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|  <p style="text-align: center;"><b>AUSTRALIA-ENVIRONMENT, HEALTH &amp; SAFETY</b></p> <p style="text-align: center;"><b>QA/QC PLAN</b></p> | Doc. No.   | MP-AU-ENVGEN-QU-022 |
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|  | Author     | P Bamford           |

SGS Environment, Health and Safety is accredited by NATA for Chemical Testing (Accreditation.No.2562) and Quality System compliance to ISO/IEC 17025. The QC parameters contained within are designed to meet current NEPM (USEPA) requirements.

**QUALITY CONTROL SAMPLES:**

Typical QC included in any analytical run is listed below.

|  |  |
|--|--|
| <b>Reagent/Analysis Blank (BLK) &amp; Method Blank (MB)</b>                                | <p>Sample free reagents carried through the preparation/extraction/digestion procedure and analysed at the beginning of every sample batch analysis. A reagent blank is prepared and analysed with every batch of samples plus with each new batch of solvent prior to use and at least thereafter every 20 samples plus at the end of each batch.</p>   |
| <b>Sample Matrix Spike (MS) &amp; Matrix Spike Duplicate (MSD)</b>                         | <p>Sample replicates spiked with identical concentrations of target analyte(s). The spiking occurs during the sample preparation and <u>prior to the extraction/ digestion procedure</u>. They are used to document the precision and bias of a method in a given sample matrix. Where there is not enough sample available to prepare a spiked sample, another known soil/sand or water may be used.</p> <p>A Sample Matrix Spike and a duplicate spiked sample is analysed in every lab batch or at every 20 samples, whichever is smaller. The MSD may be omitted where Lab Duplicates are being run.</p> |
| <b>Surrogate Spike (SS)</b>  | <p>Used to determine the extraction efficiency. They are organic compounds which are similar to the target analyte(s) in chemical composition and behaviour in the analytical process, but which are not normally found in environmental samples. Where possible they are surrogate compounds recommended by the USEPA.</p> <p>At least one but up to four surrogate compounds are added to all samples requiring analysis for organics prior to extraction.</p>   |
| <b>Control Matrix Spike (CMS)</b>  | <p>To ensure spike recoveries can be determined for every batch of samples a control matrix is spiked with identical concentrations of target analyte(s) and then analysed. These results allow recoveries to be determined in the event that the matrix spikes are unusable (e.g., matrix spikes performed on heavily contaminated samples). (This is equivalent to LCS as below)</p> <p>These are analysed in every batch or at least every 20 samples, whichever is smaller.</p>  |
| <b>Internal Standard (IS)</b>  | <p>Added to all samples requiring analysis for organics (where relevant) after the extraction process; the compounds serve to give a standard of retention time and response, which is invariant from run-to-run with the instruments. Where possible they are standard compounds recommended by the USEPA.</p>  |
| <b>Lab Duplicates (D)</b>  | <p>A separate portion of a sample being analysed that is treated the same as the other samples in the batch.</p> <p><i>The number of duplicates analysed shall be one for every 1-10 samples. i.e., Lab batch of 1-10 samples requires 1 duplicate, Lab batch of 11-20 samples requires 2 duplicates, etc.</i></p>   |
| <b>Lab Control Samples (LCS)</b>   | <p>Prepared from a source independent of the calibration standards.</p> <p>At least one control standard is included in each run to confirm calibration validity. Thereafter they are analysed at least every one in 20 samples plus at the end of each analytical run.</p>  |
| <b>Continuous Calibration Verification (CCV) or Calibration Check Standard &amp; Blank</b> | <p>A calibration check standard or CCV are run after every 20 samples of an instrumental analysis run to assess analytical drift.</p> <p>Calibration Standards are checked old versus new with criteria as follows:<br/><i>Organics ±20%, Inorganics/elements ±10%.</i></p>  |

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**INTERNAL QC SAMPLE ACCEPTANCE CRITERIA:**

Unless otherwise specified in the method or method manual the following general criteria apply to all tests. All recoveries are to be reported to 3 significant figures.

