

## APPENDIX 1: PARAMETERS AND ANALYTICAL METHODS REQUIRED FOR CLASSIFICATION OF SLUDGE AND MONITORING OF SLUDGE, WATER AND SOIL SAMPLES

### Appendix 1.1: Analyses required for classification and characterization of sludge

Characteristic	Parameter	Guidance on methodology and/or recommended extraction method
Physical characteristics	pH	Direct measurement pH on saturated paste or solution
	Total solids (TS)	Standard method 2540B <sup>1</sup>
	Volatile suspended solids (VSS)	Standard method 2540E <sup>2</sup>
	Volatile Fatty Acids (VFA)	Adapted from Standard methods. The full method is detailed in Volume 1, Appendix 2.
Nutrients	Total Kjeldahl Nitrogen (TKN)	The suggested method description has been attached in Volume 1, Appendix 2.
	Total Phosphorus (TP)	The suggested method description has been attached in Volume 1, Appendix 2.
	Potassium (K)	The suggested method description has been attached in Volume 1, Appendix 2.
Metals and micro-elements	Arsenic Cadmium Chromium Copper Lead Mercury Nickel Zinc (Any other metal or element identified during the comprehensive characterisation detailed in Volume 1)	For beneficial use land the extraction of trace elements with <i>aqua regia</i> solution is recommended. International Standard ISO 11466 (Ref number: ISO11466:1995(E))  For use as landfill cover the TCLP test is recommended. US EPA Method 1311, 1992  Note: A semi-quantitative ICP scan would give concentrations for all mentioned metals. Remind the laboratory to manage the interferences on the ICP appropriately, especially for compounds such as Arsenic.
Microbiological quality	Faecal coliforms	m-FC medium
	Total viable Helminth ova	See recommended new method further on in this Appendix
<sup>1,2</sup> Standard Methods for the Examination of Water and Wastewater, 20th edition (1998) or latest, by Leonore S. Clesceri, Arnold E. Greenbert and R. Rhodes Trussell.		

## Appendix 1.2: Sludge analyses required for monitoring purposes

Characteristic		Parameter	Guidance on methodology and/or recommended extraction method
Physical characteristics		pH	Direct measurement pH on saturated paste or solution
		Total solids (TS)	Standard method 2540B <sup>1</sup>
		Volatile suspended solids (VSS)	Standard method 2540E <sup>2</sup>
		Volatile Fatty Acids (VFA)	Adapted from Standard methods. The full method is detailed in Volume 1, Appendix 2.
Chemical characteristics	Nutrients	Total Kjeldahl Nitrogen (TKN)	The suggested method description has been attached in Volume 1, Appendix 2.
		Total Phosphorus (TP)	The suggested method description has been attached in Volume 1, Appendix 2.
		Potassium (K)	The suggested method description has been attached in Volume 1, Appendix 2.
	Metals and micro-elements	Arsenic Cadmium Chromium Copper Lead Mercury Nickel Zinc (Any other metal or element identified during the comprehensive characterisation detailed in Volume 1)	For beneficial use land the extraction of trace elements with <i>aqua regia</i> solution is recommended. International Standard ISO 11466 (Ref number: ISO11466:1995(E))  For use as landfill cover the TCLP test is recommended. US EPA Method 1311, 1992  Note: A semi-quantitative ICP scan would give concentrations for all mentioned metals. Remind the laboratory to manage the interferences on the ICP appropriately.
Microbiological quality	Faecal coliforms	m-FC medium	
	Total viable Helminth ova	See recommended new method further on in this Appendix	
<p><sup>1,2</sup> Standard Methods for the Examination of Water and Wastewater, 20th edition (1998) or latest, by Leonore S. Clesceri, Arnold E. Greenbert and R. Rhodes Trussell.</p>			

### Appendix 1.3: Surface and groundwater analyses required for monitoring purposes

Characteristic	Parameter	Guidance on methodology and/or recommended extraction method
Water chemistry	pH	Direct measurement
	EC	Direct measurement
	PO <sub>4</sub>	Standard method 4500-P <sup>1</sup>
	NH <sub>4</sub>	Standard method 4500-NH <sub>4</sub> <sup>1</sup>
	NO <sub>3</sub>	Standard method 4500-NO <sub>3</sub> <sup>1</sup>
	COD	Standard method 5220D <sup>1</sup>
Water microbiology	Faecal coliforms	Membrane filter/ m-FC medium <sup>1</sup>
	<i>E coli</i>	Standard method 9221B <sup>1</sup>
<sup>1</sup> Standard Methods for the Examination of Water and Wastewater, 20th edition (1998) or latest, by Leonore S. Clesceri, Arnold E. Greenbert and R. Rhodes Trussell		

