LQMS
LABORATORY QUALITY MANAGEMENT SERVICES PTY LTD

20 Lockhart Ave CASTLE HILL NSW 2154 Tel: +61 2 9659 3001 Fax: +61 2 9659 0033 ABN 15 064 466 502
e-mail: johneames@lqms.com.au www.lqms.com.au

REVIEW OF ANALYTICAL PROCEDURES USED & DATA PRODUCED BY SGS AUSTRALIA PTY LTD FOR THE MAGELLAN METALS PTY LTD “LEAD IN SHIPPING CONTAINER MONITORING PROGRAM”

John Eames

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LABORATORY QUALITY MANAGEMENT SERVICES PTY LTD

REVISION TABLE

Version Number	Date	Changes to Content
Version 1	February 2011	Original Document
Version 2	March 2011	Section 5.1 amended. SGS's configuration of LTech LIMS1 was the cause of the laboratory not being able to distinguish between high volume samples and preloaded cassette MCE filter assemblies rather than inadequacies in the LIMS.

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1 ABBREVIATIONS

COC	Chain of Custody
CRM	Certified Reference Material
FAA	Flame Atomic Absorption
GFAA	Graphite Furnace Atomic Absorption
HCl	Hydrochloric Acid
HNO_3	Nitric Acid
ICP-MS	Inductively Coupled Plasma Mass Spectrometer
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometer
IDL	Instrument Detection Limit
Inspectorate	Inspectorate Australia (Assay) Pty Ltd trading as Inspectorate Cox
LIMS	Laboratory Information Management System
LIMS1	LTech Australia LIMS
LOR	Limit of Reporting
LQMS	Laboratory Quality Management Service Pty Ltd
MCE	Mixed Cellulose Ester filter membranes
MDL	Method Detection Limit
NATA	National Association of Testing Authorities
OEPA	Office of the Environmental Protection Authority
pdf	Adobe Portable Document Format
QA	Quality Assurance
QC	Quality Control
% RSD	Percent Relative Standard Deviation
SGS	SGS Australia Pty Ltd
SLIMS	Mincom CCLAS LIMS
SOP	Standard Operating Procedure

2 EXECUTIVE SUMMARY

This report provides the Office of the EPA with an independent evaluation of the SGS Australia Pty Ltd analytical results of the Magellan Metals lead in container air samples.

The review process involved examining all the SGS Australia Pty Ltd ICP-OES and ICP-MS data for sample batches in which the filter digestates were run and quantified as part of the Magellan Metals lead in container air samples monitoring program. All the lead results for the filter digestates reported by SGS Australia Pty Ltd as µg/mL Pb on the latest Analytical Report revision agree with the raw data examined. The µg Pb/filter results reported by SGS Australia Pty Ltd on the latest Analytical Report revision were examined and calculations checked and found to be correct. The µg Pb/m^3 in the shipping container air results reported by SGS Australia Pty Ltd on the latest Analytical Report revision were examined and calculations checked and found to be correct. No lead in container air sample exceeded the baseline level of 20 µg/m^3. Original reported results that indicated exceedances were caused by samples being logged into the laboratory's information management system as high volume filters and subsequently the µg Pb/m^3 results were incorrectly calculated. These errors should have been identified and corrected after the first reported exceedance in November 2009.

Inadequacies were found in completeness of the Chain of Custody documents recorded by Inspectorate Australia (Assay) Pty Ltd. The method used by Inspectorate for determining air sampler flow rates should be updated to ensure best practice procedures are used.

The analytical methods used by SGS Australia Pty Ltd are sensitive enough to detect 20 µg Pb/m^3 in the shipping container air. Method validation and the estimate of measurement uncertainty for lead in air filters was poorly calculated and documented. SGS Australia Pty Ltd are currently reviewing and updating the validation and calculation of the estimate of uncertainty. Record keeping especially related to the air filter sample preparation must be improved so that records provide traceability.

Quality control samples available to the laboratory relevant to Magellan Metal mixed cellulose ester air filter samples are limited. The laboratory is using a quality control program but that program is more relevant to high volume filters and environmental waters. It is recommended that Inspectorate collect more duplicate samples and the results used as an ongoing performance indicator. It is recommended that a certified reference material (CRM) quality control program be established and administered independent of SGS Australia Pty Ltd as a check of laboratory lead in filter results.

3 BACKGROUND TO THE INDEPENDENT INVESTIGATION

Magellan Metals Pty Ltd operates a lead carbonate project located at Wiluna in Western Australia. As part of the environmental approvals for the project, Magellan is required to monitor and report on air quality, from inside containers in which bagged lead carbonate concentrate is transported from the Wiluna minesite to Fremantle Port. An independent group, Inspectorate Australia, is responsible for random selection of containers for monitoring, and for deployment and recovery of the sampling devices in those containers. Monitoring is conducted by Inspectorate Australia using modified personal particulate monitors (sampling pumps and filters), which are placed inside approximately 1% of all containers following loading with bagged lead carbonate concentrate and prior to the containers being bolt locked and loaded for transport. The monitors are retrieved at Fremantle Port, and sample filters are sent for laboratory analysis for lead.

