APPENDIX C

(Sampling Protocols and QA/QC Definitions)

SOIL AND GROUNDWATER SAMPLING PROTOCOLS

These protocols specify the basic procedures to be used when sampling soils or groundwater for environmental site assessments undertaken by EIS. The purpose of these protocols is to provide standard methods for: sampling, decontamination procedures for sampling equipment, sample preservation, sample storage and sample handling. Deviations from these procedures must be recorded.

Soil Sampling

a) Prepare a test pit/borehole log.

b) Layout sampling equipment on clean plastic sheeting to prevent direct contact with ground surface. The work area should be at a distance from the drill/rig excavator such that the drill rig/excavator can operate in a safe manner.

c) Ensure all sampling equipment has been decontaminated prior to use.

d) Remove any surface debris from the immediate area of the sampling location.

e) Collect samples and place in glass jar with a Teflon seal. This should be undertaken as quickly as possibly to prevent the loss of volatiles. If possible, fill the glass jars completely.

f) Collect samples for asbestos analysis and place in a zip-lock plastic bag.

g) Label the jar and/or bag with the EIS job number, sample location (e.g. BH1), sampling depth interval and date. If more than one sample container is used, this should also be indicated (e.g. 2 = Sample jar 1 of 2 jars).

h) Photoionisation detector (PID) screening of volatile organic compounds (VOCs) should be undertaken on samples using the soil sample headspace method. Headspace measurements are taken following equilibration of the headspace gasses in partly filled zip-lock plastic bags. PID headspace data is recorded on the borehole/test pit log and the chain of custody forms.

i) Record the lithology of the sample and sample depth on the borehole/test pit log in accordance with AS1726-1993[23].

j) Store the sample in a sample container cooled with ice or chill packs. On completion of the sampling the sample container should be delivered to the lab immediately or stored in the refrigerator prior to delivery to the lab. All samples are preserved in accordance with AS 4482.1:2005, AS 4482.2:1999 and AS/NZS 5667.1:1998.

k) Check for the presence of groundwater after completion of each borehole using an electronic dip metre or water whistle. Boreholes should be left open until the end of fieldwork. All groundwater levels in the boreholes should be rechecked on the completion of the fieldwork.

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[23] *Geotechnical Site Investigations*, Standards Australia 1993 (AS1726-1993)

l) Backfill the boreholes/test pits with the excavation cuttings or clean sand prior to leaving the site.

Decontamination Procedures for Soil Sampling Equipment

a) All of the equipment associated with the soil sampling procedure should be decontaminated between every sampling location.

b) The following equipment and materials are required for the decontamination procedure:
   - Phosphate free detergent (Decon 90)
   - Tap water
   - Stiff brushes
   - Plastic sheets

c) Ensure the decontamination materials are clean prior to proceeding with the decontamination.

d) Fill both buckets with clean tap water and add phosphate free detergent to one bucket.

e) In the bucket containing the detergent scrub the sampling equipment until all the material attached to the equipment has been removed.

f) Rinse sampling equipment in the bucket containing tap water.

g) Place cleaned equipment on clean plastic sheets.

If all materials are not removed by this procedure, high-pressure water cleaning is recommended. If any equipment is not completely decontaminated by both these processes that equipment should not be used until it has been thoroughly cleaned.

Groundwater Sampling

Groundwater samples are more sensitive to contamination than soil samples and therefore adhesion to this protocol is particularly important to obtain reliable, reproducible results. The recommendations detailed in AS/NZS 5667.1:1998 are considered to form a minimum standard.

The basis of this protocol is to maintain the security of the borehole and obtain accurate and representative groundwater samples. The following procedure should be used for collection of groundwater samples from previously installed groundwater monitoring wells.

a) After groundwater monitoring wells installation, at least three bore volumes should be pumped from the monitoring wells (well development) to remove any water introduced during the drilling process and/or the water that is disturbed during installation of the groundwater monitoring wells. This should be completed prior to purging and sampling.

b) Groundwater monitoring wells should then be left to recharge for at least three days before purging and sampling. Prior to purging or sampling the condition of each

well should observed and any anomalies recorded on the field data sheets. The following information should be noted: the condition of the well, noting any signs of damage, tampering or complete destruction; the condition and operation of the well lock; the condition of the protective casing and the cement footing (raised or cracked); and, the presence of water between protective casing and well.

