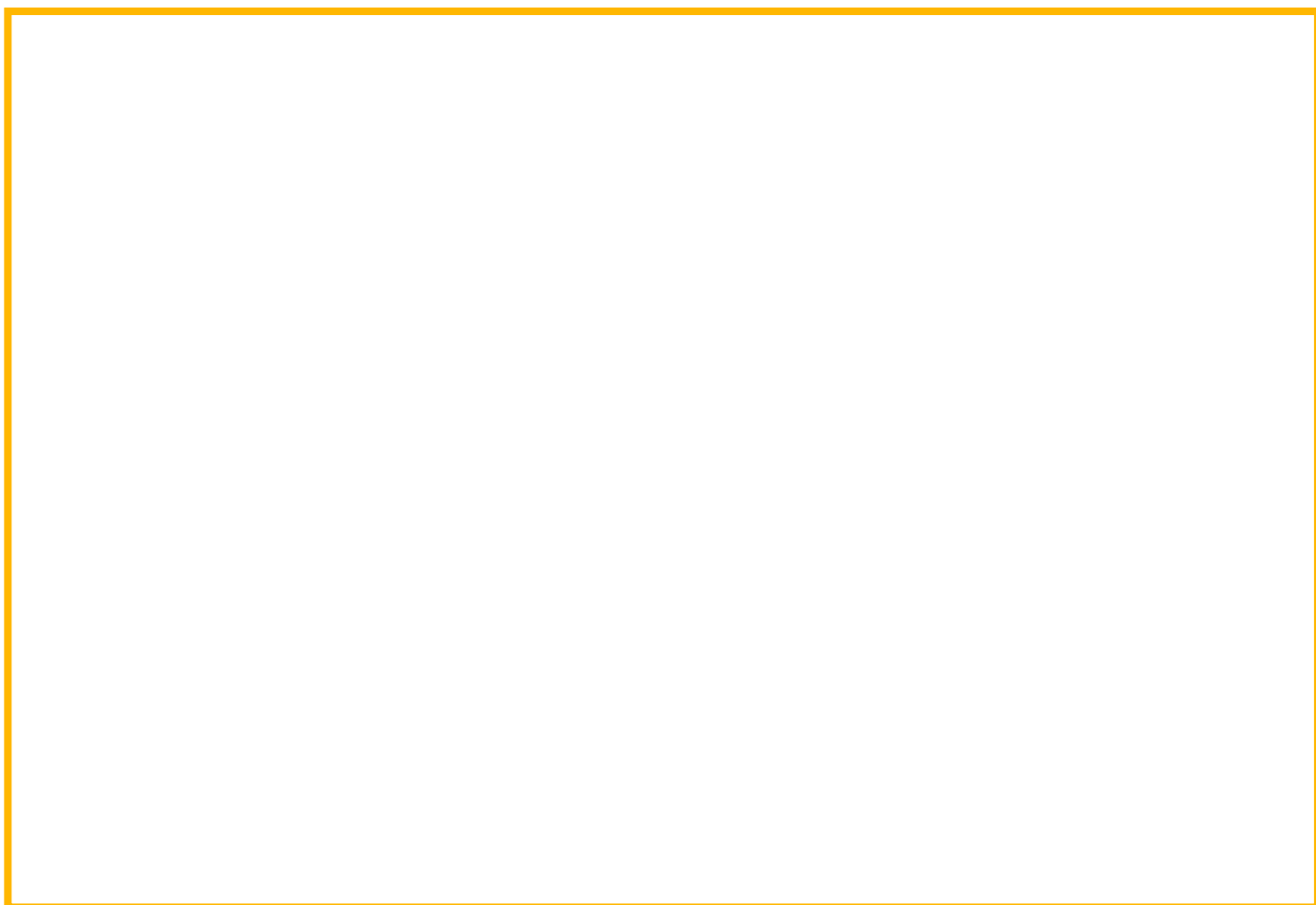


# LICENCE

for

**DR AS 4439.2:2019, Wastes, sediments and contaminated soils, Part 2: Preparation of leachates - Zero headspace procedure**



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# Draft

## Australian Standard

Public Comment is invited for:

DR AS 4439.2:2019, *Wastes, sediments and contaminated soils, Part 2: Preparation of leachates — Zero headspace procedure*

Public Comment period:

Start date: 28 February 2019

Close date: 02 May 2019

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During their development process, Australian Standards are available in draft form during the public consultation period to allow any interests concerned with the application of the proposed Standard to review the draft and submit their comments.