#### Appendix 1.4: Soil analyses required for monitoring purposes

Characteristic	Parameter	Guidance on methodology and/or recommended extraction method
Nutrients	Total Kjeldahl Nitrogen (TKN)	The suggested method description has been attached in Volume 1, Appendix 2.
	Total Phosphorus (TP)	The suggested method description has been attached in Volume 1, Appendix 2.
Metals to assess compliance in terms of the TMT and MPL	<p>Arsenic Cadmium Chromium Copper Lead Mercury Nickel Zinc</p> <p>(Any other metal or element identified during the comprehensive characterisation detailed in Volume 1)</p>	<p>Extraction of trace elements soluble in <i>aqua regia</i> solution.</p> <p>International Standard ISO 11466 Method Reference number: ISO 11466:1995 (E)</p> <p>Note: A semi-quantitative ICP scan would give concentrations for all mentioned metals. Remind the laboratory to manage the interferences on the ICP appropriately.</p>

## Appendix 1.5: Recommended new procedure to determine Helminth ova in wastewater sludge

**Note:** This is a new method which was developed after Volume 1 of the New Sludge Guidelines have been published and differs from the method published in Volume 1.

### Method for analyses of sludge

**Note:** It is always preferable to work with small sub-samples as eggs may not be as easily released from a large sample to float out of the sludge when doing the ZnSO<sub>4</sub> flotation technique. Rather increase the number of sub-samples than overload each test-tube in order to keep the number of tubes down.

The number of sub-samples will also be dependent on the helminth ova load expected. This will require knowledge of the epidemiology of helminths in the particular area in South Africa. Consequently, more sub-samples must be done in an area of low endemicity and less in a highly endemic area.

1. Mix the sludge sample well by swirling and stirring with a plastic rod. From the total sample take 4 x 15ml sub-samples and place them into 4 x 50ml test tubes. (If the solid content is high this should be sufficient sample. If it is low, take more 15ml sub-samples).
2. Add either a few millilitres of 0.1% Tween80 or AmBic solution to the samples, vortex and add more wash solution. Repeat this procedure until the tubes are filled to approximately a centimetre from the top.
3. Place the 150µm sieve in a funnel in a retort stand with a plastic beaker underneath to catch the filtrate. Filter the well-mixed contents of the tubes one at a time, rinsing out each tube and washing this water through the sieve as well.
4. Pour the filtrate into test tubes and centrifuge at 1389g (±3000rpm) for 3 minutes. Suction off the supernatant fluids and discard. Combine the deposits into a suitable number of tubes so that there is not more than 1ml in a 15ml tube or 5ml in a 50ml tube
5. Re-suspend each of these deposits in a few millilitres of ZnSO<sub>4</sub> and vortex well to mix. Keep adding more ZnSO<sub>4</sub> and mixing until the tube is almost full.
6. Centrifuge the tubes at 617g (±2000rpm) for 3 minutes. Remove from the centrifuge and pour the supernatant fluids through the 20µm filter, washing well with water.
7. Collect the matter retained on the sieve and wash it into test tubes.
8. Centrifuge the tubes at 964g (±2500rpm) for 3 minutes; remove & discard the supernatant fluid. Combine the deposits into one test tube, using water to recover all the eggs from the other tubes. Then centrifuge again at 964g for 3 minutes to get one deposit.
9. Once there is one final deposit, remove all of it using a plastic Pasteur pipette and place it onto one or more microscope slides. Place a coverslip over each deposit and examine microscopically using the 10x objective and the 40x objective to confirm any unsure diagnoses.

10. Each species of helminth ova is enumerated separately and reported as eggs per gram of sludge.

**Note:** Samples may be examined slightly differently from that described in step No. 10 above by doing the following:

The deposits are filtered through a 12µm ISOPORE membrane, which is then rinsed with distilled water. The membrane is air-dried, cut in half and placed on a microscope slide. Immersion oil is used to clear the membrane before examining under the microscope.

