Measurement Uncertainty

Eurofins Environment Testing Australia Pty Ltd ABN: 50 005 085 521
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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.

MEASUREMENT UNCERTAINTY

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Measurand	Matrix	
	Soil	Aqueous
Per- and Polyfluoroalkyl Substances (PFAS)		
Perfluoropropanesulfonic acid (PFPrS)	33.0 %	42.0 %
Perfluorobutanoic acid (PFBA)	14.7 %	17.6 %
Perfluorobutanesulfonic acid (PFBS)	18.8 %	18.9 %
Perfluoropentanoic acid (PFPeA)	26.8 %	25.9 %
Perfluorohexanoic acid (PFHxA)	19.0 %	24.9 %
Perfluorohexanesulfonic acid (PFHxS)	14.9%	17.7 %
Perfluoroheptanoic acid (PFHpA)	18.3 %	24.5 %
Perfluorooctanesulfonic acid (PFOS)	17.4 %	21.3 %
Perfluorooctanoic acid (PFOA)	15.8 %	18.8 %
Perfluorononanoic acid (PFNA)	14.8 %	18.3 %
Perfluorodecanoic acid (PFDA)	18.6 %	21.3 %
Perfluorodecanesulfonic acid (PFDS)	35.3 %	45.2 %
Perfluoroundecanoic acid (PFUnA)	20.1 %	23.1 %
Perfluorododecanoic acid (PFDoA)	15.4 %	24.9 %
Perfluorotridecanoic acid (PFTrDA)	36.2 %	41.9 %
Perfluorotetradecanoic acid (PFTeDA)	19.5 %	28.1 %
Perfluorooctanesulfonamide (PFOSA)	20.0 %	23.0 %
1H.1H.2H.2H-perfluorohexanesulfonic acid (4:2 FTSA)	17.2 %	21.5 %
1H.1H.2H.2H-perfluorooctansulfonic acid (6:2 FTSA)	20.0 %	22.9 %
1H.1H.2H.2H-perfluorodecanesulfonic acid (8:2 FTSA)	25.5 %	28.6 %
1H, 1H, 2H, 2H-perfluorododecane sulfonate (10:2 FTSA)	47.8 %	54.3 %
N-ethyl-perfluorooctanesulfonamidoacetic acid	21.9 %	27.3 %
N-methyl-perfluorooctanesulfonamidoacetic acid	29.2 %	25.2 %
N-Methylperfluorooctane sulfonamide (MeFOSA)	19.9 %	27.3 %
N-Ethylperfluorooctane sulfonamide (EtFOSA)	30.4 %	37.6 %
N-Methylperfluorooctane sulfonamidoethanol (MeFOSE)	22.8 %	29.7 %
N-Ethylperfluorooctane sulfonamidoethanol (EtFOSE)	30.4 %	27.5 %
Organophosphorus Pesticides (OPP)		
Diazinon	28.4 %	23.7 %
Ethion	32.9 %	28.8 %
Mevinphos	31.5 %	25.2 %
Dimethoate	30.3 %	31.5 %
Organochlorine Pesticides (OCP) & Aroclor 1260		
4,4'-DDT	25.6 %	20.2 %
4,4'-DDE	27.3 %	30.2 %
Dieldrin	26.6 %	25.2 %
Hexachlorobenzene	29.0 %	31.5 %
Chlordanes - Total	27.1 %	25.2 %
γ-HCH (Lindane)	27.3 %	30.7 %
Aroclor 1260	27.9 %	26.1 %
Polycyclic Aromatic Hydrocarbons (PAH)		
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 %
Fluoranthene	31 %	27 %
Fluorene	24 %	31 %

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Measurand	Matrix	
	Soil	Aqueous
Indeno(1.2.3-cd)pyrene	33 %	29 %
Naphthalene	25 %	27 %
Phenanthrene	26 %	24 %
Pyrene	28 %	29 %
Dioxins (PCDDs/PCDFs)		
2,3,7,8-TCDD	9.2 %	8.0 %
1,2,3,7,8-PeCDD	9.0 %	7.4 %
2,3,7,8-TCDF	11.0 %	9.5 %
1,2,3,7,8-PeCDF	8.1 %	4.8 %
2,3,4,7,8-TCDF	8.5 %	5.3 %
OCDD	6.3 %	4.3 %
OCDF		13.3 %
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 %
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 %

