurement Uncertainty of Chemical Test Results

In metrology, measurement uncertainty is a non-negative parameter characterising the dispersion of the values attributed to a measured quantity. All measurements are subject to uncertainty and a measurement result is complete only when it is accompanied by a statement of the associated uncertainty. By international agreement, this uncertainty has a probabilistic basis and reflects incomplete knowledge of the quantity value. Measurement uncertainty has been calculated from the respective laboratory control samples (LCS) conducted in each batch of samples (one in every batch of 20 samples) using a minimum of 25 data points according to ASTM E2554-13 Standard Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques. A coverage factor of two ($k=2$) has been used.

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Measurand	Matrix	
	Soil	Aqueous
Per- and Polyfluoro Alkyl Substances (PFASs)		
Perfluorobutanoic acid (PFBA)	24%	29%
Perfluorobutanesulfonic acid (PFBS)	29%	18%
Perfluoro-n-pentanoic acid (PFPeA)	32%	22%
Perfluorohexanoic acid (PFHxA)	22%	22%
Perfluorohexanesulfonic acid (PFHxS)	30%	17%
Perfluoroheptanoic acid (PFHpA)	30%	19%
Perfluorooctanesulfonic acid (PFOS)	19%	10%
Perfluorooctanoic acid (PFOA)	14%	21%
Perfluorononanoic acid (PFNA)	32%	18%
Perfluorodecanoic acid (PFDA)	25%	22%
Perfluorodecanesulfonic acid (PFDS)	30%	38%
Perfluorododecanoic acid (PFDoA)	29%	31%
Perfluoroundecanoic acid (PFUnA)	31%	25%
Perfluorotridecanoic acid (PFTrDA)	36%	40%
Perfluorotetradecanoic acid (PFTeDA)	29%	40%
Perfluorooctanesulfonamide (PFOSA)	32%	26%
1H.1H.2H.2H-perfluorohexanesulfonic acid (4:2 FTS)	34%	31%
1H.1H.2H.2H-perfluorooctansulfonic acid (6:2 FTS)	22%	24%
1H.1H.2H.2H-perfluorodecanesulfonic acid (8:2 FTS)	31%	19%
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	32%	28%
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	27%	27%
Polycyclic Aromatic Hydrocarbons		
Acenaphthene	25%	26%
Acenaphthylene	27%	32%
Anthracene	26%	27%
Benz(a)anthracene	29%	33%
Benzo(a)pyrene	30%	29%
Benzo(b&j)fluoranthene	29%	36%
Benzo(g,h,i)perylene	40%	32%
Benzo(k)fluoranthene	27%	29%
Chrysene	25%	24%
Dibenz(a,h)anthracene	31%	26%

Measurand	Matrix	
	Soil	Aqueous
Fluoranthene	31%	27%
Fluorene	24%	31%
Indeno(1.2.3-cd)pyrene	33%	29%
Naphthalene	25%	27%
Phenanthrene	26%	24%
Pyrene	28%	29%
Phenols (Halogenated)		
2.4.5-Trichlorophenol	29%	41%
2.4.6-Trichlorophenol	33%	41%
2.4-Dichlorophenol	29%	40%
2.6-Dichlorophenol	26%	39%
2-Chlorophenol	26%	40%
4-Chloro-3-methylphenol	30%	42%
Pentachlorophenol	39%	47%
Tetrachlorophenols - Total	33%	42%
Phenols (non-Halogenated)		
2.4-Dimethylphenol	26%	41%
2.4-Dinitrophenol	41%	56%
2-Cyclohexyl-4.6-dinitrophenol	44%	56%
2-Methyl-4.6-dinitrophenol	39%	49%
2-Methylphenol (o-Cresol)	25%	34%
2-Nitrophenol	32%	42%
4-Nitrophenol	42%	40%
Dinoseb	37%	54%
Acid Sulfate Soils - Chromium Suite		
Acid Neutralising Capacity - acidity (ANCbt)	7%	N/A
Acid trail - Titratable Actual Acidity	14%	N/A
Chromium Reducible Sulfur	11%	N/A
HCl Extractable Sulfur	24%	N/A
pH-KCL	2%	N/A
Heavy Metals		
Arsenic	16%	12%
Cadmium	14%	11%
Chromium	17%	10%
Cobalt	15%	11%