To test for viability:

Perform steps 1 to 8 of the procedure above and continue as follows:

9. Once there is a final deposit in the test tube, re-suspend it in 4ml of 0.1 H<sub>2</sub>SO<sub>4</sub>. Before incubating mark the test tube with the level of liquid and incubate at a temperature of 26°C for three to four weeks. Check the level of liquid in each one of the test tubes and add the reagent every time that is necessary, compensating for any evaporation that may occur.
10. Once the incubation time is over, homogenize the deposit and proceed to quantify the eggs. Remove all of the deposit using a plastic Pasteur pipette and place it onto one or more microscope slides. Place a coverslip over each deposit and examine microscopically using the 10x objective and the 40x objective to confirm any unsure diagnoses. Only those ova where the larva is observed are considered viable.

#### Equipment required and related information

1. A centrifuge with a swing-out rotor and buckets that can take 15ml and/or 50ml plastic conical test tubes.
2. Vortex mixer.
3. Retort Stand with at least 2 clamps on it.
4. Large plastic funnels to support the filters (±220mm diameter).
5. Filters / Sieves : 1x 150µm; 1x 100µm; 1x 20µm.
6. Approx. 6 Plastic beakers (500ml) & 3 Plastic wash bottles.
7. At least 4 glass "Schott" bottles (1lt, 2lt & 5lt sizes) for make-up and storage of the chemical solutions and de-ionized water.
8. Magnetic stirrer and stirring magnets.
9. 15ml and 50ml plastic conical test tubes.
10. 3 x Small glass beakers (100ml).
11. Plastic Pasteur Pipettes & Plastic Stirring Rods.
12. Glass microscope slides (76 x 26 x 1,2 mm).
13. Square & Rectangular Cover-slips (22 x 22mm & 22 x 40mm).

14. A binocular compound microscope with 10x eyepieces, a 10x objective and a 40x objective.

#### Working out the g-force of your centrifuge

$$\text{G-force (or g)} = (1,118 \times 10^{-5}) r s^2 = 0,00001118 \times r \times s^2$$

where :  $s$  = revolutions per minute (i.e. the speed you spin at)

$r$  = the radius (the distance in centimetres from the centre of the rotor to the bottom of the bucket holding the tubes, when the bucket is in the swing-out position)

#### Reagents

##### **Zinc Sulphate**

- $\text{ZnSO}_4$  (heptahydrate) is made up by dissolving 500g of the chemical in 880ml de-ionised or distilled water.
- A hydrometer must be used to adjust the specific gravity (SG) to 1.3, using more chemical if the SG is too low or more water if it is  $>1,3$ .

This high specific gravity facilitates the floating of heavier ova such as *Taenia* sp. (SG = 1.27). It is not critical if the SG of the  $\text{ZnSO}_4$  solution is just over 1.3 but it should never be below this value!

##### **Ammonium Bicarbonate**

The AMBIC solution is essentially a saturated ammonium bicarbonate solution. Ammonium bicarbonate can be obtained from Merck Chemicals and is made up by dissolving 119g of the chemical in 1000ml of de-ionised water.

##### **0,1% Tween80**

1ml of Tween80 is measured out using a pipette and placed in 1000ml of de-ionized or distilled water to give a 0,1% wash solution.

**Note:** Tween80 is extremely viscous and it is necessary to wash **all** of it out into the water in which it is made up, by alternately sucking up water and blowing it out using the same pipette.

##### **References:**

WRC Report number: TT 321/08. Standard methods for the recovery and enumeration of Helminth ova in wastewater, sludge, compost and urine diversion waste in South Africa.

Posters: Standard methods and photographs of Helminth ova.

## Appendix 1.6: Toxicity Characteristic Leaching Procedure (TCLP) extraction for sludge destined for co-disposal (USEPA Method 1311)

### Summary of method

- For liquid wastes (containing <0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8  $\mu\text{m}$  glass fiber filter, is defined as the TCLP extract.
- For wastes containing  $\geq 0.5\%$  solids, the liquid, if any, is separated from the solid phase and stored for later analyses.

### Apparatus

- Agitation apparatus capable of rotating the extraction vessel in an end-over-end fashion at  $30 \pm 2$  r.p.m.
- Extraction bottles for inorganics. These may be constructed from various materials. Borosilicate glass bottles are highly recommended. Polytetrafluoroethylene (PTFE), high density polyethylene (HDPE), polypropylene (PP), Polyvinyl chloride (PVC) and stainless steel bottles may also be used.

### TCLP solution 1

- Add 5.7 ml glacial Acetic Acid to 500 ml of reagent quality water (double distilled water).
- Add 64.3 ml of 1N NaOH.
- Dilute to a volume of 1 litre.
- When correctly prepared, the pH of this solution will be  $4.93 \pm 0.05$ .