On 15 December 2010, Magellan provided container monitoring data to the Office of the Environmental Protection Authority (OEPA) which suggested that on two occasions the airborne lead concentrations in the sealed containers transporting lead carbonate exceeded the baseline level of 20µg/m^3. On 31 December 2010, a Stop Order was issued by the Minister for Environment to Magellan Metals Pty Ltd to cease transport of its product from the Wiluna minesite, and a further Order was issued on 3 January also requiring Magellan Metals Pty Ltd to cease transporting lead carbonate from the minesite until Magellan Metals Pty Ltd had reported on an investigation to the satisfaction of OEPA.

SGS Australia Pty Ltd (SGS), which conducted the original analyses indicating exceedances, subsequently reviewed the original work and informed Magellan Metals Pty Ltd that the results were incorrect and that no samples contained lead concentrations which exceeded the baseline value (20µg/m^3). Magellan Metals Pty Ltd then engaged Synergetics Environmental Engineering to review SGS methodology and the reported results, who confirmed that laboratory errors had been made with the original analyses, and that the revised results showing that there were no exceedances above the designated baseline, were in fact correct.

As a result of reporting these revised monitoring results to the OEPA, it was determined that a further independent review was required, to be conducted by a ‘third party expert’ (TPE), into the SGS methodology used to determine the lead levels in the container air monitoring samples, and to confirm, as far as possible, all previously provided results (including the revised results). Laboratory Quality Management Services Pty Ltd was engaged by the OEPA to provide the ‘third party expert’ review.

4 SAMPLING METHODOLOGIES

Sampling of airborne particulate matter was carried out by Inspectorate Australia (Assay) Pty Ltd who trade as Inspectorate Cox using Standard Operating Procedure (SOP -3 Standard Operating Procedure – Container Air Monitoring). This SOP developed by Inspectorate describes the calibration, loading and operation of the sampling equipment as well as the details of the Chain of Custody (COC) and reporting requirements.

Preloaded cassette, mixed cellulose ester (MCE), 0.8µm, 37mm filter assembly is connected via plastic tubing to a battery operated Aircheck sampler Model 224-PCXR4 sampling pump which is operated to draw air through the filter at a nominal 2 L/min. Each of the Aircheck samplers has been modified to incorporate a heavy duty 6-12V DiaMec sealed rechargeable battery which gives the units a typical transit life of 5-7 days on average.

The flow rate of each pump and filter combination is measured using a Defender 510 flow meter at an office before deployment inside the container prior to dispatch from Magellan’s Wiluna Operations. Best practice dictates that pump flow rate should be measured prior to the commencement of sampling, at the sampling site, and again at the completion of sampling when the filter and pump are retrieved. If the before and after pump flow rate results are not within acceptance limits then the flow rate cannot be established and the monitoring run should not be sent to the laboratory for analysis.

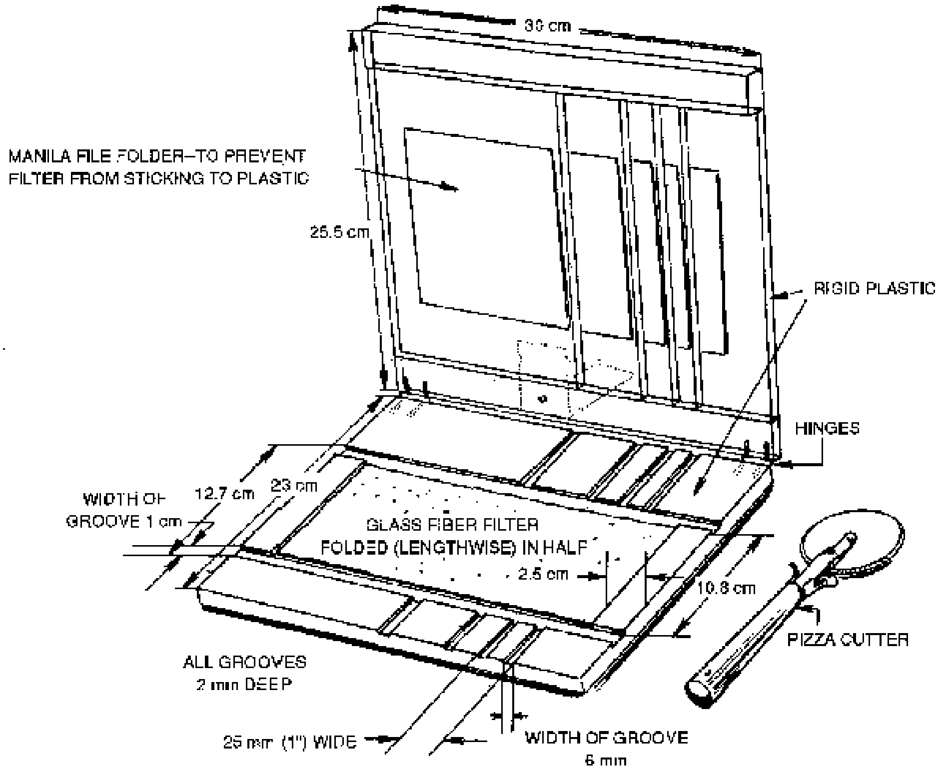
Examination of the chain of custody documentation revealed that on many occasions the chain of custody was incomplete. At times this incomplete information resulted in the monitoring run being invalid as no flow rate or total run time was recorded. All fields of the chain of custody must be completed every time a monitoring run is initiated, anything less is unacceptable.