Failure to meet the internal acceptance criteria will result in sample batch repeats dependent upon investigation outcomes. For data to be accepted:

**Inorganics - water samples**

- For all inorganic analytes the Reagent & Method Blanks must be less than the LOR. Failures apply to preparation batch only; all positives to be repeated – negatives not affected.
- The Calibration Check Standards or Continuous Calibration Verification (CCV) must be within  $\pm 15\%$ . Failures apply to samples run since last “successful” calibration check. These samples are to be re run.
- Control Standards must be 80-120% of the accepted value. Failures require a review of the procedure, not a repeat of the work unless other QC fails.
- The Calibration Check Blanks must be less than the LOR. Failures apply to calibration only.
- Lab Duplicates RPD to be  $<15\%^2$ . Note: If client field duplicates do not meet this criteria, it may indicate heterogeneity and shall be noted on the data reports for QC samples. Failure applied to preparation batch only; all positives and 1/3 negatives to be repeated (with new QC).
- Sample (and if applicable Control) Matrix Spike Duplicate recovery RPD to be  $<30\%$ . Failure applied to preparation batch only; all positives and 1/3 negatives to be repeated (with new QC).
- Where CRMs are used, results to be within  $\pm 2$  standard deviations of the expected value. Failures require a review of the procedure, not a repeat of the work unless other QC fails.

**Inorganics - soil samples**

- For all inorganic analytes the Reagent & Method Blanks must be less than the LOR. Failures apply to preparation batch only; all positives to be repeated – negatives not affected.
- The Calibration Check Standards or Continuous Calibration Verification (CCV) must be within  $\pm 15\%$ . Failures apply to samples run since last “successful” calibration check. These samples are to be re-run.
- Control Standards must be 80-120% of the accepted value. Failures require a review of the procedure, not a repeat of the work unless other QC fails.
- The Calibration Check Blanks must be less than the LOR. Failures apply to calibration only.
- Lab duplicate RPD to be  $<30\%^2$ . Failure applied to preparation batch only; all positives and 1/3 negatives to be repeated (with new QC).
- Sample Matrix Spike Duplicate (MS<sup>3</sup>/MSD) recovery RPD to be  $<30\%$ . In the event that the matrix spike has been applied to samples whose matrix or contamination is problematic to the method then these acceptance criteria apply to the Control Matrix Spike (CMS/D). Failure applied to preparation batch only; all positives and 1/3 negatives to be repeated (with new QC).
- Where CRMs are used, results to be within  $\pm 2$  standard deviations of the expected value. Failures require a review of the procedure, not a repeat of the work unless other QC fails.

**Organics (including Airtox)**

- Volatile & extractable Reagent & Method Blanks must contain levels less than or equal to LOR. Failures apply to preparation batch only; all positives to be repeated – negatives not affected.
- The Calibration Check Standards or Continuous Calibration Verification (CCV) must be within  $\pm 20\%$ . Some analytes may have specific criteria. Failures apply to samples run since last “successful” calibration check. These samples are to be re run.



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- Control Standards (LCS/CMS) and Certified Reference Materials (CRM) recoveries are to be within established control limits or as a default 60-140% unless compound specific limits apply. Failures require a review of the procedure, not a repeat of the work unless other QC fails.
- Retention times are to vary by no more than 0.2 min.
- **At least two of three** routine level soil sample Surrogate Spike (SS) recoveries are to be within 70-130% where control charts have not been developed and within the established control limits for charted surrogates. Matrix effects may void this as acceptance criterion. Any recoveries outside these limits will have comment. Failures apply to relevant sample only.
- Water sample Surrogates Spike (SS) recoveries are to be within 40-130%. The presence of emulsions, surfactants and particulates may void this as an acceptance criterion. Any recoveries outside these limits will have comment. Failures apply to relevant sample only.
- Lab Duplicates<sup>1</sup>(D) must have a RPD <30%<sup>2</sup>. Failure applied to preparation batch only; all positives and 1/3 negatives to be repeated (with new QC).
- Sample Matrix Spike Duplicate<sup>1</sup>: (MS<sup>3</sup>/MSD) recovery RPD to be <30%. In the event that the matrix spike has been applied to samples whose matrix or contamination is problematic to the method then these acceptance criteria apply to the Control Matrix Spike (CMS/D). Failure applied to preparation batch only; all positives and 1/3 negatives to be repeated (with new QC).