### TCLP solution 2

- Dilute 5.7 ml glacial acetic acid with double distilled water to a volume of 1 litre.
- When correctly prepared, the pH of this solution will be  $2.88 \pm 0.05$ .

### Samples

- The sample must be a minimum of 100 grams.
- The sample must be able to pass through a 9.5 mm sieve, i.e. particle size of the solid must be smaller than 10 mm.

### TCLP extractions

**Note** that the TCLP test requires that a waste be pre-tested for its acid neutralization capacity. Those with low acid neutralization capacity are extracted with TCLP solution 1 (0.1M Sodium Acetate Buffer, pH  $4.93 \pm 0.05$ ) and those with high acid neutralization capacity are extracted with TCLP solution 2 (0.1M Acetic Acid, pH  $2.88 \pm 0.05$ ). Most sludges have a low acid neutralization capacity and will, therefore, be extracted with TCLP solution 1. After addition of lime, the acid neutralization capacity of the sludge is increased, but note that the treated sludge should be leached using the TCLP solution used for original sludge,

*i.e.* in most cases TCLP solution 1, so that the results are directly comparable and one can evaluate the effect of the lime treatment. This is correct even though the pre-test used in the TCLP on the lime treated sludge may indicate that TCLP solution number 2 should be used.

#### **A. Preliminary evaluation:**

This part of the extraction procedure must be performed to determine which TCLP (No. 1 or 2) solution should be used (see extraction solutions).

1. Weigh out 5.0 grams of the dry waste into a 500 ml beaker or Erlenmeyer flask. (In this exercise the particle size of the 5 grams should be 1 mm or less).
2. Add 96.5 ml of double distilled water, cover with a watch glass and stir vigorously for 5 minutes with a magnetic stirrer.
3. Measure the pH.
4. If the pH is less than 5.0, then use TCLP solution - No 1.
5. If the pH is greater than 5.0, then proceed as follows:
  - 5.1 Add 3.5 ml 1N HCl and stir briefly.
  - 5.2 Cover with a watch glass, heat to 50°C and hold at 50°C for ten minutes.
  - 5.3 Let cool to room temperature and record the pH.
6. If the pH is less than 5.0, then use TCLP solution - No 1.
7. If the pH is more than 5.0, then use TCLP solution - No 2.

#### **B. Extraction for analysis of contaminants:**

1. Weigh out 100 gram of the dry waste, which passes through a 9.5 mm sieve, and quantitatively transfer it to the extraction bottle.
2. Add two litres (2l) of the appropriate TCLP solution (No. 1 or 2 as determined by preliminary evaluation) and close bottle tightly.
3. Rotate in agitation apparatus at 30 r.p.m. for 20 hours. Temperature of room in which extraction takes place should be maintained at  $23 \pm 2^\circ\text{C}$ .
4. Filter through a glass fibre filter and collect filtrate. Record pH of filtrate.
5. Take aliquot samples from the filtrate for determination of metal concentrations.
6. Immediately acidify each aliquot sample with nitric acid to a pH just less than 2.
7. Analyse by AA or other sensitive and appropriate techniques for different metals.
8. If analysis cannot be performed immediately after extraction, then store the acidified aliquots at 4°C, until analysis (as soon as possible).

Reference: USEPA Test Methods SW-846 On-line  
<http://www.epa.gov/epaoswer/hazwaste/test/pdfs/1311.pdf>

## APPENDIX 2: VECTOR ATTRACTION REDUCTION OPTIONS

The following options are available to reduce the vector attraction potential. These options have been adopted from the US EPA Part 503 Rule.

### Option 1: Reduction in Volatile Solids Content

Vector attraction is reduced if the fraction of volatile solids in the primary sludge is reduced by at least 38 percent during the treatment of the sludge. This percentage is the amount of volatile solids reduction that is attained by anaerobic or aerobic digestion plus any additional volatile solids reduction that occurs before the sludge leaves the treatment works, such as through processing in drying beds or lagoons, or by composting.

Digestion process efficiency can be measured by the reduction in the volatile solids content of the feed sludge to the digester and the sludge withdrawn from the digester. Anaerobic digestion of primary sludge generally results in a reduction of between 40 and 60% of the volatile solids.