5 SGS AUSTRALIA PTY LTD ANALYSIS

5.1. Logging Samples into LIMS

Samples logged into the laboratory for analysis prior to the end of June 2010 were logged into the LTech LIMS1 laboratory information management system, the system that was in place at SGS Newburn laboratory. SGS had configured the LTech LIMS1 so that high volume samples and preloaded cassette MCE filter assemblies could not be distinguished and the MCE filter assemblies were logged in as high volume filters.

For high volume filters, the filters are subsampled by cutting a filter strip consisting of one-ninth of the overall filter i.e. a 1" x 8" strip is cut from the 8" x 10" filter using a template (see figure below) and cutting tool. A laboratory microwave extraction system or hot-acid extraction procedure is used to extract the metals with a hydrochloric/nitric acid solution. After cooling, the digestates are mixed and Acrodisc® syringe filters are used to remove any insoluble material. The results, obtained by ICP-OES, ICP-MS, FAA, or GFAA are then multiplied by a factor of 9 to obtain the actual total µg of each metal found on the entire 8" x 10" filter.



Whole MCE filters from preloaded cassette filter assemblies are digested with a hydrochloric/nitric acid solution and the factor of 9 should therefore not be applied to these digest solutions.

From the end of June 2010 samples were logged into SLIMS a CCLAS laboratory information management system. When preloaded cassette MCE filter assemblies where logged into SLIMS the MCE filter assemblies could be distinguished from high volume filters so that the factor of 9 was no longer inadvertently applied to the digest solution.

5.2. Methodologies

5.2.1. Filter Digest Preparation

Prior to the documentation of ME(AU-[ENV]AN042.doc “Acid Digestion of Filter Materials based on USEPA Compendium Method IO-3.1 for Metals” the SGS laboratory had no in-house documented method for the digestion of filters. The laboratory used the USEPA Compendium Method IO-3.1 “Selection, Preparation and Extraction of Filter Material” as the basis for digestion.

The in-house test method ME(AU-[ENV]AN042).doc “Acid Digestion of Filter Materials based on USEPA Compendium Method IO-3.1 for Metals” is technically similar to the USEPA Compendium Method IO-3.1. The USEPA method outlines a microwave and hot acid in beaker extraction technique while the SGS method details a beaker and disposable plastic digestion tube (Digi-Tube) extraction. Acid concentrations of the extraction solution used in both methods are the same, 5.55% HNO_3/16.75%HCl. The USPEA hot acid in beaker extraction method digests samples for 30 minutes ensuring that the samples does not go to dryness while the SGS method digests at 95°C for 60 minutes ensuring that samples do not go to dryness.

When digested samples are made to final volume the SGS digestion paperwork does not indicate how the samples were made to volume, i.e. volumetric flask or in the Digi-Tube digestion tubes or whether the acid matrix was maintained at a final concentration of 3%HNO_3/8%HCl when the final volume was varied from the method. It is recommended that the SGS laboratory keep more detailed records of the final solution preparation to ensure traceability.

5.2.2. Detection of Lead in Filter Digestate Solutions

The lead concentration in filter extracts has been historically determined by SGS using either or both ICP-OES and ICP-MS techniques. The in-house method AN318 “Determination of Elements in Waters by ICP-MS” is the method used when the ICP-MS technique is used and this method references the USEPA SW 846 Method 6020A “Inductively Coupled Plasma Mass Spectrometry (ICP-MS)”. A method for ICP-OES analysis was not provided but information supplied by the Office of the EPA suggests that the USEPA Compendium Method 3.4 “Determination of Metals in Ambient Particulate Matter using Inductively Coupled Plasma (ICP) Spectroscopy” was used.

During the conduct of the audit, the ICP-MS and ICP-OES techniques were reviewed and both techniques used by the SGS Newburn laboratory were found to be appropriate for the analysis of filter digestate solutions. The sensitivity of both the ICP-OES and ICP-MS techniques is sufficient to detect 20 µg Pb/m^3 in the shipping container air.