**Microbiology**

- Lab duplicates are run once per test for each day the test is run.
- Positive and negative controls are run each day the test is performed

**Notes:**

<sup>1</sup>For water samples, laboratory duplicates and matrix spikes will be performed when sufficient additional sample is provided.  
<sup>2</sup>The RPD is evaluated against the Maximum Allowable Difference (MAD) criteria and can be graphically represented by a curve calculated from the Statistical Detection Limit (SDL) and Limiting Repeatability (LR) using the formula: MAD = 100 x SDL / Mean + LR. Where the Maximum Allowable Difference evaluates to a number larger than 200 it is displayed as 200. Application of more stringent criteria may be applied for clean water sample from water boards and any other nominated client contracts.  
<sup>3</sup>Matrix Spike do not readily equate to definitive recovery due to inherent matrix interferences and thus do not have recovery compliance values set. As a guide, inorganic recoveries should be between 70-130% and for organics 60-130%

**ANALYTICAL BATCH STRUCTURE:**

An analytical batch is nominally considered as 20 samples or smaller. As a standard template the following should be **used as a guide** according to the above Quality Control Types:

|    |                           |    |                           |
|----|---------------------------|----|---------------------------|
| 1  | MB                        | 17 | UNK_DUP                   |
| 2  | STD1                      | 18 | MS                        |
| 3  | STD2                      | 19 | MS_DUP                    |
| 4  | STD3                      | 20 | UNK 11                    |
| 5  | LCS                       | 21 | UNK 12                    |
| 6  | BLK                       | 22 | UNK 13                    |
| 7  | UNK 1                     | 23 | UNK 14                    |
| 8  | UNK 2                     | 24 | UNK 15                    |
| 9  | UNK 3                     | 25 | UNK 16                    |
| 10 | UNK 4                     | 26 | UNK 17                    |
| 11 | UNK 5                     | 27 | UNK 18                    |
| 12 | UNK 6                     | 28 | UNK 19                    |
| 13 | UNK 7                     | 29 | UNK 20 (SS if applicable) |
| 14 | UNK 8                     | 30 | UNK_DUP                   |
| 15 | UNK 9                     | 31 | CCV                       |
| 16 | UNK 10 (SS if applicable) | 32 | SRM / CMS / LCS           |



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**QC SAMPLE FAILURES:**

QC failures may occur for a number of reasons and may include the following:

- **Matrix Interference:** Matrix interference refers to sample characteristics that interfere with the test method execution such that reliable data cannot be generated. Examples of matrix interference include samples with extreme pH, high alkalinity or acidity, and chemical constituents that react with target analytes. Common matrix interference is the presence of a non-target compound in high concentrations. Matrix interferences may void surrogate recoveries. Failures will apply to specific samples only. Comment to be included on the final analytical report.
- **Sample Heterogeneity:** refers to where a sample is not uniform (or homogeneous) in composition or character. Sample heterogeneities may cause lab duplicates to fail acceptance criteria. Issues of this nature will be considered on a case by case basis and reported as required.
- **Highly Concentrated Samples:** Some samples may be affected by or have significant concentrations of a spike compound present in the sample matrix which may make it difficult to obtain an acceptable recovery for a spike compound. Issues of this nature will be considered on a case by case basis and reported as required. In cases of this nature samples may require subsequent dilution which may result in the limit of reporting (LOR) being increased.

Where QC failures have been noted during the analytical process, appropriate comments will be provided on the final analytical reports providing detail of the probable cause of the failure.

**QUALITY ASSURANCE PROGRAMS:**

The following QAPs listed below are routinely used to monitor the validity of results:

|   |   |
|---|---|
| <b>Statistical analysis of Quality Control data (SQC)</b> | Quality control data is plotted on control charts using the APHA procedure with warning and control limits at 2 and 3 standard deviations respectively.   |
| <b>Certified Reference Materials (CRM/SRM)</b>            | Certified Reference Materials and Standards are analysed for method validation. These materials/standards have certified reference values for various parameters (Provider accredited to ISO 17034). May also be used for method troubleshooting.   |
| <b>Proficiency Testing</b>                                | Regular proficiency test samples are analysed by our laboratories. SGS Environment, Health & Safety participates in a number of programs. Results and proficiency status are compiled and sent to participating laboratory post data interpretation. Failure to comply with acceptable values result in further investigations. |
| <b>Inter-laboratory &amp; Intra-laboratory Testing</b>    | SGS Environment, Health & Safety has schedules in the Quality Systems to participate in Inter/Intra laboratory testing conducted internally and by other parties. This to cover tests not provided by PT providers and/or to cover infrequently used methods.   |