O'Shaunessy's formula can be used to calculate the volatile solids (VS) reduction in a digester:

$$\text{VS reduction (\%)} = \{(V_i - V_o) / V_i - (V_i \times V_o)\} \times 100$$

Where  $V_i$  = volatile fraction in feed sludge

$V_o$  = volatile fraction in digested sludge

Example of calculation of VS reduction

Assume volatile solids in feed sludge = 84%

Therefore volatile fraction of feed sludge = 0.84 =  $V_i$

Assume volatile solids of digested sludge = 68%

Therefore volatile fraction of digested sludge = 0.68 =  $V_o$

$$\begin{aligned} \text{VS reduction (\%)} &= \{(0.84 - 0.68) / 0.84 - (0.84 \times 0.68)\} \times 100 \\ &= 59\% \end{aligned}$$

### Option 2: Additional Digestion of Anaerobically Digested Sludge

Frequently, primary sludge is recycled to generate fatty acids or the sludge is recycled through the biological wastewater treatment section of a treatment works or has resided for long periods of time in the wastewater collection system. During this time, the sludge undergoes substantial biological degradation. If the sludge is subsequently treated by anaerobic digestion for a period of time, it adequately reduces vector attraction. Because the sludge will have entered the digester already partially stabilized, the volatile solids reduction after treatment is frequently less than 38 percent.

Under these circumstances, the 38 percent reduction required by Option 1 may not be achievable. Option 2 allows the operator to demonstrate vector attraction reduction by testing a portion of the previously digested sludge in a **bench-scale unit** in the laboratory. Vector attraction reduction is demonstrated if, after anaerobic digestion of the sludge for an additional 40 days at a temperature between 30°C and 37°C, the volatile solids in the sludge are reduced by less than 17 percent from the beginning to the end of the bench test.

#### Option 3: Additional Digestion of Aerobically Digested Sludge

This option is appropriate for aerobically digested sludge that cannot meet the 38 percent volatile solids reduction required by Option 1. This includes activated sludge from extended aeration plants, where the minimum residence time of sludge leaving the wastewater treatment processes section generally exceeds 20 days. In these cases, the sludge will already have been substantially degraded biologically prior to aerobic digestion.

Under this option, aerobically digested sludge with 2 percent or less solids is considered to have achieved vector attraction reduction, if in the laboratory after 30 days of aerobic digestion in a batch test at 20°C, volatile solids are reduced by less than 15 percent. This test is only applicable to liquid aerobically digested sludge.

#### Option 4: Specific Oxygen Uptake Rate (SOUR) for Aerobically Digested Sludge

Frequently, aerobically digested sludge is circulated through the aerobic biological wastewater treatment process for as long as 30 days. In these cases, the sludge entering the aerobic digester is already partially digested, which makes it difficult to demonstrate the 38 percent reduction required by Option 1.

The specific oxygen uptake rate (SOUR) is the mass of oxygen consumed per unit time per unit mass of total solids (dry-weight basis) in the sludge. Reduction in vector attraction can be demonstrated if the SOUR of the sludge that is used or disposed, determined at 20°C, is equal to or less than 2 milligrams of oxygen per hour per gram of total sludge (dry-weight basis). This test is based on the fact that if the sludge consumes very little oxygen, its value as a food source for micro-organisms is very low and therefore micro-organisms are unlikely to be attracted to it. Other temperatures can be used for this test, provided the results are corrected to a 20 °C basis. This test is only applicable to liquid aerobic sludge withdrawn from an aerobic treatment process.

#### Option 5: Aerobic Processes at Greater than 40°C

This option applies primarily to composted sludge that also contains partially decomposed organic bulking agents. The sludge must be aerobically treated for 14 days or longer, during which time the temperature must always be over 40°C and the average temperature must be higher than 45°C.

This option can be applied to other aerobic processes, such as aerobic digestion, but Options 3 and 4 are likely to be easier to meet than the other aerobic processes.

#### Option 6: Addition of Alkaline Material

Sludge is considered to be adequately reduced in vector attraction if sufficient alkaline material is added to achieve the following:

- Raise the pH to at least 12, measured at 25°C, and without the addition of more alkaline material, maintain a pH of 12 for at least 2 hours.
- Maintain a pH of at least 11.5 without addition of more alkaline material for an additional 22 hours.

The conditions required under this option are designed to ensure that the sludge can be stored for at least several days at the treatment works, transported, and then used or disposed without the pH falling to the point where putrefaction occurs and vectors are attracted.

#### Option 7: Moisture Reduction of Sludge Containing no Un-stabilised Solids

Under this option, vector attraction is considered to be reduced if the sludge does not contain unstabilised solids generated during primary treatment and if the solids content of the sludge is at least 75 percent before the sludge is mixed with other materials. Thus, the reduction must be achieved by removing water, not by adding inert materials.