5.3. ICP-OES & ICP-MS Method Validation

The following key performance characteristics expected to be determined in the validation process are:

- Detection limit
- Accuracy – closeness of the measured value to the true value for the sample. Accuracy can be determined using two approaches:
  - Analysis of certified reference materials (CRM)
  - Recovery of known amounts of analyte spiked into sample matrix
- Bias – the difference between the measured value and the accepted value. The systematic component of the error of the instrument or the test method.
- Precision – random error introduced by using the test method, a general term for the variability between repeated tests.
- Linearity – linearity study verifies that the sample solutions are in a concentration range where analyte response is linearly proportional to concentration
- Concentration range – concentration interval over which acceptable accuracy, linearity, and precision are obtained
- Method ruggedness – stability of the result produced when steps in the method are varied
- Selectivity / Specificity / Matrix Effects

The validation that has been formally collated by SGS for the ICP-MS and ICP-OES techniques does not adequately cover these key performance characteristics.

Below is the data that SGS used to validate the ICP-OES and ICP-MS methods. These methods were validated for a water matrix using an acid matrix of 1% HNO_3.

The validation data is inadequate for a filter digestion matrix and this has been recognised by the SGS laboratory and the laboratory is currently reviewing and updating their method validation.

Lead (ICP-OES)										
Wavelength nm	**182.143**	**220.353**	**182.143**	**220.353**	**182.143**	**220.353**	**182.143**	**220.353**	**182.143**	**220.353**
Expected Concentration µg/L	**Blank**	**Blank**	**1**	**1**	**2**	**2**	**5**	**5**	**20**	**20**
1	2.5	3.0	3.8	2.3	4.6	5.5	7.9	8.0	27.1	21.7
2	1.7	1.0	1.2	2.4	7.2	4.9	10.5	6.7	22.8	21.5
3	2.6	2.0	5.9	1.5	5.5	4.2	5.8	5.6	29.7	21.8
4	2.2	2.2	5.7	-0.6	2.9	2.1	7.4	7.7	25.1	21.6
5	1.8	2.0	7.6	2.1	6.7	4.6	9.6	8.8	23.5	20.9
6	1.1	2.0	-0.4	1.5	5.0	4.1	8.7	8.4	24.2	23.5
7	1.8	2.3	9.9	2.8	4.8	4.1	7.5	6.2	26.9	22.9
8	1.1	2.4	10.6	2.6	4.4	4.4	6.1	6.2	21.3	20.6
9	2.2	3.0	2.6	2.0	5.4	5.3	9.9	6.3	22.0	21.4
10	2.3	2.9	0.9	3.5	4.5	4.0	8.6	6.6	24.4	21.3
Mean	1.918	2.270	4.773	2.010	5.116	4.327	8.230	7.043	24.701	21.708
Std dev	0.520	0.615	3.805	1.106	1.220	0.941	1.559	1.093	2.592	0.864
% RSD	27.1	27.1	79.7	55.0	23.8	21.7	18.9	15.5	10.5	4.0
MDL	1.632	1.932	11.947	3.472	3.829	2.954	4.897	3.433	8.139	2.714
LOR	1.299	1.538	29.869	8.681	9.574	7.386	12.242	8.582	20.348	6.784
Bias	1.918	2.270	3.773	1.010	3.116	2.327	3.230	2.043	4.701	1.708
Recovery			285 BC	-26 BC	256	216	165	141	124	109
Precision	2.088	2.471	5.504	1.535	3.503	2.627	3.745	2.415	5.593	1.997
Uncertainty$_{95\%}$	2.054	2.431	6.572	1.862	3.561	2.680	3.911	2.562	5.962	2.101

BC - blank corrected

Lead (ICP-MS)

Isotope	**206**	**207**	**208**
Expected Concentration µg/L	**0.01**	**0.01**	**0.01**
1	0.021	0.024	0.031
2	0.034	0.054	0.041
3	0.021	0.030	0.030
4	0.023	0.038	0.036
5	0.012	0.038	0.026
6	0.010	0.033	0.027
7	0.017	0.023	0.027
8	0.016	0.032	0.026
9	0.030	0.038	0.044
10	0.009	0.030	0.018
11	0.018	0.054	0.038
12	0.007	0.028	0.020
13	0.024	0.023	0.027
14	0.019	0.027	0.032
Mean	0.019	0.034	0.030
Std dev	0.008	0.010	0.007
% RSD	42.0	29.9	24.8
MDL	0.024	0.032	0.024
LOR	0.061	0.080	0.059
Bias	0.009	0.024	0.020
Recovery	186	339	302
Precision	0.012	0.027	0.022
Uncertainty$_{95\%}$	0.014	0.028	0.023

The Method Detection Limit (MDL) must take into account sample preparation and analytical conditions and must be established for each type of matrix to which the method is going to be applied, i.e. filter digestate solutions that have a final acid concentration 3% HNO_3/8% HCl. The MDL is the concentration of an analyte that produces an instrument response equal to the upper 99% confidence limit for repeated measurements on a sample blank (3% HNO_3/8% HCl), on at least seven (7) replicate measurements. Where a filter digestate solutions does not produce a measurable signal, the standard deviation may be estimated by spiking sample blank solution with a standard solution of the analyte at a low concentration (so that the final solution has an analyte concentration less than 5 times the estimated MDL).