c) Take the groundwater level from the collar of the piezometer using an electronic dipmeter. The collar level should be taken (if required) during the site visit using a dumpy level and staff.

d) Purging and sampling of piezometers is done on the same site visit when using micro-purge (or low flow) techniques. Layout and organize all equipment associated with groundwater sampling in a location where they will not interfere with the sampling procedure and will not pose a risk of contaminating samples. Equipment generally required includes:

- Micropore filtration system or Stericup single-use filters (for heavy metals samples).
- Filter paper for Micropore filtration system.
- Bucket with volume increments.
- Sample containers: Teflon bottles with 1 ml nitric acid, 75mL glass vials with 1 mL hydrochloric acid, 1 L amber glass bottles.
- Bucket with volume increments.
- Flow cell.
- pH/EC/Eh/T meters.
- Plastic drums used for transportation of purged water.
- Esky and ice.
- Nitrile gloves.
- Distilled water (for cleaning).
- Electronic dip meter.
- Micro-purge pump pack and pump head.
- Air and water tubing for Micro-purge.
- Groundwater sampling forms.

e) If single-use stericup filtration is not being used, clean the Micropore filtration system thoroughly with distilled water prior to use and between each sample. Filter paper should be changed between samples. 0.45um filter paper should be placed below the glass fibre filter paper in the filtration system.

f) Ensure all non-disposable sampling equipment is decontaminated or that new disposable equipment is available prior to any work commencing at a new location. The procedure for decontamination of groundwater equipment is outlined at the end of this section.

g) Disposable gloves should be used whenever samples are taken to protect the sampler and to assist in avoidance of contamination.

h) Groundwater samples are obtained from the monitoring wells using low flow/micro-purge sampling equipment to reduce the disturbance of the water column and loss of volatiles.

i) During pumping to purge the well, the pH, temperature, conductivity, dissolved oxygen, redox potential and groundwater levels are monitored (where possible) using calibrated field instruments to assess the development of steady state conditions. Steady state conditions are generally considered to have been achieved when the difference in the pH measurements was less than 0.2 units and the difference in conductivity was less than 10%.

j) All measurements are recorded on specific data sheets.

k) Once steady state conditions are considered to have been achieved, groundwater samples are obtained directly from the pump tubing and placed in appropriate glass bottles, BTEX vials or plastic bottles.

l) All samples are preserved in accordance with water sampling requirements detailed in the NEPM Guidelines (1999) and placed in an insulated container with ice. Groundwater samples are preserved by immediate storage in an insulated sample container with ice in accordance with AS/NZS 5667.1-1 998.

m) Record the sample on the appropriate log in accordance with AS 1726-1 993. At the end of each water sampling complete a chain of custody form.

Decontamination Procedures for Groundwater Sampling Equipment

a) All of the equipment associated with the groundwater sampling procedure (other than single-use items) should be decontaminated between every sampling location.

b) The following equipment and materials are required for the decontamination procedure:
   - Phosphate free detergent.
   - Tap water.
   - Distilled water
   - Plastic Sheets or bulk bags (plastic bags)

c) Fill one bucket with clean tap water and phosphate free detergent, and one bucket with distilled water.

d) Flush tap water and detergent through pump head. Wash sampling equipment and pump head using brushes in the bucket containing detergent until all materials attached to the equipment are removed.

e) Flush pump head with distilled water.

f) Change water and detergent solution after each sampling location.

g) Rinse sampling equipment in the bucket containing distilled water.

h) Place cleaned equipment on clean plastic sheets.

i) If all materials are not removed by this procedure that equipment should not be used until it has been thoroughly cleaned

QA/QC DEFINITIONS

The QA/QC terms used in this report are defined below. The definitions are in accordance with US EPA publication SW-846, entitled *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (1994[24]) methods and those described in *Environmental Sampling and Analysis, A Practical Guide,* (H. Keith 1991[25]).

Practical Quantitation Limit (PQL), Limit of Reporting (LOR) and Estimated Quantitation Limit (EQL)

These terms all refer to the concentration above which results can be expressed with a minimum 95% confidence level. The laboratory reporting limits are generally set at ten times the standard deviation for the Method Detection limit (MDL) for each specific analyte. For the purposes of this report the LOR, PQL, and EQL are considered to be equivalent.