Measurand	Matrix	
	Soil	Aqueous
Dinoseb	37 %	54 %
BETXN		
Benzene	23.3 %	22.2 %
Ethyl benzene	26.3 %	22.3 %
Toluene	23.6 %	21.1 %
Xylenes	24.7 %	22.9 %
Naphthalene	31.3 %	23.6 %
VOC		
Ethanol	NT	11.6 %
Methyl-tert-butyl ether (MTBE)	26.2 %	20.5 %
1,1,1-Trichloroethane	22.0 %	21.4 %
1,2-dichlorobenzene	24.3 %	22.2 %
Trichloroethene (TCE)	24.9 %	20.8 %
Total Recoverable Hydrocarbons (TRH)		
TRH $>C_6$-C_{10}	26 %	28 %
TRH $>C_{10}$-C_{16}	31 %	31 %
TRH $>C_{16}$-C_{34}	20 %	20 %
TRH $>C_{34}$-C_{40}	24 %	24 %
Acid Sulfate Soils - CRS 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		
Aluminium	18.9 %	13.7 %
Arsenic	16.0 %	12.0 %
Barium	18.8 %	11.5 %

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Measurand	Matrix	
	Soil	Aqueous
Beryllium	20.5 %	14.2 %
Boron	22.1 %	18.5 %
Cadmium	14.0 %	11.0 %
Chromium	17.0 %	10.0 %
Hexavalent Chromium	10.4 %	13.6 %
Cobalt	15.0 %	11.0 %
Copper	17.0 %	12.0 %
Lead	17.0 %	26.0 %
Manganese	15.0 %	11.0 %
Mercury	20.0 %	14.0 %
Molybdenum	16.8 %	12.3 %
Nickel	17.0 %	10.0 %
Selenium	21.1 %	11.1 %
Silver	14.1 %	15.5 %
Tin	18.5 %	11.4 %
Uranium	17.1 %	14.4 %
Zinc	17.0 %	12.0 %
Heavy Metals (filtered)		
Arsenic (filtered)	NT	13.5 %
Cadmium (filtered)	NT	10.8 %
Chromium (filtered)	NT	13.0 %
Cobalt (filtered)	NT	14.2 %
Copper (filtered)	NT	13.9 %
Lead (filtered)	NT	13.1 %
Manganese (filtered)	NT	11.7 %
Mercury (filtered)	NT	14.8 %
Nickel (filtered)	NT	13.8 %
Zinc (filtered)	NT	13.5 %

Measurand	Matrix	
	Soil	Aqueous
Silver (filtered)	NT	11.3 %
Alkali Metals		
Magnesium	NT	16 %
Sodium	NT	21 %
Potassium	NT	17 %
Calcium	NT	19 %
Water Laboratory		
Acidity (as $CaCO_3$)	NT	7.6 %
Total Alkalinity (as $CaCO_3$)	NT	12.5 %
Colour (Pt/Co) True	NT	12.2 %
Cyanide Total	28.9 %	22.2 %
Cyanide WAD	NT	19.2 %
Cyanide Free	NT	22.5 %
Chloride (1:5 aqueous extract)	18.8 %	NT
Chloride	NT	11.1 %
Fluoride (ISE)	NT	29.1 %
MBAS (MW: 288)	NT	12.1 %
Sulfate (as SO4) (1:5 aqueous extract)	20.6 %	NT
Sulfate (as SO4)	NT	9.1 %
Sulfide (as S)	NT	10.0 %
Sulfite (as S)	NT	6.3 %
Thiosulfate (as S)	NT	16.0 %
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	20.2 %

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Measurand	Matrix	
	Soil	Aqueous
Ortho Phosphate (as P)	NT	15.9 %
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	15.4 %

Measurand	Matrix	
	Soil	Aqueous
Biochemical Oxygen Demand (BOD5 Day)	NT	14.2 %
Chemical Oxygen Demand (COD)	NT	12.6 %
Oil & Grease (HEM)	NT	10.7 %
Total Organic Carbon (TOC)	NT	12.8 %
Turbidity	NT	8.2 %