The standard deviation of the results multiplied by the critical value of t (3.143, one-sided t distribution for 7 replicates at 99% confidence or 2.821 for 10 replicates at 99% confidence).

Calculated by $\quad MDL = t \times s$

Where t = taken from a one-sided t distribution for n-1 degrees of freedom
s = standard deviation of analytical result

SGS have used 3.143 (7 replicates) to calculate MDL using 1% HNO_3 rather than 2.821 (10 replicates) using filter digestate solution matrix (3% HNO_3/8% HCl). The Limit of Reporting (LOR) quoted by SGS is 2.5 times the calculated MDL.

As part of the SGS validation of ICP-OES and ICP-MS of lead in filters the following data should have been presented in the validation document

- Method Detection limit in 3% HNO_3/8% HCl matrix
- Accuracy by analysis of certified reference materials (CRM) or matrix spikes
- Precision determined by calculating the percent relative standard deviation (%RSD) of the spiked analyte recoveries for at least the seven (7) replicates at three concentrations over the calibration range (e.g. LOR, mid range, maximum level).
- Concentration range over which acceptable accuracy and precision are obtained
- Selectivity / Specificity / Matrix Effects would have been undertaken to compensate for spectral interferences but the data has not been formalised in a validation report

Although the formalised method validation data is currently inadequate, the SGS laboratory should be able to collate validation data that is fit for purpose with minimal effort. Much of the data required to update the method validation documentation has been undertaken by SGS but not formally collated.

5.4. Measurement Uncertainty Calculation

The measurement uncertainty calculations presented in the tables in Section 5.3 are inadequate and would not adequately estimate the uncertainty of the analytical method.

There are two general strategies proposed for the estimation of measurement uncertainty, an outline of these approaches is given below.

Bottom –up Approach

The ISO approach, supported by IUPAC, EURACHEM as well as other organisations, is to identify all individual components such as uncertainty of level of extraction, uncertainty in the preparation of solutions or weighing of portions of substances etc., then to quantify the contribution of each component and finally to combine all the components to give a total uncertainty budget, the so-called combined standard uncertainty. The uncertainties of the components and the uncertainty of the combined standard uncertainty are stated as standard deviations. The contributions of the type A uncertainties are quantified by statistical well-known procedures. The quantification of the contributions of the type B components may be performed on estimations derived from previous measurement data, from experience with or knowledge of the behaviour and characteristics of the object under investigation and of the measurement technique being used, from information quoted by the manufacturer, from data based on calibration or certificates or from uncertainties quoted from reference data taken from manuals. This non-statistical estimation of uncertainties demands wide knowledge and experience on the relationships. Often it is convenient for the estimation to group several diverse uncertainty factors within one single uncertainty component. To increase confidence the combined standard uncertainty is multiplied with a coverage factor, which gives the so-called expanded uncertainty. A factor of 2 provides a confidence level of approximately 95 %, a factor of 3 a level of approximately 99 %.

(1) Eurochem /CITAC Guide, *Quantifying Uncertainty in Analytical Measurement*, Second Edition, QUAM:2000.P1

Top-down Approach

The **“top-down”** approach aims to get the most reliable estimate of the uncertainty overall, without necessarily identifying the contributions from all of the possible sources. Realistic and useful uncertainty estimates are readily obtained using a laboratory’s existing data which include validation data, instrument calibration data, data obtained from routine quality control procedures as well as from the documented test method. This top-down approach provides all the information necessary to estimate measurement uncertainty compliant with ISO/IEC 17025:2005.

(2) Paul Armishaw, *“Estimating measurement uncertainty in an afternoon. A case study in the practical application of measurement uncertainty”*, Accred. Qual. Assur., 8, 2003, pp218–224

SGS needs to recalculate their estimate of measurement uncertainty for the determination of lead in MCE filters. Much of the data required to do the calculations would be readily available to the laboratory.

5.5. Quality Assurance

Within each batch of samples run on the ICP-OES and the ICP-MS, quality control samples were routinely run. As filter samples were batched with other samples the quality control samples run were often not relevant to Magellan MCE filter samples but these quality control samples did give an overall indication that the analytical process was in control.