When assessing laboratory data it should be borne in mind that values at or near the PQL have two important limitations. *"The uncertainty of the measurement value can approach, and even equal, the reported value. Secondly, confirmation of the analytes reported is virtually impossible unless identification uses highly selective methods. These issues diminish when reliably measurable amounts of analytes are present. Accordingly, legal and regulatory actions should be limited to data at or above the reliable detection limit"*, Keith (1 991).

Precision

The degree to which data generated from repeated measurements differ from one another due to random errors. Precision is measured using the standard deviation or Relative Percent Difference (RPD). Acceptable targets for precision in this report will be less than 50% RPD for concentrations greater than ten times the PQL, less than 75% RPD for concentrations between five and ten times the PQL and less than 100% RPD for concentrations that are less than five times the PQL.

Accuracy

Accuracy is a measure of the agreement between an experimental result and the true value of the parameter being measured. The assessment of accuracy for an analysis can be achieved through the analysis of known reference materials or assessed by the analysis of surrogates, field blanks, trip spikes and matrix spikes.

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[24] *SW-846: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* US EPA, 1994 (US EPA SW-846)

[25] *Environmental Sampling and Analysis, A Practical Guide*, Keith, H, 1991 (Keith 1991)

The proximity of an averaged result to the true value, where all random errors have been statistically removed. Accuracy is measured by percent recovery. Acceptable limits for accuracy generally lie between 70% to 130% recoveries. Certain laboratory methods may allow for values that lie outside these limits.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is primarily dependent upon the design and implementation of the sampling program. Representativeness of the data is partially ensured by the avoidance of contamination, adherence to sample handing and analysis protocols and use of proper chain-of-custody and documentation procedures.

Completeness

Completeness is a measure of the number of valid measurements in a data set compared to the total number of measurements made and overall performance against DQIs. The following information is assessed for completeness:

- Chain-of-custody forms;
- Sample receipt form;
- All sample results reported;
- All blank data reported;
- All laboratory duplicate and RPDs calculated;
- All surrogate spike data reported;
- All matrix spike and lab control spike (LCS) data reported and RPDs calculated;
- Spike recovery acceptable limits reported; and
- NATA stamp on reports.

Comparability

Comparability is the evaluation of the similarity of conditions (e.g. sample depth, sample homogeneity) under which separate sets of data are produced. Data comparability checks include a bias assessment that may arise from the following sources:

- Collection and analysis of samples by different personnel;
- Use of different techniques;
- Collection and analysis by the same personnel using the same methods but at different times; and
- Spatial and temporal changes (due to environmental dynamics).

Blanks

The purpose of laboratory and field blanks is to check for artifacts and interferences that may arise during sampling and analysis.

Matrix Spikes

Samples are spiked with laboratory grade standards to detect interactive effects between the sample matrix and the analytes being measured. Matrix Spikes are reported as a percent recovery and are prepared for 1 in every 20 samples. Sample batches that contain less than 20 samples may be reported with a Matrix Spike from another batch. The percent recovery is calculated using the formula;

$$\frac{\text{(Spike Sample Result – Sample Result)}}{\text{Concentration of Spike Added}} \times 100$$

Acceptable recovery limits are 70% to 130%.

Surrogate Spikes

Samples are spiked with a known concentration of compounds that are chemically related to the analyte being investigated but unlikely to be detected in the environment. The purpose of the Surrogate Spikes is to check the accuracy of the analytical technique. Surrogate Spikes are reported as percent recovery.

Duplicates

Laboratory duplicates measure precision, expressed as Relative Percent Difference. Duplicates are prepared from a single field sample and analysed as two separate extraction procedures in the laboratory. The RPD is calculated using the formula where D1 is the sample concentration and D2 is the duplicate sample concentration:

$$\frac{(D1 - D2)}{\{(D1 + D2)/2\}} \times 100$$

APPENDIX D

(ProUCL statistical calculations)

General UCL Statistics for Full Data Sets

User Selected Options

From File	WorkSheet.wst
Full Precision	OFF
Confidence Coefficient	95%
Number of Bootstrap Operations	2000

Total PAHs

General Statistics

Number of Valid Observations	13	Number of Distinct Observations	12
Number of Missing Values	4		

Raw Statistics		Log-transformed Statistics	
Minimum	0.06	Minimum of Log Data	-2.813
Maximum	216.8	Maximum of Log Data	5.379
Mean	24.62	Mean of log Data	0.824
Median	1.3	SD of log Data	2.331
SD	60.68		
Std. Error of Mean	16.83		
Coefficient of Variation	2.465		
Skewness	3.114		