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Measurand	Matrix Air
US EPA Method TO-15 Air Toxics – Pressurised Canister	
Vinyl Chloride	16.8 %
Trichlorofluoromethane (Freon 11)	12.7 %
1,2-Dichlorotetrafluoroethane (Freon 114)	14.5 %
1.2-Dichloroethane	21.3 %
1.4-Dichlorobenzene	21.1 %
1.1-Dichloroethene	11.4 %
Tetrachloroethene (PCE)	13.3 %
1,1,1-Trichloroethane (TCE)	15.8 %
Benzene	13.3 %
Toluene	16.2 %
Ethylbenzene	16.1 %
Chlorobenzene	14.6 %
Naphthalene	18.3 %
ASTM D1945/D1946 Air Toxics – Pressurised Canister	
Methane	9 %
Hydrogen	2 %
Oxygen	2 %
Carbon Dioxide	9 %
Helium	6 %
Ethane	11 %
US EPA Method TO-17 Air Toxics – Thermal Desorption	
Vinyl Chloride	27 %
Trichlorofluoromethane (Freon 11)	27 %
1,1,1-trichloroethane (TCE)	31 %
Benzene	26 %
Chlorobenzene	27 %
Naphthalene	29 %

Measurand	Matrix Air
US EPA Method 23 – XAD-2 Emission Cartridge	
2,3,7,8-TCDD	9.5 %
1,2,3,7,8-PeCDD	5.7 %
OCDD	2.5 %
2,3,7,8-TCDF	9.0 %
2,3,4,7,8-PeCDF	8.1 %
OCDF	10.2 %
CARB Method 429 – XAD-2 Emission Cartridge	
Benzo(a)pyrene	22.5 %
Acenaphthene	9.6 %
Benz(a)anthracene	9.6 %
Dibenz(a.h)anthracene	20.8 %
Benzo(e)pyrene	30.1 %
Benzo(g.h.i)perylene	23.5 %
Asbestos (fibre counts)	**Air**
Low Density (Fibres ≤33 f/mm^2)	9.8 %
Medium Density (Fibres <33 f/mm^2 and <50 f/mm^2)	8.0 %
High Density (Fibres ≥50 f/mm^2)	17.3 %

Measurand	Matrix Wipes
Methamphetamine and Associated Precursor Compounds	
Ephedrine	8.2 %
Pseudoephedrine	2.5 %
Amphetamine	7.8 %
Methamphetamine	5.8 %
MDA	14.9 %
MDMA	2.3 %

NT = Not Tested

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Asbestos - Because of the nature of the Membrane Filter Method, it is not possible to know the `true' airborne fibre concentration of a given dust cloud. For this reason it is not possible to assess the likely accuracy of the method. Even the precision (or repeatability) of the method is difficult to quantify because of systematic errors which tend to arise both within and between laboratories. Taken as a whole, by `randomly' selecting observers and laboratories, these systematic errors take on a random nature such that it may be possible in the future to provide estimates of empirical precision (that is the closest approach possible to a statement of accuracy for a method with known `true' values).
Much work has been done in an attempt to arrive at these estimates, and to date only a partial conclusion has been reached. Examples of confidence intervals calculated from the Poisson distribution are presented in Table 1 below:

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TABLE 1: THEORETICAL CONFIDENCE INTERVAL FOR RESULTS USING POISSON DISTRIBUTION

Number of Fibres Counted per 100 Graticule Areas	95 % Confidence Interval for Result
100	± 20 % of the calculated result
40	-26 % to +36 % of the calculated result
10	-50 % to +84 % of the calculated result (that is, the true result may be in the range of 50-184 % of the calculated result)

Confidence limits apply to the measured result and not the final reported result, which is a rounded-off representation of the measured result. Other sources of random and systematic errors add significantly to the uncertainty in estimating the airborne asbestos dust concentration, and these have been known to increase the above confidence intervals by up to a factor of 2 or 3. Table 2 and Table 3 present the findings of empirical studies in the United States into the precision of the Membrane Filter Method in estimating airborne asbestos concentrations. There is no reason to assume that this variability would not be reflected in Australia.

TABLE 2: COEFFICIENTS OF VARIATIONS FOR EXPERIENCED LABORATORIES

Total No. of Fibres Counted	Coefficients of Variations[1] Analytical Only	Sampling & Analytical
10	0.60	0.90
15	0.55	0.80
40	0.45	0.70
100	0.40	0.65

[1] The Coefficient of Variation (CV) is calculated by dividing the standard deviation by the arithmetical average of a set of fibre concentrations determined with a number no reason to assume that this variability would not be reflected in Australia.