Inspectorate did not provide the SGS laboratory with blank filters that could be used as filter lot/batch blank controls. Although MCE filters should contain minimal metal contamination, good laboratory practice dictates that lot/batch filter blanks are undertaken routinely. It is recommended that Inspectorate provide SGS with blank filters for each batch or lot of filters used.

A reagent blank was routinely run with each sample batch, this blank determined the background concentration of lead from the acid digest.

A blank matrix spike was often run within the sample batch, but on reviewing the quality control samples during the conduct of the audit in many cases the expected concentration of the matrix spike could not be determined. More thorough record keeping needs to be undertaken by SGS during the sample preparation stage. This situation of not having a "standard" blank spiking concentration has occurred due to the fact that an in-house method for the preparation of MCE filters was not documented until January 2011.

Sample duplicates are often used as a measure of analytical performance but because the whole MCE filter was digested, sample duplicates could not be used as a routine analytical performance indicator. Duplicate samples were collected from within container MSKU2596229 and reported on SGS Analytical Report numbers PE027948 and PE027949. The µg/mL Pb in the filter digestates for these duplicate samples was 0.17 and 0.15 respectively, which indicates that both the sampling and analytical processes are in control. It is recommended that more duplicate samples are collected and that the results are used as an ongoing performance indicator. It should be noted that sample duplicates were run with each sample batch that contained the Magellan MCE filter digestates but except for the duplicate samples from within container MSKU2596229, the duplicates were not Magellan MCE filter samples.

Matrix spike samples have not been used as a quality control sample when analysing Magellan MCE filter samples. Matrix control samples would require that duplicate samples are collected and that one sample is spiked and the spike recovery calculated. Matrix spike recovery is used to evaluate the impact of matrix effects on the overall analytical performance. Spikes are added prior to the sample being taken through the digestion process, it should be noted that matrix spike recovery do not give any measure of the extraction efficiency.

When reviewing the ICP-OES and ICP-MS results it became clear that SGS routinely reported results that exceed the calibration range. For example, in Analytical Report PE030889 the filter digestate contained 4.3 µg/mL Pb and the top calibration standard was 1 µg/mL Pb. The result was reported although the concentration of the sample exceeded the calibration range. Although this is not good analytical practice, SGS did run a laboratory control sample at 10 µg/mL Pb and that result was deemed acceptable and the filter digestate result was reported. It is recommended that SGS extend the calibration range or dilute samples so that they fell within the calibration range.

An independent check of SGS lead in filter results could be established by purchasing Certified Reference Materials (CRM) from an accredited (ISO Guide 34:2000) reference material producer. These CRMs would be submitted to SGS for analysis. ERA are just one example of an accredited reference material producer and ERA CRMs can be purchased through Graham B Jackson (Aust) P/L, 125 Thomas Street Dandenong, Victoria 3175, contact Andrew Millner, Tel: 03 9793 3322. The CRM quality control program would need to be managed so that these CRMs are unknowns. As the filter will be different from those submitted by Inspectorate, SGS will know that the CRMs are check samples but as long as the expected values are unknown to the laboratory the program will be fit for purpose.

5.6. Review of Calculations

Calculations of airborne lead concentrations in containers reported by SGS have been reviewed. The calculations are listed below:

$$\mu g\ Pb/\ filter = \mu g/mL\ Pb_{Filter\ Digestate} \times Volume_{FilterDigestate}\ (mL)$$

$$\mu g/m^3\ Pb_{\ containerair} = \mu g\ Pb/\ filter\ \times air\ volume_{samplingpump}\ (m^3)$$

where

$$air\ volume_{samplingpump}\ (m^3) = Pump\ flow\ rate\ (m^3/\mathrm{min}) \times Sampling\ pump\ run\ time\ (\mathrm{min})$$

SGS have analysed the MCE filter digestates by either ICP-OES and/or ICP-MS to give the concentration of lead as µg Pb/filter. Inspectorate provide the sampling pump flow rate and the sampling pump run time on the sample Chain of Custody and this data is then used by SGS to report µg Pb/m^3 in the shipping container air.

All the data provided to SGS by Inspectorate on the sample Chain of Custodies (sampling pump flow rate and the sampling pump run time) was checked during the audit. One error was found on SGS report number PE051229 R2 where an air volume of 14m^3 was used instead of 12.19m^3, this error made no difference to the result reported, <1 µg Pb/m^3.

See Appendix 1 for details of the calculations used in the SGS Analytical Reports.

5.7. Review of ICP-OES & ICP-MS Data

During the conduct of the audit all the ICP-OES and ICP-MS results of samples listed on the “Operational Air Monitoring in Container Results – Updated 25 Jan 2011” pdf supplied by the OEPA were reviewed. The review process involved examining all the SGS ICP-OES and ICP-MS data for sample batches in which the filter digestates were run and quantified. All the lead results for the filter digestates reported by SGS as µg/mL Pb on the latest Analytical Report revision agree with the raw data examined.