Relevant UCL Statistics

Normal Distribution Test		Lognormal Distribution Test	
Shapiro Wilk Test Statistic	0.47	Shapiro Wilk Test Statistic	0.961
Shapiro Wilk Critical Value	0.866	Shapiro Wilk Critical Value	0.866
Data not Normal at 5% Significance Level		Data appear Lognormal at 5% Significance Level	

Assuming Normal Distribution		Assuming Lognormal Distribution	
95% Student's-t UCL	54.62	95% H-UCL	1418
95% UCLs (Adjusted for Skewness)		95% Chebyshev (MVUE) UCL	81.98
95% Adjusted-CLT UCL (Chen-1995)	67.84	97.5% Chebyshev (MVUE) UCL	108.7
95% Modified-t UCL (Johnson-1978)	57.04	99% Chebyshev (MVUE) UCL	161.2

Gamma Distribution Test		Data Distribution	
k star (bias corrected)	0.275	Data Follow Appr. Gamma Distribution at 5% Significance Level	
Theta Star	89.45		
MLE of Mean	24.62		
MLE of Standard Deviation	46.93		
nu star	7.157		
Approximate Chi Square Value (.05)	2.257	**Nonparametric Statistics**	
Adjusted Level of Significance	0.0301	95% CLT UCL	52.31
Adjusted Chi Square Value	1.886	95% Jackknife UCL	54.62
		95% Standard Bootstrap UCL	50.64
Anderson-Darling Test Statistic	1.034	95% Bootstrap-t UCL	366.2
Anderson-Darling 5% Critical Value	0.833	95% Hall's Bootstrap UCL	214
Kolmogorov-Smirnov Test Statistic	0.249	95% Percentile Bootstrap UCL	55.95
Kolmogorov-Smirnov 5% Critical Value	0.257	95% BCA Bootstrap UCL	73.77
Data follow Appr. Gamma Distribution at 5% Significance Level		95% Chebyshev(Mean, Sd) UCL	97.99

	97.5% Chebyshev(Mean, Sd) UCL 129.7
Assuming Gamma Distribution	99% Chebyshev(Mean, Sd) UCL 192.1
95% Approximate Gamma UCL 78.07	
95% Adjusted Gamma UCL 93.45	
Potential UCL to Use	Use 95% Adjusted Gamma UCL 93.45

Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL. These recommendations are based upon the results of the simulation studies summarized in Singh, Singh, and Iaci (2002) and Singh and Singh (2003). For additional insight, the user may want to consult a statistician.

BaP

General Statistics

Number of Valid Observations	13	Number of Distinct Observations	9
Number of Missing Values	4		

Raw Statistics		Log-transformed Statistics	
Minimum	0.06	Minimum of Log Data	-2.813
Maximum	17	Maximum of Log Data	2.833
Mean	2.269	Mean of log Data	-1.104
Median	0.1	SD of log Data	1.888
SD	5.06		
Std. Error of Mean	1.404		
Coefficient of Variation	2.23		
Skewness	2.603		

Relevant UCL Statistics

Normal Distribution Test		Lognormal Distribution Test	
Shapiro Wilk Test Statistic	0.51	Shapiro Wilk Test Statistic	0.807
Shapiro Wilk Critical Value	0.866	Shapiro Wilk Critical Value	0.866
Data not Normal at 5% Significance Level		Data not Lognormal at 5% Significance Level	
Assuming Normal Distribution		Assuming Lognormal Distribution	
95% Student's-t UCL	4.771	95% H-UCL	23.33
95% UCLs (Adjusted for Skewness)		95% Chebyshev (MVUE) UCL	5.216
95% Adjusted-CLT UCL (Chen-1995)	5.66	97.5% Chebyshev (MVUE) UCL	6.835
95% Modified-t UCL (Johnson-1978)	4.94	99% Chebyshev (MVUE) UCL	10.02
Gamma Distribution Test		Data Distribution	
k star (bias corrected)	0.32	Data do not follow a Discernable Distribution (0.05)	
Theta Star	7.086		
MLE of Mean	2.269		
MLE of Standard Deviation	4.01		
nu star	8.327		
Approximate Chi Square Value (.05)	2.926	Nonparametric Statistics	
Adjusted Level of Significance	0.0301	95% CLT UCL	4.578
Adjusted Chi Square Value	2.489	95% Jackknife UCL	4.771
		95% Standard Bootstrap UCL	4.471