It is important that the sludge does not contain un-stabilised solids because the partially degraded food scraps likely to be present in such sludge would attract birds, some mammals, and possibly insects, even if the solids content of the sludge exceeds 75 percent. In other words, simply dewatering primary sludge to a 75% solid is not adequate to comply with this option. Activated sludge, humus sludge and anaerobically digested sludge can, however be dewatered to 75 % solids and comply with option 7.

#### Option 8: Moisture Reduction of Sludge Containing Unstabilised Solids

The ability of any sludge to attract vectors is considered to be adequately reduced if the solids content of the sludge is increased to 90 percent or greater, regardless of whether this contains primary sludge or raw unstabilised sludge. The solids increase should be achieved by removal of water and not by dilution with inert solids. Drying to this extent severely limits biological activity and strips off or decomposes the volatile compounds that attract vectors.

The way dried sludge is handled, including storage before use or disposal, can again create the opportunity for vector attraction. If dried sludge is exposed to high humidity, the outer surface of the sludge will increase in moisture content and possibly attract vectors. This should be properly guarded against.

#### Option 9: Sludge Injection

Vector attraction reduction can be demonstrated by injecting the sludge below the ground surface. Under this option, no significant amount of sludge can be present on the land surface within 1 hour of injection, and if the sludge is Microbiological Class A or B, it must be injected within 8 hours after discharge from the pathogen-reducing process.

The reason for this special consideration for Microbiological class A and B sludge (assuming vector attraction has not been reduced by some other means) is that pathogens could re-

grow and Microbiological class A and B sludge has no site restrictions to provide crop, animal grazing or access protection.

**Note:** Microbiological class A and B can be applied to soil much later than 8 hours after discharge from the pathogen-reducing process if another vector attraction reduction option such as dewatering and/or drying is applied. The time periods referred to in Option 9 are intended for liquid sludge application of Microbiological classes A and B.

Injection of sludge beneath the soil places a barrier of earth between the sludge and vectors. The soil removes water from the sludge, which reduces the mobility and odour of the sludge. Odour is usually present at the site during the injection process, but quickly dissipates once injection is complete. This option is applicable all land disposal options and co-disposal on landfill.

#### Option 10: Incorporation of Sludge into the Soil

Under this option, sludge must be incorporated into the soil within 6 hours of application to or placement on the land. Incorporation is accomplished by ploughing or by some other means of mixing the sludge into the soil. If the sludge is Microbiological class A or B with respect to pathogens, the time between processing and application or placement must not exceed 8 hours – the same as for injection under Option 9. See the note under Option 9. This option is applicable all land disposal options and co-disposal on landfill.

**Note:** Practical restrictions, such as the ability of the plough to function immediately after application, could cause delays in the incorporation of the sludge within the 6 hours. This could cause the development of odours and increase risk of vector attraction. In these cases the sludge producer needs to monitor the development of odours and manage the situation diligently.

## **APPENDIX 3: ESSENTIAL CONDITIONS TO BE INCLUDED IN A CONTRACTUAL AGREEMENT BETWEEN A SLUDGE PRODUCER AND SLUDGE USER**

### **Producer**

1. Name and address
2. Name and contact details of responsible person (signatory)
3. Classification of sludge
4. Statement on permissible beneficial use option based on sludge classification (Refer to Table 7 of this document)
5. Volume and type (liquid or dewatered) of sludge to be supplied
6. Notification of authorities involved where applicable

### **User**

1. Name and address
2. Name and contact details of responsible person (signatory)
3. Name of transporter of sludge
4. Name and location of site where sludge will be used
5. Specification of beneficial use option
6. Previous sludge application – annual rate and frequency
7. Metal and inorganic content of soil. Soil to be analysed before commencing sludge application and monitored as described in this document where applicable

### **Agreement**

1. Sludge to be used subject to Guideline Volume 4 (Site considerations, odour control, soil quality, sludge application rate, crop restrictions, monitoring requirements and record keeping requirements)
2. Inspection of user's activities by any appropriate authority
3. Breach of contract – termination of sludge supply and punitive measures

## APPENDIX 4: SAMPLING METHODS AND PROCEDURES FOR WATER AND SOIL SAMPLES

### WATER SAMPLING<sup>4</sup>

#### *Sampling equipment needed*

- Equipment to collect microbiological samples
  - Sterile sample bottles (see Table 25 and 26 for the type of sample bottle needed)
  - Sealed container or cool box which can be kept cool (preferably with ice)
- Equipment to collect chemical and physical samples
  - Correct sample bottles (see Table 25 and 26 for the different types of sample bottles required)
  - Cooler box with ice (if necessary)