5.8. Review of Filter Digestate Solution Volumes

During the audit the sample preparation worksheets were inspected to check solution filter digestate volumes. Unfortunately not all the records could be located.

Job No.	Volume used in original Analytical Reports	Volume used in revised Analytical Reports	Volume on original sample preparation worksheets
26623	20	20	Unable to locate worksheets
26955	25	25	Unable to locate worksheets
24431	25	50	50
27870	20		50
27949	20	20	Unable to locate worksheets
27948	20	20	Unable to locate worksheets
28350	25	25	25
30161	25	25	25
30286	25	25	25
30451	25	20	20

Job No.	Volume used in original Analytical Reports	Volume used in revised Analytical Reports	Volume on original sample preparation worksheets
30514	20	20	Unable to locate worksheets
30667	20	20	20
30889	25	20	20
51052	20	20	20
51246	20	20	20
51229	20	20	20
51520	20	20	20
51955	20	20	20
52435	20	20	Unable to locate worksheets
52436	20	20	Unable to locate worksheets
52748	20	20	Unable to locate worksheets
53207	20	20	Unable to locate worksheets
53332	20	20	Unable to locate worksheets
53399	20	20	20
53889	40	40	40
54080	40	40	Unable to locate worksheets
54872	20	20	20
54874	20	20	20

5.9. SGS Reported Results

The SGS Analytical Reports were examined and many reporting errors were found. These errors included;

1. Results reported as µg/mL Pb rather than being reported as µg/L Pb
2. Incorrect filter digestate volumes were used to calculate results
3. As detailed in Section 5.1 of this report, samples were logged into the LIMS system as high volume filters and were accordingly subjected to a “dilution” factor of nine (9)
4. Sampled air volume was incorrectly calculated and reported

The µg Pb/filter results reported by SGS on the latest Analytical Report revision were examined and calculated and found to be correct.

The µg Pb/m^3 in the shipping container air results reported by SGS on the latest Analytical Report revision were examined and calculated and found to be correct.

6 RECOMMENDATIONS

(i) Inspectorate staff must be trained on completing sample Chain of Custody documentation. A checklist may assist with the documentation process but often it does not and is just another document to be completed. If the boxes that MUST be completed were shaded and the Inspectorate staff member signs off at each stage of the documentation that all parts of the Chain of Custody have been completed, this may assist with the document completion process. If a sampling pump fails and the required information cannot be provided on the Chain of Custody then the reasons why the information was not recorded should be documented.

The inspectorate Chain of Custody does not contain the relinquishing of custody information, this should be part of the document. The SGS stamp used when samples are received by the laboratory provides the sample receipt information. The information on the stamp should be incorporated into the Chain of Custody.

(ii) The flow rate of each pump and filter combination is measured using a Defender 510 flow meter at an office before deployment inside the container prior to dispatch from Magellan’s Wiluna Operations. Best practice dictates that pump flow rate should be measured prior to the commencement of sampling, at the sampling site, and again at the completion of sampling when the filter and pump are retrieved. If the before and after pump flow rate results are not within acceptance limits then the flow rate cannot be established and the monitoring run should not be sent to the laboratory for analysis.

(iii) Inspectorate should collect more duplicate lead in container air samples and the results from these duplicate analysis used as an ongoing performance indicator.

(iv) Inspectorate provide SGS with blank filters for each batch or lot of filters used.

(v) SGS laboratory keep more detailed records of the final solution preparation to ensure traceability. This includes a record of how the samples were made to volume, volumetric flask or in the Digi-Tube digestion tubes. Records of the spiking solutions used for the quality control blank matrix spike must also be kept.

(vi) SGS should update ICP-OES & ICP-MS methods to include the analysis of air filter digestates.

(vii) SGS should only report analytical results that are within the calibration range of the ICP-OES & ICP-MS methods, this would be achieved if SGS extended the calibration range or diluted samples so that they fell within the calibration range

(viii) SGS to collate and document method validation for the determination of lead in air filters

(ix) SGS to collate data and calculate an estimate of measurement uncertainty for the determination of lead in air filters

(x) SGS reissue Analytical Report PE51229-R2 and correct the “Air Volume Sampled (m^3)” value

(xi) Establish a CRM quality control program as an independent check of SGS lead in filter results.