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Measurand	Matrix	
	Soil	Aqueous
Copper	17%	12%
Lead	17%	26%
Manganese	15%	11%
Mercury	20%	14%
Nickel	17%	10%
Zinc	17%	12%
Alkali Metals		
Magnesium	NT	16%
Sodium	NT	21%
Potassium	NT	17%
Calcium	NT	19%
Nutrients		
Ammonia (as N)	NT	8.3%
Nitrite (as N)	NT	6.4%
Nitrate (as N)	NT	8.4%
Nitrate & Nitrite (as N)	NT	8.4%
Total Kjeldahl Nitrogen (as N)	NT	27.7%
Ortho Phosphate (as P)	NT	15.87%
Phosphate total (as P)	NT	22.3%
Physico-Chemical Measurements		
pH	NT	2.5%
Conductivity (at 25°C)	NT	12.7%
Suspended Solids (SS)	NT	12.3%
Total Dissolved Solids (TDS)	NT	9.6%
Biochemical Oxygen Demand (BOD-5 Day)	NT	14.2%
Chemical Oxygen Demand (COD)	NT	12.6%
Total Organic Carbon (TOC)	NT	12.8%

Air Toxics – Summa Canister US EPA Method TO-15	
Measurand	Air
Vinyl Chloride	19%
Trichlorofluoromethane (Freon 11)	12%
1,1,1-trichloroethane (TCE)	17%
Benzene	16%
Chlorobenzene	16%
Naphthalene	24%

Air Toxics – Thermal Desorption US EPA Method TO-17	
Measurand	Air
Vinyl Chloride	27%
Trichlorofluoromethane (Freon 11)	27%
1,1,1-trichloroethane (TCE)	31%
Benzene	26%
Chlorobenzene	27%
Naphthalene	29%

Air Toxics – Summa Canister ASTM D1945/D1946	
Measurand	Air
Methane	9%
Hydrogen	2%
Oxygen	2%
Carbon Dioxide	9%
Helium	6%
Ethane	11%

NT – Not Tested

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Reporting Measurement Uncertainty of Microbiology Test Results

The American Association for Laboratory Accreditation (A2LA) provides a technical note G108 - Guidelines for Estimating Uncertainty for Microbiological Counting Methods that is used for the estimation of measurement uncertainty for methods that use counting for determining the number of colonies in a test sample. The data has been applied for all quantitative microbiological methods, including plate count and MPN. The data below are based on 20 data points each but larger datasets when available produce more reliable estimates and smaller data sets may be used with caution. The coverage factor used is obtained from the Student t-tables to estimate expanded uncertainty for smaller datasets.

Reproducibility Replicates for Laboratory Control Samples

This procedure illustrates the use of "reproducibility replicates" to estimate uncertainty for the same type of sample matrix analysed. This technique captures various sources of uncertainty that can affect routine samples by having "replicates" produced independently under as many different conditions as possible that are received routinely. This procedure presents the techniques recommended in ISO TS19036: *Microbiology of foods and animal feeding stuffs – Guidelines for the estimation of measurement uncertainty for quantitative determinations*. The results are from control samples which have been analysed through all of the steps of the test method and were set up on different days, in duplicate, by different analysts, using different equipment (e.g. balances, pipettors) and also using different batches of media/reagents.

Measurand	Water Matrix	
	Low range	Upper range
Microbiology		
Legionella by AS3896: 2008	-33%	+50%
Total Coliforms by filtration (MF)	-22%	+28%
Thermotolerant Coliforms by filtration	-22%	+28%
<i>E.coli</i> by filtration (MF)	-17%	+21%
Enterococci by filtration (MF)	-18%	+22%
<i>Pseudomonas aeruginosa</i> by MF	-30%	+42%
<i>Clostridium perfringens</i> by MF	-14%	+16%
<i>E.coli</i> by Defined Substrate Technology	-20%	+25%
Total Coliforms by Defined Substrate#	-22%	+29%
Enterococci by Defined Substrate	-14%	+16%
Standard Plate Count (TPC-2)	-20%	+25%
Cooling Towers Plate Count (TPC-4)	-27%	+36%
Somatic Coliphages (100mL)	-13%	+15%
Male-specific or fRNA Coliphages*	-27%	+36%

- Defined Substrate Technologies (DST) include enzyme detection methods such as Colilert™, Enterolert™, Colitag™