

## **ELIG Submission to NEPC re NEPM Schedule B(3).**

The Environmental Laboratory Industry Group (ELIG) was formed in 2002 and represents commercially and regulatory focused laboratories holding NATA Accreditation. Current members consist of the following laboratories:

- 1) **Advanced Analytical Australia** (attila.totszer@advancedanalytical.com.au)
  - 2) **Agrisearch Analytical** (ross\_shields@agrisearchanalytical.com.au)
  - 3) **ALS** (marc.centner@alsenviro.com)
  - 4) **Amdel** (ryan\_hamilton@amdell.com)
  - 5) **Envirolab Services** (tnotaras@envirolabservices.com.au)
  - 6) **Hill Laboratories NZ** (terry.cooney@hill-labs.co.nz)
  - 7) **Labmark** (ivan.povolny@labmark.com.au)
  - 8) **MGT** (sefton@mgtenv.com.au)
  - 9) **National Measurement Institute** (daniel.slee@measurement.gov.au)
  - 10) **SGS** (james\_mcmahon@sgs.com)
  - 11) **Sydney Analytical Laboratories** (Sydney\_analytical@bigpond.com)
- NATA is an observing, non voting member only, (mark.worrell@nata.asn.au)

ELIG represents all the major Environmental Laboratories in Australia & NZ and it is estimated that ELIG labs analyse over 90% of all the samples from contaminated sites in Australia. As such ELIG should be considered a major stakeholder in the NEPM.

It is proposed that ELIG be officially recognised in the NEPM as a professional body of laboratories that analyse over 90% of Australia's contaminated sites and that ELIG is eminently positioned to give advice on how to test for specific analytes in practice, not just theory.

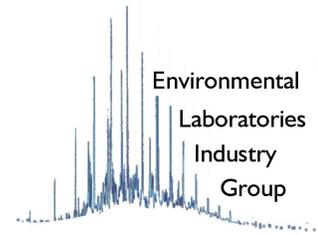
The following submission to NEPC is based on sound scientific principals and best practices and has been endorsed by all 11 ELIG members.

Please feel free to contact any members, or the office bearers below for further details.

Kind Regards,

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### **Section 1, Page 1**

#### **NEPM:**

Expertise in the analysis of contaminated soil is still in the developmental stage in Australia.

#### **ELIG Suggestion:**

This statement is no longer true and should be deleted. Australian laboratories are now as advanced as those in any country.

### **Section 4, Page 13**

#### **ELIG Suggestion:**

There needs to be a statement about the cooling of samples. Various guidelines recommend that samples be received at the laboratory at 4 degrees. In reality, few samples are actually received in any laboratory at 4 degrees. The attached ELIG study (appendix 1) shows that when sand or water is placed into an esky with either ice or ice bricks the cooling rate is very slow.

#### **Sand**

It took sand samples 2 hours in an esky with ice to reach 4 degrees.

It took sand samples 4 hours in an esky with ice bricks to reach 7 degrees. Samples will never reach 4 degrees with ice bricks.

#### **Water**

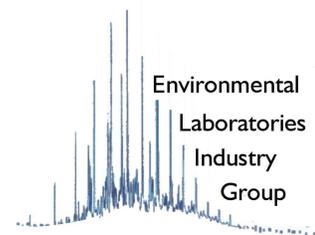
It took water samples 5 hours in an esky with ice to reach 4 degrees.

It took water samples 6 hours in an esky with ice bricks to reach 12 degrees. Samples will never reach 4 degrees with ice bricks.

ELIG believes that it is important that samples are sent to a laboratory as soon as possible after collection, with either ice or ice bricks as the cooling medium.

Laboratories will then measure temperatures on receipt. If consultants have used an esky and ice or ice bricks then the samples are on a cooling cycle – it is irrelevant if they are actually at 4 degrees, at least they are on a cool down cycle. If samples are actually received at 4 degrees then it is likely that the consultant has held onto the samples for an extended time period.