7 APPENDIX 1

Sampler No.	COC Number	SGS Analytical Report No.	Air Volume Sampled (m^3)	µg/mL Pb	Digestate Volume (mL)	µg Pb/filter	µg Pb/m^3	SGS Analytical Report No.	µg Pb/m^3
		PE026623	14 (unkn.)*	,0.04	20				
884506	26955	PE26955-R-1	9	0.61	25	140 (0.61x25x9)	15 (140/9)	PE26955-R3	1.7 (0.61x25)/9
884131	27431	PE27431	13	1.8	25 (50)	400 (1.8x25x9)	30 (400/13)	PE27431-R5	6.8 (1.8x50)/9
27870	27870	PE27870	24 (unkn.)*	0.16	20	<10 (0.16x20x9)	1.4 (28.8/24)	PE27870-R2	NT [0.16x50)/unkn.
27949	27949	PE27949	12	0.15	20	<10 (0.15x20)	<1.0 [(0.15x20)/12]	PE27949-R	<1.0 (0.15x20)/12
27948	27948	PE27948	12	0.17	20	<10 (0.17x20)	<1.0 [(0.17x20)/12]	PE27948-R	<1.0 [(0.17x20)/12]
28350	28350	PE28350	19	0.73	25	160 (0.73x25x9)	8.8 (160/19)	PE28350-R2	<1.0 (0.73x25)/19
30161	30161	PE30161	12	0.07	25	16 (0.07x25x9)	1.3 (16/12)	PE30161-R1	<1.0 (0.07x25)/12
30286	30286	PE30286	7	<0.04	25	<10 (<0.04x25x9)	<0.02 [(<0.04x25x9)]/7	PE30286-R	<1.0 (<0.04x25)/7
30451	30451	PE30451	9	0.52	20	120 (0.52x25x9)	13 (120/9) 93.6/9=10.4	PE30451-R1	1.1 (0.52x20)/9
30514	30514	PE30514	8 (unkn.)*	0.55	20	120 (0.55x25x9)	15 (120/8)	PE30514-R3	NT (0.55x20)/unkn.
30667	30667	PE30667	12(unkn.)*	<0.04	20	<10 (<0.04x20x9)	<0.020 [(<0.04x20x9)/12]	PE30667-R1	NT (<0.04x20)/unkn.
30889	30889	PE30889	9	4.3	20	960 (4.3x25x9)	100 [(4.3x25x9)/9]	PE30889-R2	9.3 (4.3x20)/9
51052	51052	PE51052	13	<0.04	20	7 (<0.04x20x9)	0.52 [(<0.04x20x9)/13]	PE51052-R3	<1 (<0.04x20)/13
51246	51246	PE51246	6	<0.04	20	<0.1 (0.002x20)	<0.02 [(0.002x20)/6]	PE51246-R4	<1 (<0.04x20)/6
51229	51229	PE51229	14 (12.2)*	<0.04	20	7.8 (390x20)	0.55 (7.8/14)	PE51229-R2	<1 (<0.04x20)/12.2

Sampler No.	COC Number	SGS Analytical Report No.	Air Volume Sampled (m^3)	µg/mL Pb	Digestate Volume (mL)	µg Pb/filter	µg Pb/m^3	SGS Analytical Report No.	µg Pb/m^3
51520	51520	PE51520	10	1.9	20	38 (1.9x20)	4 (1.9x20)/10	PE51520-R2	4 (1.9x20)/10
51955	51955	PE51955	9	0.27	20	5.4 (0.270x20)	0.60 (0.270x20)/10	PE51955-R2	<1 (0.270x20)/10
52435	52435	PE52435	9	0.22	20	39 (200x20)	4.5 (200x20)/9	PE52435-R3	<1 (0.2x20)/9
52436	52436	PE 52436	9	0.25	20	44 (250x20)	5.2 (250x20)/9	PE 52436-R3	<1 (0.250x20)/9
52748	52748	PE52748	16	0.39	20	69 (390x20)	4.3 (390x20)/16	PE52748-R3	<1 (0.390x20)/16
53207	53207	PE53207	9	0.11	20	2.1 (110x20)	0.23 (110x20)/9	PE53207-R2	<1 (0.11x20)/9
53332	53332	PE53332	13	0.13	20	2.6 (130x20)	0.2 (130x20)/13	PE53332-R2	<1 (0.130x20)/13
53399	53399	PE53399	15	0.17	20	3.3 (0.17x20)	0.22 (0.17x20)/15	PE53399-R2	<1 (0.17x20)/15
53889	53889	PE53889	20	<0.04	40	2.0 (0.049x40)	0.10 (0.049x40)/20	PE53889-R2	<1 (<0.040x40)/20
54080	54080	PE54080	15	<0.04	40	2.5 (62x40)	0.16 (62x40)/15	PE54080-R2	<1 (<0.040x40)/15
54872	54872	PE54872-R0	18	0.24	20	<10 (0.240x20)	<1 (0.240x20)/18	PE54872-R0	<1 (0.240x20)/18
54874	54874	PE54874-R0	15	<0.04	20	<10 (<0.040x20)	<1 (0.240x20)/18	PE54874-R0	<1 (<0.040x20)/15

NT – Not Tested
unkn. – unknown
* - Value in brackets should have been used in the calculation of air volume sampled