This draft is liable to alteration. It is not to be regarded as an Australian Standard until finally issued as such by Standards Australia.

Upon successful conclusion of the Public Comment period it is proposed to publish this Standard as AS 4439.2:201X.

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## Conditions for comment

Comments are welcome on the technical content, wording and general arrangement of the draft. How the requirements of this draft coordinate with other Standards is of particular importance and you are invited to point out any areas where changes or additions to this draft may be necessary. Editorial matters (i.e. spelling, punctuation, grammar, etc.) will be corrected before final publication.

Please provide supporting reasons and suggested wording for each comment. Where you consider that specific content is too simplistic, too complex or too detailed please provide an alternative.

If the proposed Standard is acceptable for Australia without change, an acknowledgement to this effect would be appreciated.

If you know of other persons or organizations that may wish to comment on this draft Australian Standard, please advise them of its availability. Copies of drafts and other publications from Standards Australia are available from SAI Global at [www.saiglobal.com](http://www.saiglobal.com)

Only comments submitted via the Standards Australia Standards Hub site before midnight on the closing date will be reviewed by the committee. The Hub automatically submits comments to the committee. Any other communication will not be considered by the committee.

At the expiry of the comment period, the committee responsible for the document is obliged to give serious consideration to all comments received. However, normally no acknowledgement of comment is sent.

## Preface

This Standard was prepared by the Standards Australia Committee CH-035, Examination of Wastes, to supersede AS 4439.2—1997, and forms part of a series of standards prepared to provide a standardized method to determine the contamination potential of groundwater with inorganic and semi-volatile organic materials from liquid and solid wastes, soils, sediments and sludges.

The objective of this Standard is to provide a standardized method to determine the contamination potential of groundwater with volatile, organic materials from liquid and solid wastes, soils, sediments and sludges.

The major changes in this edition are as follows:

- (a) Option to use AS 4439.2 for potentially volatile Hydrogen Cyanide forming Cyanide species.
- (b) Additional information on use of this method for Per- and Polyfluoroalkyl Substances (PFAS) in [Clause 6.5](#).
- (c) Additional options for determination of percentage solids in [Clause 7.3](#).
- (d) Modified reporting requirements in [Clause 10](#) to allow for changes in [Clause 7.3](#).

The terms “normative” and “informative” are used in Standards to define the application of the appendix to which they apply. A “normative” appendix is an integral part of a Standard, whereas an “informative” appendix is only for information and guidance.

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# Australian Standard®

## Wastes, sediments and contaminated soils

### Part 2: Preparation of leachates—Zero headspace procedure

#### 1 Scope

This Standard provides a method for the preparation of leachates using zero headspace conditions, from liquid and solid wastes, sediments, sludges and soils for assessing the potential of volatile organic contamination of groundwater in a variety of disposal-to-land scenarios.

This Standard deals only with the preparation of the leachate. This Standard excludes procedures prior to submitting samples to the laboratory for analysis or sub-sampling.

The range of analytes includes any organic compound which is liquid or gaseous at normal temperatures and for which a suitable analytical method exists.

NOTE A list of analyte compounds is given in [Appendix A](#).

The use of zero headspace in this Standard may also be used for cyanide forms that may be susceptible to losses due to volatilisation when using pH < 7 leaching fluids (refer to AS 4439.3).

The procedure is not applicable to encapsulated wastes which cannot be reduced to the specified maximum particle size without breaking the integrity of encapsulation.

The pH and the oxidation-reduction (redox) potential, or  $E_h$  of a leaching fluid may vary with each disposal environment and is known to affect the leaching of metals and possibly some organic species. No provision is made in this procedure, however, to control pH and  $E_h$  during leaching.

#### 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document.

NOTE Documents referenced for informative purposes are listed in the Bibliography.