The suggested statement for NEPM is; ‘samples should be paced into eskies with either ice or ice bricks and transported to the laboratory as soon as practical. On receipt, laboratories will record the temperature. An acceptable receipt temperature is one that is less than the temperature at the time of sampling’.



### **Section 3.11.3, Page 10**

#### **NEPM:**

‘Preferred techniques, which usually incorporate mass-selective detection.....’

#### **ELIG Suggestion:**

The concept of preferred techniques should be completely abolished since other detectors such as ECD, FPD, PID and ELCD may not only be more sensitive but often are also highly selective. It is suggested that this section be re written to be more ‘performance based’ rather than prescribed. ELIG can assist with this task if requested.

### **Section 4.2, Page 14**

#### **NEPM:**

Process Batch should be defined.

#### **ELIG Suggestion:**

A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit for QC purposes, if the number of samples in a group is > 20, then each group of 20 samples or less will all be handled as a separate batch.

### **Section 4.2.4, Page 15**

#### **NEPM:**

‘When recovery of matrix spikes fails, it may be necessary to use other internal calibration methods, or to modify the analytical method...’

#### **ELIG Suggestion:**

To use other internal calibration methods is odd and requires removal. If the method is validated, and QC is used to measure method quality, the method should not be changed to satisfy QC. Suggest a change to ‘The failure of a spike recovery must be investigated and a decision to accept or reject the data must be made using the professional judgment of the senior chemist’.

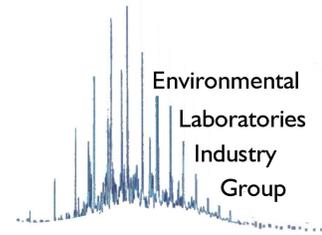
### **Section 4.2.4, Page 15**

#### **NEPM:**

‘matrix spikes should be added at a concentration corresponding to regulatory level’

#### **ELIG Suggestion:**

Spiking where Measurement Uncertainty is greatest is not sensible. This should be changed to ‘a level that is appropriate for the matrix’.



**Section 4.3.1, Page 17**

**NEPM:**

15% is too tight for reference methods.

**ELIG Suggestion:**

Any proficiency study such as NATA or NARL would indicate variations much higher. It has been suggested that if this was the case then >50% of QC samples will fail. Variability of results is often dependant on the analyte tested. Pentachlorophenol is an example of an analyte that could be accepted at >50%.

It is suggested these limits be revised to 'analyte specific as detailed in the individual laboratory manual'.

**Section 4.3.3, Page 19**

**NEPM:**

+/- 15% recovery for spike recovery is over zealous and not always practical in a commercial lab.

**ELIG Suggestion:**

Change this to ' +/- 30% or within the acceptable range as specified in the individual laboratory manual'

**Section 4.3.3, Page 19**

**NEPM:**

'The longer the residence time of the spiked analyte before extraction.....'

**ELIG Suggestion:**

This statement is not correct, especially for volatiles and needs to be removed or amended.

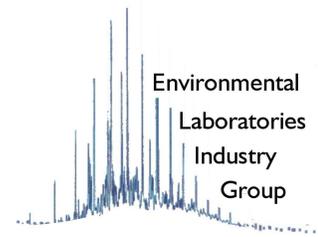
**Section 4.3.4.2, Page 20**

**NEPM:**

PQL

**ELIG Suggestion:**

NEPM suggests that PQL is the only accepted acronym for detection limits. This needs to be changed to include others such as PQL, LOR, EQL or any other recognized term.



#### **Section 4.6, Page 21**

##### **NEPM:**

'accompanied by a statement of uncertainty...'

##### **ELIG Suggestion:**

ISO 17025 only requires the reporting of uncertainty when requested by the client. This section needs removing or amending. ELIG's official line is to discourage clients from asking for MU data (see point below).

#### **Section 6, Page 21**

There needs to be a general section in the NEPM about MU (uncertainty).

##### **ELIG Suggestion:**

MU is an important aspect of results, it is a powerful tool but it does have limitations and should not be used to judge a laboratory. The greatest MU comes not from labs who are doing very precise analysis but from the consultant who is sampling in a stockpile for example.