TABLE 3: 90% CONFIDENCE LIMITS DERIVED FROM EMPIRICAL STUDIES

Total No. of Fibres Counted	Analytical		Sampling & Analytical	
	LCL	UCL	LCL	UCL
10	3	21	2	26
15	6	31	4	37
40	18	74	12	93
100	49	175	31	222

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REPORTING MEASUREMENT UNCERTAINTY OF MYCOLOGY 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 below are based on at least 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, microscopes, stages etc.) and were calculated from seven cross-checks at each debris rating. The genera/phyla highlighted in bold below were the most frequently detected and used to calculate MU.

Acremonium sp.
Aspergillus sp.
Aspergillus/Penicillium Types
Chaetomium sp.
Cladosporium sp.
Epicoccum sp.
Stachybotrys sp.
Tricoderma sp.
Alternaria sp.
Arthrinium sp.
Ascoscarp
Ascospores
Aureobasidium sp.
Basidiospores
Bipolaris/Drechslera
Botrytis sp.
Cercospora
Curvularia sp.
Fusarium sp.
Ganoderma
Geotrichium sp.
Memnoniella sp.
Nigrospora sp.
Paecilomyces sp.
Pithomyces sp.
Polythrincium
Pyricularia sp.
"Smuts/*Myxomycetes*/*Periconia*/Rusts"
Scopulariopsis sp.
Spegazzinia sp.
Stemphylium sp.
Tetraploa sp.
Torula sp.
locladium sp.
Yeast
Zygomycetes

Measured	Air-O-Cells® Matrix		
	Upper Range	Medium Range	Low Range
Fungal Structures (fs/m^3)	60	20	5

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SAMPLING[2]

The main purpose of measurement is to enable decisions to be made. The reliability of these decisions depends on knowing the uncertainty of the measurement results. If the uncertainty of measurements is underestimated, for example because the sampling is not taken into account, then erroneous decisions may be made that can have large financial consequences. The fitness for purpose of measurement results can only be judged by having reliable estimates of their uncertainty. For this reason, it is essential that effective procedures are available for estimating the uncertainties arising from all parts of the measurement process. These must include uncertainties arising from any relevant sampling and physical preparation. Judgements on whether the analytical contribution to the uncertainty is acceptable can only be made with knowledge of the uncertainty originating in the rest of the measurement procedure.

Sampling theory has developed largely independently of analytical chemistry and chemical metrology. Sampling quality has generally been addressed in sampling theory by the selection of a 'correct' sampling protocol, appropriate validation, and training of sampling personnel (i.e. samplers) to ensure that this protocol is applied correctly. It is then assumed that the samples will be representative and unbiased, and the variance will be that predicted by the model. An alternative approach is to estimate the uncertainty of sampling for typical materials, or for sampling targets, during validation of the sampling protocol, and to confirm compliance in practice using ongoing quality control. This is more consistent with procedures already in place for the rest of the measurement process. Interestingly, the quality of sampling is only quantifiable through the measurements that are made upon the resultant samples.

Sampling protocols have been written to describe the recommended procedure for the sampling of innumerable types of material and for many different chemical components. These protocols are sometimes specified in regulation or in international agreements. These procedures rarely identify the relative contributions of sampling and chemical analysis to the combined uncertainty.
Figure 1 shows the 'cause-and-effect diagram' for the measurement process. In the sampling and sample preparation steps the sources of uncertainty contributions are given; for the analysis, only the analytical quality parameters are indicated.

[2] EURACHEM / CITAC Guide Measurement uncertainty arising from sampling A guide to methods and approaches Produced jointly with EUROLAB, Nordtest and the UK RSC Analytical Methods Committee First Edition 2007