#### *Special precautions*

- Microbiological water samples
  - Keep sample bottle closed and in a clean condition up to the point where it has to be filled with the water to be sampled.
  - Do not rinse bottle with any water prior to sampling.
  - When samples for chemical and microbiological analysis are to be collected from the same location, the microbiological sample should be collected first to avoid the danger of microbiological contamination of the sampling point.
  - The sampler (person taking the sample) should wear gloves (if possible) or wash his/her hands thoroughly before taking each sample. Avoid hand contact with the neck of the sampling bottle.
- Chemical water samples
  - Some plastic caps or cap liners may cause metal contamination of the water sample. Please consult with the laboratory on the correct use of bottle caps.
  - Keep sample bottle closed and in a clean condition up to the point where it has to be filled with the water to be analysed.
  - Never leave the sample bottles (empty or filled with the water sample) unprotected in the sun.
  - After the sample has been collected the sample bottle should be placed directly in a cooled container (e.g. portable cooler box). Try and keep cooled container dust-free.

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<sup>4</sup> For more detail on the water sampling procedure, consult the following documents:  
Department of Water Affairs and Forestry. 1998. Waste Management Series. Minimum Requirements for Water Monitoring at Waste Management Facilities.  
WRC. 2000. Quality of domestic water supplies. Volume 2: Sampling Guide. WRC no TT117/99.

### *Surface water sampling technique*

The following procedures should be followed when taking water samples in rivers and streams:

- At the sampling point remove cap of sample bottle but do not contaminate inner surface of cap and neck of sample bottle with hands.
- Take samples by holding bottle with hand near base and plunge the sample bottle, neck downward, below the water surface (wear gloves to protect your hands from contact with the water).
- Turn bottle until neck points slightly upward and mouth is directed toward the current (can also be created artificially by pushing bottle forward horizontally in a direction away from the hand).
- Fill sample bottle without rinsing and replace cap immediately.
- Before closing the sample bottle, preserve the sample (if applicable, see Table 25) and leave ample air space in the bottle (at least 2.5 cm) to facilitate mixing by shaking before examination.
- Label the sample.
- Submit for analysis to a reputable analytical laboratory.

### *Composite Borehole Water Sampling*

Composite water sampling is done by pumping water from a borehole. The recommended procedure for composite sampling is as follows:

- Activate the pump and remove (purge) at least three times the volume of water contained in the hole.
- Collect a water sample in a clean container (see Table 26).
- Filter and preserve the sample (if applicable, see Table 26) and submit for analysis to a reputable analytical laboratory.

Various types of pumps may be used. As a portable system, a submersible pump may be considered. Submersible pumps are generally available in South Africa. For sampling, a small submersible pump that yields 1 l/sec would be sufficient for most sampling applications.

Where low-yielding monitoring boreholes are pumped, the borehole could temporarily run dry while being purged. In such instances, samples should be taken of the newly accumulated groundwater after recovery or partial recovery of the water level in the holes. It may be necessary to sample such boreholes a day or more after having purged the hole.

## SOIL SAMPLING<sup>5</sup>

### *Sampling equipment needed*

- Soil auger
- Plastic sheets
- Plastic or glass containers (bottles or bags) that can be closed tightly
- Tags and a permanent marker to label the samples

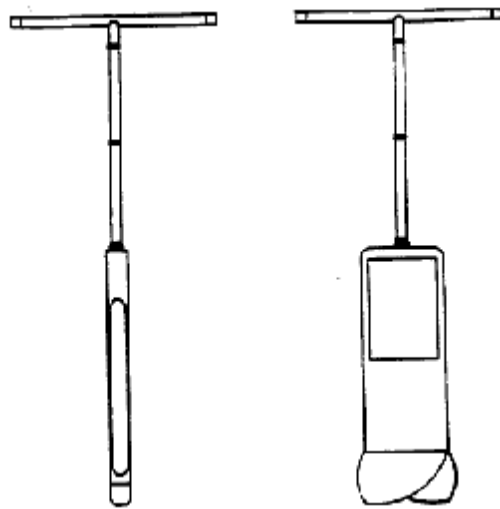


Figure A3: Soil augers

### *Number of samples*

The number of samples will vary according to the size of the beneficial use site and different soil types present at the site. At least three composite samples for each depth increment for every hectare of the site is required.