ELIG's experience so far is that MU is confusing to clients and ELIG is recommending to clients to not ask for MU data.

If MU is important to consultants then they need to be able to estimate MU from their sampling techniques and add this figure to the laboratory MU.

ELIG is willing to provide further guidance and recommendations on MU if requested.

#### **Section 5.1.2, Page 25**

##### **NEPM:**

'The sub sample should be 50% or 200g...'

##### **ELIG Suggestion:**

This is too much to take and is not practical. Consultants do not supply enough sample to laboratories to do this. This should be changed to 25% or 100g.

#### **Section 5.1.4, Page 26**

##### **NEPM:**

The comment on air drying and its affect on forms of Fe and Mn is irrelevant.

##### **ELIG Suggestion:**

As we are dealing with a strong acid digest of soil matter, therefore the forms are generally irrelevant.

The NEPM statements and references seem to be relevant to bioavailability studies and methods of indicating metal bioavailability such as the use of metal complexing agents. Drying at 60 degrees should not affect the oxidation state of Fe and Mn. A soil has already been exposed to the sun and air and reached an equilibrium state. This statement requires removal.

### **Section 5A, Page 29**

#### **NEPM:**

1. Statement to solvent rinse jars.
2. note 'd' at the footer table.
3. The holding time for chloride and sulfate is too strict.
4. Fluoride should be collected in plastic.
5. Metals should be collected in plastic.
6. Extra holding time information.
7. The holding time for moisture is too strict.

#### **ELIG Suggestion:**

1. This is time consuming, costly and environmentally unfriendly and may introduce contamination. It also doesn't make sense to solvent rinse jars that will eventually be tested for solvents. All labs are buying food grade jars so this statement needs removing.
2. The 'D' needs to be in superscript for sulfide.
3. Change these holding times to at least 28 days. They are not going anywhere.
4. Collecting a separate sample in plastic is not practical and we believe consultants will not do this. Remove this as Fluoride leaching from glass is expected to be insignificant.
5. Collecting a separate sample in plastic is not practical and we believe consultants will not do this. Remove this. See attached experiment (appendix 2) that shows there is minimal leaching of metals from glass. ELIG has found that a glass jar is suitable for all analytes. Unless a soil is extremely acidic it will not leach metals from non porous glass.
6. There should be an extra column for extending holding times eg USEPA have done studies on freezing OC's and PAH's and extending holding times for hundreds of days. This would assist laboratories and consultants in remote areas.
7. Extend moisture holding time to at least 14 days, in line with volatiles.

### **Section 5.1.4.3, Page 27**

#### **NEPM:**

'at least pass a 2mm aperture sieve....'

#### **ELIG Suggestion:**

Different sieve sizes may be used – depending on the sample size the chemist will take for analysis. The guide below needs inclusion in the document.

A normal synthetic window screen is suitable for sieving for metals.

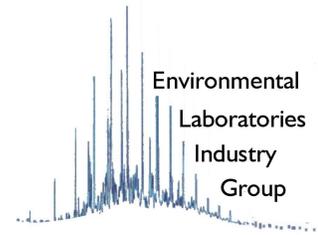
The following table has been used by Dr Honway Louie of NMI to give reproducible results:

2mm sieve = >2g sample weight for analysis

1mm sieve = 1-2 g sample weight

0.5mm sieve = 0.5-1g sample weight

0.210mm sieve = 0.1-0.5g sample weight



### **Section 5.3.3.1, Page 33**

#### **NEPM:**

- a) 'two or more pre weighed 40ml glass vials....'
- b) Note 1: 'The 40ml VOA vials....'
- c) Note 2: 'field immersion into methanol....'

#### **ELIG Suggestion:**

- a) Sampling soils into 40ml VOC vials should be strongly discouraged, unless the whole sample is placed on the Purge & Trap – which rarely occurs in Australia. Trying to squeeze a soil sample in the field into a 40ml vial will result in the loss of volatiles. ELIG suggests that soil samples be collected into 125ml or 250ml glass jars and sub sampled for analysis under laboratory conditions.
- b) Closed system P&T are acceptable for sands but not suitable for clays (much of Eastern Australia). Clay samples must be completely disintegrated before purging can commence.
- c) Methanol has OH&S issues and should only be used in the laboratory.