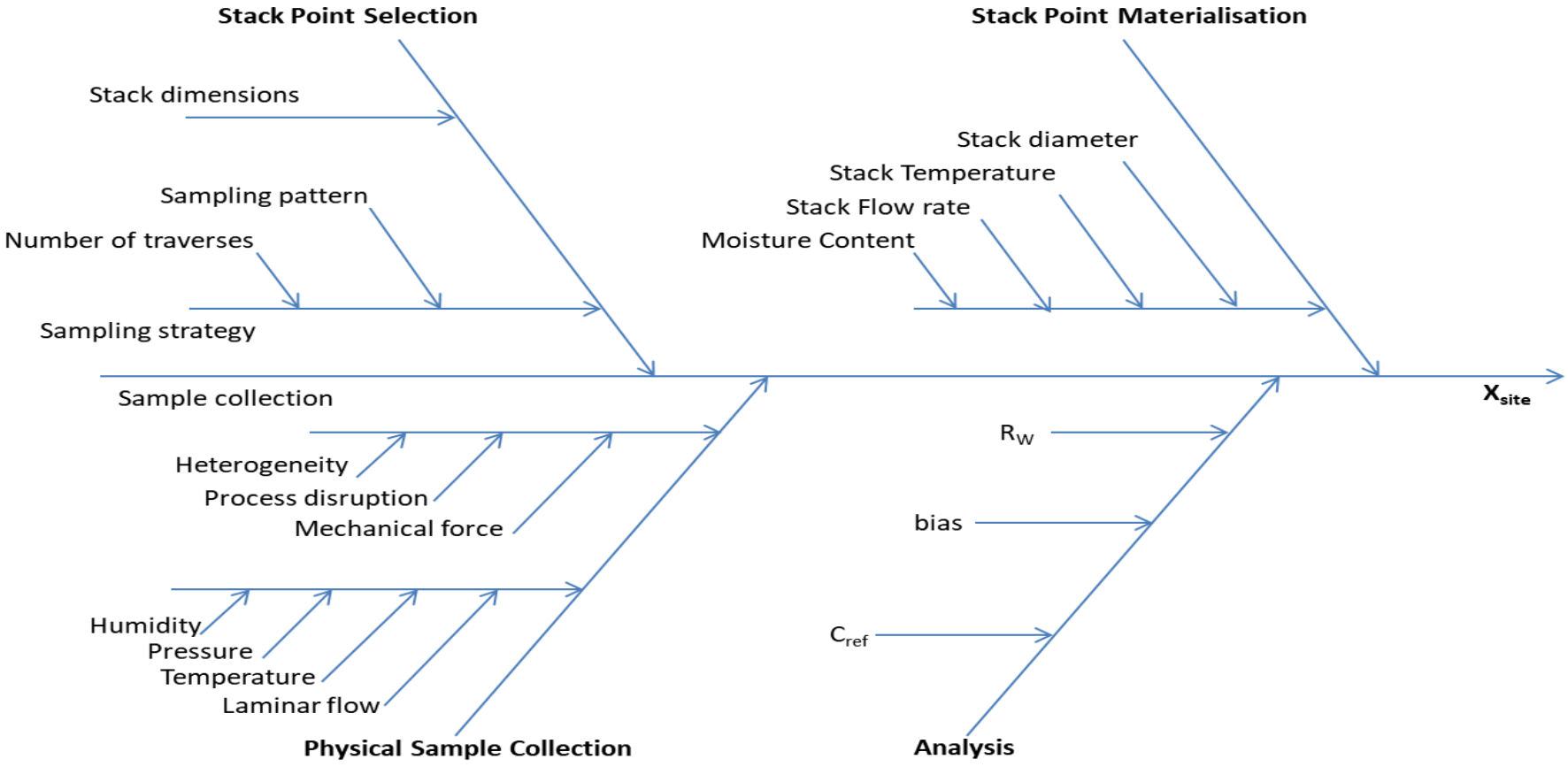

FIGURE 1: CAUSE-AND-EFFECT DIAGRAM FOR STACK SAMPLING OF EMISSIONS FROM A STATIONARY SOURCE (RW IS WITHIN-LABORATORY REPRODUCIBILITY)

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Table 4: STANDARD UNCERTAINTY COMPONENTS AND COMBINED UNCERTAINTY IN THE ANALYSIS OF THE EMISSION SAMPLE FOR PCDDS/PCDFS

R_W	Uncertainty from within-laboratory reproducibility, evaluated from the repeatability standard deviation of the mean from n=1 test samples	U_{Rw} = 1.7%
C_{ref} Bias S_{bias}	Uncertainty for the trueness of the results estimated as the reproducibility precision sR from one interlaboratory comparison (worse case estimate)	U_{bias} = 9.5%
	Combined analytical uncertainty	U_{anly} = 9.7%

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