### *Sampling procedure*

The **soil auger** is used to bore a hole to a desired sampling depth, and is then withdrawn. The sample may be collected directly from the auger. The following procedure is recommended:

1. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter).
2. Begin augering and after reaching the desired depth, slowly and carefully remove the auger from the hole. Deposit the soil onto a plastic sheet spread near the hole. For

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<sup>5</sup> For more information on soil sampling procedures, consult the following documents:  
USEPA Environmental Response Team. 2000. Standard operating procedures: Soil sampling  
USEPA 1989. Soil sampling quality assurance: User's Guide. EPA 600/8-89/046

soil monitoring at disposal sites these depths are 0-100mm, 100-200mm, 200-300mm, 300-400mm and 400-500mm.

3. Place the samples into plastic or other appropriate containers, secure the caps tightly and label the sample.
4. If composite samples are to be collected, place a sample from another sampling site into the same container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.
5. Preserve the samples as recommended in Table A4 and submit to a reputable laboratory.

**TABLE A4: RECOMMENDED SOIL SAMPLE CONTAINERS, PRESERVATION AND HOLDING TIMES**

Contaminant	Container	Preservation	Holding Time
Acidity	Plastic/Glass	Cool, 4°C	14 days
Ammonia	Plastic/Glass	Cool, 4°C	28 days
Sulfate	Plastic/Glass	Cool, 4°C	28 days
Nitrate	Plastic/Glass	Cool, 4°C	48 hours
Organic Carbon	Plastic/Glass	Cool, 4°C	28 days
Chromium VI	Plastic/Glass	Cool, 4°C	48 hours
Mercury	Plastic/Glass	Cool, 4°C	28 days
Other Metals	Plastic/Glass	Cool, 4°C	6 months

Soil samples can also be collected from a **test pit or trench excavation**. The following procedure is recommended:

1. A shovel is used to remove a one to two inch layer of soil from the vertical face of the pit where sampling is to be done.
2. Samples are taken using a trowel, scoop, or coring device at the desired intervals. Be sure to scrape the vertical face at the point of sampling to remove any soil that may have fallen from above, and to expose fresh soil for sampling.
3. Place the samples into plastic or other appropriate containers, secure the caps tightly and label the sample.
4. If composite samples are to be collected, place a sample from another sampling site into the same container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.
5. Preserve the samples as recommended in Table A4 and submit to a reputable laboratory.

## APPENDIX 5: CALCULATING THE TOTAL LOAD / MAXIMUM MIXING RATIO FOR SLUDGE USED AS LANDFILL COVER

Example: Calculating the allowable mass of sludge with soil for use as a vegetative layer using the EEC principle.

A landfill with a surface area of 70 ha is to be covered. The 200 mm topsoil or vegetative layer is to contain sewage sludge as a soil conditioner.

Sludge properties:

Zn concentration from TCLP test : 5.8 mg Zn/kg dry solids

Sludge moisture content : 20% solids

Sludge density : 600 kg/m<sup>3</sup>

Solution: (following the procedure in Section 8.6 of the Minimum Requirements)

LC50 = 7 mg/l

AE = 0.1 x LC<sub>50</sub>

AE = 0.1 x 7 mg/l

AE = 700 ppb

EEC = AE

g/ha/month x 0.66 = AE

= 700 / 0.66

= 1061 g/ha/month

but, this is a once-off application, therefore

EEC = 1061 g/ha

Mass of sludge (dry solids) = 1061 g/ha / 5.8 mg/kg

= 18.293 tonnes/ha

Mass of sludge (wet mass) = 18.293 / (20/100) tonnes/ha

= 91.465 tonnes/ha

Volume of sludge = 91.465 tonnes/ha / 600 kg/m<sup>3</sup>

= 152.4 m<sup>3</sup>/ha

Volume of vegetative layer/ha = 0.2 x 1000 x 1000 m<sup>3</sup>/ha

= 200 000 m<sup>3</sup>/ha (this is the final, compacted volume)

The moisture in the sludge impacts on the compaction density that can be achieved and in fact, on the optimum moisture content and density of the mixture. The actual mass of soil can therefore not be determined for this example since a series of compaction trials of soil and sludge mixtures should be undertaken. Once a suitable ratio is chosen, the maximum mass of sludge permitted in the compacted layer is 91.5 tonnes/ha. Despite the maximum permissible load in terms of the EEC procedure, the properties of the mixture must still be considered for construction of the cover.