In summary, ELIG recommends that soil samples be collected in glass jars and transported to the laboratory for analysis.

### **Section 5.3.3, Page 32**

#### **NEPM:**

'samples taken for volatile compound analysis be separate from those for semi-volatile....'

#### **ELIG Suggestion**

This is not required and should be removed. As soon as one removes a sub sample from a jar, a head space is created regardless whether the jar was intended for VOC or any other analysis. One glass jar is suitable for all tests.

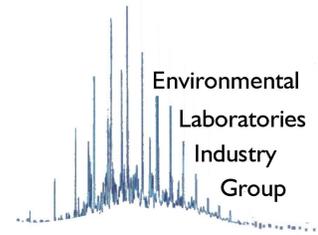
### **Section 5.3.3.1, Page 33**

#### **NEPM:**

NEPM infers that only 125ml jars can be used.

#### **ELIG Suggestion**

Add in the use of 250ml jars also.



### **Section 6 onwards, Page 37 onwards**

#### **NEPM:**

Methods section.

#### **ELIG Suggestion**

These 'methods' are quite vague and seem to be oversimplified 'cut & pastes' from SW846. 'Method 101 – TCLP's' could hardly be called a method – it is just a brief outline.

During our review it has been noted that many of the techniques listed are now out dated and irrelevant – this has occurred in the 5 years since the document was issued and we have every reason to believe that by the next review in 2010 the same will apply. ELIG suggests they be removed and a statement that 'ISO-17025 accredited methods are used supported by performance based proficiency' be inserted. Alternatively, ELIG will offer to re write Section 6 for NEPC if requested.

### **Section 9.5, Page 45**

#### **NEPM:**

EC 'method' – keep the temperature at 25 degrees +/- 0.1 degree.

#### **ELIG Suggestion**

+/- 0.1 degree is not a practical tolerance, this is unnecessarily tight. There would be no problem at +/- 1-2 degree.

This needs to be amended.

### **Section 23.2 , Page 82**

#### **NEPM:**

Volatile Organics 'method'

#### **ELIG Suggestion**

Target analytes are mixed up with TPH – there needs to be 2 methods.

Also, some compounds are questionable eg CS<sub>2</sub> is not recommended to be done by FID due to low response.

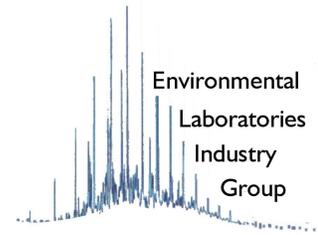
### **Section 23.3.3, Page 85**

#### **NEPM:**

Method 8015B

#### **ELIG Suggestion**

USEPA 8015B should not be applied to target analysis.



### **Section 24, Page 86**

#### **NEPM:**

PAH 'method' – many errors.

#### **ELIG Suggestion**

Re write this eg: why different solvent selections, silica gel should be optional as not needed in 99% of samples, PAH by ECD is not correct.

### **Section 26, Page 91**

#### **NEPM:**

OC 'method' – needs re write

#### **ELIG Suggestion**

Chlordane has major isomers.

Has toxaphene ever been used in Australia?

MSD is marked 'preferred' for OC's. ECD should also be 'preferred'.

### **Section 27, Page 93**

#### **NEPM:**

OP 'method' – needs re write

#### **ELIG Suggestion**

Hexane is not the best solvent for many OP's.

Why specifically list carbophenothion as a low recovery analyte. What about others? Is this true for most labs? Where is the data for this?

### **Section 28, Page 95**

#### **NEPM:**

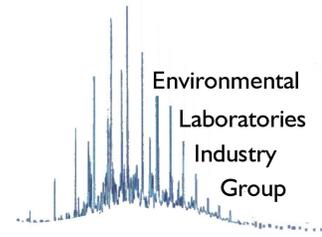
TPH 'method' – needs re write

#### **ELIG Suggestion**

NEPM suggests 16 PAH's be done with TPH – this is wrong.