Anderson-Darling Test Statistic	1.718	95% Bootstrap-t UCL	32.16
Anderson-Darling 5% Critical Value	0.82	95% Hall's Bootstrap UCL	22.96
Kolmogorov-Smirnov Test Statistic	0.286	95% Percentile Bootstrap UCL	4.817
Kolmogorov-Smirnov 5% Critical Value	0.255	95% BCA Bootstrap UCL	5.52
Data not Gamma Distributed at 5% Significance Level		95% Chebyshev(Mean, Sd) UCL	8.387
		97.5% Chebyshev(Mean, Sd) UCL	11.03
Assuming Gamma Distribution		99% Chebyshev(Mean, Sd) UCL	16.23
95% Approximate Gamma UCL	6.458		
95% Adjusted Gamma UCL	7.593		
Potential UCL to Use		Use 99% Chebyshev (Mean, Sd) UCL	16.23

Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL. These recommendations are based upon the results of the simulation studies summarized in Singh, Singh, and Iaci (2002) and Singh and Singh (2003). For additional insight, the user may want to consult a statistician.

General UCL Statistics for Full Data Sets

User Selected Options

From File	WorkSheet.wst
Full Precision	OFF
Confidence Coefficient	95%
Number of Bootstrap Operations	2000

Lead

General Statistics

Number of Valid Observations	22	Number of Distinct Observations	20

Raw Statistics

Minimum	30
Maximum	450
Mean	170.3
Median	120
SD	128.3
Std. Error of Mean	27.35
Coefficient of Variation	0.753
Skewness	0.754

Log-transformed Statistics

Minimum of Log Data	3.401
Maximum of Log Data	6.109
Mean of log Data	4.836
SD of log Data	0.824

Relevant UCL Statistics

Normal Distribution Test

Shapiro Wilk Test Statistic	0.872
Shapiro Wilk Critical Value	0.911

Data not Normal at 5% Significance Level

Lognormal Distribution Test

Shapiro Wilk Test Statistic	0.945
Shapiro Wilk Critical Value	0.911

Data appear Lognormal at 5% Significance Level

Assuming Normal Distribution

95% Student's-t UCL	217.3

95% UCLs (Adjusted for Skewness)

95% Adjusted-CLT UCL (Chen-1995)	220
95% Modified-t UCL (Johnson-1978)	218.1

Gamma Distribution Test

k star (bias corrected)	1.592
Theta Star	106.9
MLE of Mean	170.3
MLE of Standard Deviation	134.9
nu star	70.06
Approximate Chi Square Value (.05)	51.79
Adjusted Level of Significance	0.0386
Adjusted Chi Square Value	50.62
Anderson-Darling Test Statistic	0.605
Anderson-Darling 5% Critical Value	0.757
Kolmogorov-Smirnov Test Statistic	0.152
Kolmogorov-Smirnov 5% Critical Value	0.188

Data appear Gamma Distributed at 5% Significance Level

Assuming Lognormal Distribution

95% H-UCL	269.9
95% Chebyshev (MVUE) UCL	318.5
97.5% Chebyshev (MVUE) UCL	381.4
99% Chebyshev (MVUE) UCL	504.7

Data Distribution

Data appear Gamma Distributed at 5% Significance Level

Nonparametric Statistics

95% CLT UCL	215.3
95% Jackknife UCL	217.3
95% Standard Bootstrap UCL	213.9
95% Bootstrap-t UCL	225.3
95% Hall's Bootstrap UCL	215.9
95% Percentile Bootstrap UCL	216.1
95% BCA Bootstrap UCL	218.5
95% Chebyshev(Mean, Sd) UCL	289.5
97.5% Chebyshev(Mean, Sd) UCL	341.1

Assuming Gamma Distribution		99% Chebyshev(Mean, Sd) UCL	442.4
95% Approximate Gamma UCL	230.3		
95% Adjusted Gamma UCL	235.7		
Potential UCL to Use		Use 95% Approximate Gamma UCL	230.3

Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL. These recommendations are based upon the results of the simulation studies summarized in Singh, Singh, and Iaci (2002) and Singh and Singh (2003). For additional insight, the user may want to consult a statistician.