TPH is not a replacement for SVOC as is hinted.

ELIG has submitted a re write of TPH to the Australian Standards TPH Committee for review and Ivan Povolny from Labmark represents ELIG on this committee.



## **Other**

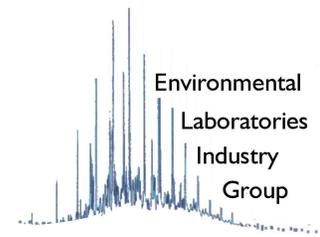
*Issue 34 from NEPC Web Site: Should the NEPM specify the use of particular analytical procedures and methods or should it be more appropriate to specify performance objectives and outcomes for analytical procedures?*

As discussed in the above reply, ELIG does not endorse the use of specific methodology. ELIG is in support of a performance based system.

*Issue 35 from NEPC Web Site: By what mechanism should new analytical techniques in developing areas be incorporated into site assessment work?*

As discussed, ELIG labs analyse almost all of the environmental samples taken in Australia. As such we believe that ELIG should be involved in decisions regarding any new techniques or methodology – after all, we will be the labs having to analyse these samples and use the methodology.

In the past review panels have generally been made up from government and academia, with little or no practical experience in the operations of a commercial laboratory. We believe that this has resulted in the many unworkable and in some cases strange statements in various guidelines. ELIG proposes that a Technical Committee be formed to assess any new methodology and that ELIG plays a key role on this committee. This committee could easily work via email.



## **Appendix 1 Sample Cooling Rates**

ELIG members have noticed that sample batches submitted for laboratory analysis are often lacking in attention to detail with regards to the requirement to comply with the preservation temperature of  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ .

In addition, ELIG members have noted market pressure to not report the sample batch preservation temperature on sample receipt notices, but instead, record the type of chilling medium and a statement that the samples were/ were not chilled only.

A solution to this issue was investigated by ELIG, which consisted of each laboratory conducting a standardised experiment.

### **The Experiment**

The experiment was designed to determine the time necessary to lower the temperature of samples collected in the field to  $4 \pm 2$  degrees in a standard 26L plastic esky using either ice or ice bricks.

The first step was to heat 250ml jars of sand and 1L glass bottles of water to 40 degrees in a drying oven. These samples had holes drilled in the lids so that a thermometer could be inserted into the centre of the matrix.

A jar of sand and bottle of water were then placed in 2 eskies – one containing a 2.5kg emptied bag of ice, the other contained 3 standard sized ice bricks.

The temperature was then recorded hourly for 8 hours by each lab and over 36 hours by 1 lab. The experiment allowed a consensus sample cool-down temperature plot versus time to be constructed which is attached.

### **The Results**

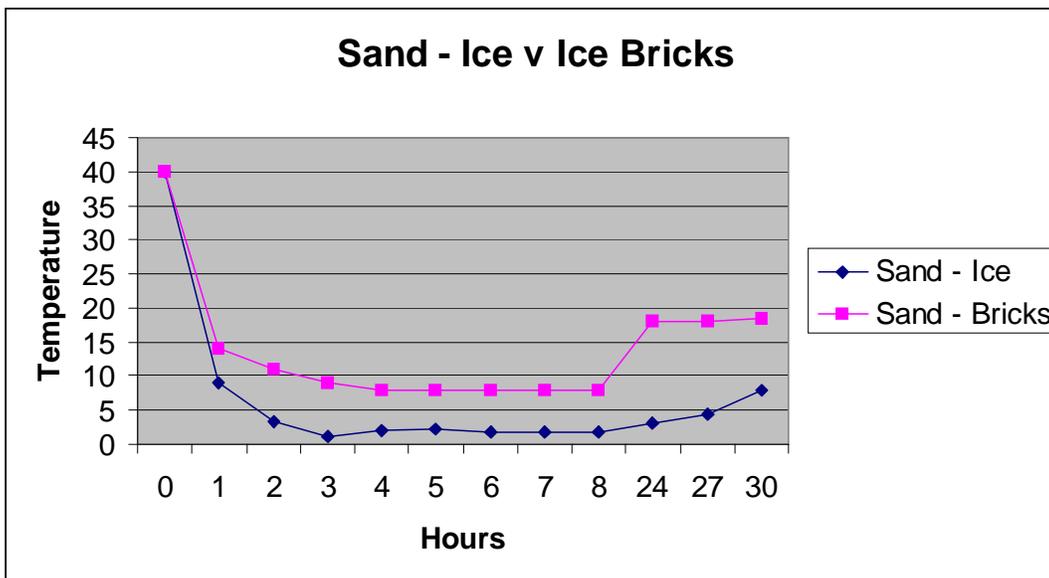
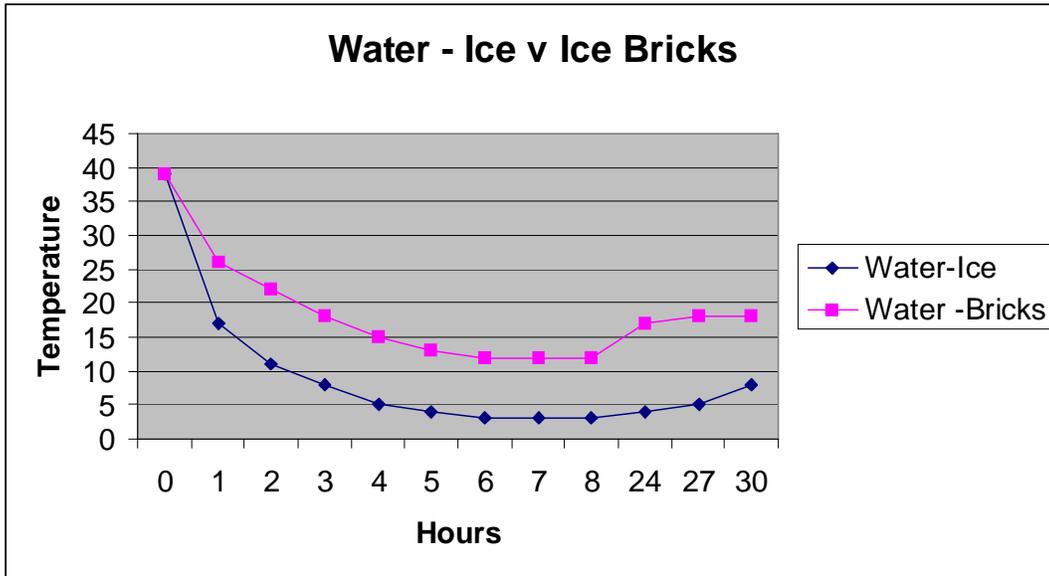
#### **Water Sample.**

- A 1L water sample took 5 hours to cool to 4 degrees in ice. This temperature was maintained for 27 hours before beginning to increase above the compliance temperature.
- A 1L water sample reached 12 degrees with ice bricks at the 8 hour mark.

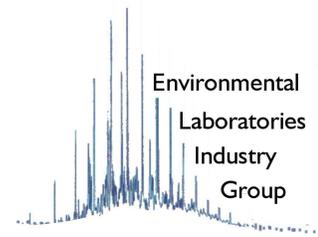
#### **Sand Sample**

- A 250ml sand sample took 2 hours to cool to 4 degrees in ice. This temperature was maintained for 27 hours before beginning to increase above the compliance temperature.
- A 250ml sand sample reached 8 degrees with ice bricks at the 8 hour mark.

As can be seen ice is the preferred cooling medium and will keep samples below 4 degrees for up to 27 hours.



Original data was supplied by AGAL/NMI, Amdel, ALS, Labmark, SAL, SGS and averaged before plotting. There was no significant deviation between original data.



## Solution

1. Preservation temperatures will be recorded on sample receipt notifications. While it is impossible for labs to measure the actual matrix temperature of samples without compromising sample integrity, labs should either record the temperature of the outside of the sample with an infrared scanner or the ambient temperature of the esky with a thermometer probe. Both methods will be investigated further for comparison.
2. The type of chilling medium shall be recorded on the sample receipt notification.
3. It is recommended that all other issues observed by the laboratory relevant to sample presentation be recorded on the sample receipt notice.
4. It is recommended that the Field Scientist shall record the daytime air and ground temperatures during the sampling event so that reference to the sample cool-down temperature curve can establish preservation temperature compliance.

### **Treatment of Samples Presented to the Laboratory > 4°C ± 2°C**

1. Normal analytical procedures shall proceed where samples are received within the sample cool-down temperature curve (ie. samples are on the cool-down gradient after sampling),
2. Consideration will be given to samples received on the same day of sampling.
3. Consideration will be given to the type of analysis (ie. volatile, semi-volatile, non-volatile).
4. Consideration will be given to recommended technical holding times.
5. The laboratory will not provide advice as to whether the sample preservation temperature recorded on the sample receipt notification is compliant, but rather indicate the recorded facts for environmental professional to consider.

### **Note**

1. Due to time constraints within which commercial laboratories operate, the laboratory shall proceed with the COC requested analysis within 24hrs of issuing a sample receipt notification indicating any sample preservation temperature qualification. The analysis shall be reported with a qualification on the report or associated paperwork.
2. Once the analytical determination has proceeded, and where notification had not been received from the client by the laboratory to cancel analysis of a sample not complying with the sample cool-down temperature curve, then the analysis shall be paid for on a pro-rata basis at the stage completed.

Enquiries and comment may be submitted to:

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David Springer at [dspringer@envirolabservices.com.au](mailto:dspringer@envirolabservices.com.au) ELIG President

## Appendix 2 – Metals data – Glass Jars Leaching

NEPM recommends that soil samples for metals analysis be collected in a plastic container instead of glass. This is not practical and ELIG requests this be changed.

ELIG have organized a glass jar leaching experiment to prove that the metals that leach from glass jars are insignificant.

### The Experiment

Laboratories were asked to add a 0.1% Nitric Acid solution to both 125ml and 250ml standard sample glass jars then metals were analysed in the nitric solution by ICP after Day 1, 5 & 10.

### The Results

3 of each jar type were analysed. Results are averaged. This data supplied by NMI with additional data supplied by Envirolab and SAL.

Metal	Day 1 (mg/L)	Day 5 (mg/L)	Day 10 (mg/L)
Aluminium	<0.0001	<0.0001	<0.0001
Arsenic	<0.0001	<0.0001	<0.0001
Antimony	<0.0001	<0.0001	<0.0001
Barium	<0.005	<0.005	<0.005
Boron	<0.006	<0.006	<0.006
Cadmium	<0.0001	<0.0001	<0.0001
Cobalt	<0.0001	<0.0001	<0.0001
Copper	<0.005	<0.005	<0.005
Iron	<0.05	<0.05	<0.05
Lead	<0.005	<0.005	<0.005
Manganese	<0.005	<0.005	<0.005
Mercury	<0.0001	<0.0001	<0.0001
Molybdenum	<0.0005	<0.0005	<0.0005
Nickel	<0.0005	<0.0005	<0.0005
Selenium	<0.0005	<0.0005	<0.0005
Calcium	0.4	0.5	0.6
Potassium	<0.05	<0.05	<0.05
Magnesium	<0.05	<0.05	<0.05
Sodium	1.2	1.6	1.9
Silicon	0.05	0.13	0.17
Sulphur	<0.05	<0.05	<0.05
Tin	<0.05	<0.05	<0.05
Zinc	<0.005	<0.005	<0.005

### Conclusion

There are slight increases with time for Ca, Na and Si, however, soils are rarely required to be tested for these analytes and these levels are considered insignificant. It is recommended that soils for metals analysis be collected in either glass or plastic.