AS 1289-2.1.1, *Methods of testing soils for engineering purposes, Method 2.1.1: Soil moisture content tests — Determination of the moisture content of a soil — Oven drying method (standard method)*

AS 2162-1, *Verification and use of volumetric apparatus, Part 1: General — Volumetric glassware*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

Method 1313, Revision 1, July 2017, Proposed Update VI to the Third Edition of the Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, U.S. Environmental Protection Agency publication SW-846

#### 3 Terms and definitions

For the purpose of this Standard, the following terms and definitions apply.

##### 3.1

##### **blank test**

test performed without sample in the same manner as, and parallel with, a test using an analytical sample

[SOURCE: ISO 11323:2010, 8.13]

**3.2****laboratory sample**

sample as received by the laboratory from the field and intended for inspection or testing

**3.3****leaching fluid**

solution produced in the laboratory to be used for the leaching of the test sample

**3.4****may**

indicates the existence of an option

**3.5****percentage solids**

that amount of a test portion remaining after all liquids have been expelled by pressure filtration under a pressure of 350 kPa, expressed as a percentage

**3.6****sample**

one or more items taken from a lot and intended to provide information about the lot and, possibly, to serve as the basis for a decision on the lot or on the process which has produced it

**3.7****sample liquid**

liquid which is separated from a test sample by pressure filtration as described in [Clause 7.3](#)

**3.8****shall**

indicates that a statement is mandatory

**3.9****should**

indicates a recommendation

**3.10****solids leachate**

liquid remaining after treatment of the solids with the leaching fluid and subsequent filtration

**3.11****test portion**

quantity of material taken from the test sample (or, if both are the same, from the laboratory sample) and on which the test or observation is actually carried out

**3.12****test sample**

sample prepared from the laboratory sample and from which test portions will be taken

**3.13****zero headspace**

in the absence of a gaseous phase above the liquid or solid in a container

**4 Principle**

The percentage solids are determined by pressure filtering a weighed test portion and weighing the extracted sample liquid. A second test portion is reduced in particle size if necessary, subjected to pressure filtration and the sample liquid is collected and stored under zero headspace conditions. The solids remaining are leached by agitation with a selected leaching fluid under zero headspace conditions and the solids leachate recovered by filtration through a glass fibre filter with 0.6 µm to 0.8 µm effective pore size. The sample liquid and solids leachate are then analysed by appropriate test methods.



## 5 Reagents

### 5.1 General requirement

Unless otherwise specified, all reagents shall be of analytical reagent grade. All containers shall meet the requirements of [Clause 6.1](#).

### 5.2 Reagent water

Water of laboratory Grade 1 in accordance with ISO 3696 shall be used.

### 5.3 Leaching fluid

#### 5.3.1 General

The leaching fluid shall be tested immediately before use to ensure conformity to the specified pH value. Leaching fluids not prepared immediately prior to use shall also be inspected for indication of contamination or deterioration. In the presence of microbial growth, colouration, solid or suspended matter, the solution shall be discarded and fresh leaching fluid prepared.

#### 5.3.2 Leaching fluid pH 5.0

Leaching fluid pH 5.0 shall be prepared as follows:

- Add 5.7 mL of glacial acetic acid ( $\rho_{20} = 1.01 \text{ g/mL}$ ) to 900 mL of water.
- Add 64.3 mL of 1 mol/L sodium hydroxide solution dilute to 1 L with water and mix well.
- Where the pH is not 4.9 to 5.0, adjust by dropwise addition of 1 mol/L acetic acid or 1 mol/L sodium hydroxide solution as appropriate.
- Store in an airtight container.

#### 5.3.3 Leaching fluid pH 9.2

Leaching fluid pH 9.2 shall be prepared as follows:

- Dissolve 38.2 g of sodium tetraborate decahydrate (borax) in approximately 900 mL of water.
- Dilute to 1 L with water and mix well. The pH of this fluid shall be 9.1 to 9.3 otherwise it shall be discarded. The pH shall not be adjusted.
- Store in an airtight container.

#### 5.3.4 Compressed gas

Pressure controllable within the range 0 kPa to 350 kPa, and free of particulate and organic contaminants.

## 6 Apparatus

### 6.1 General

All devices with which the sample or leaching fluids come into contact shall be made of a material(s) which will not leach or adsorb analytes, such as:

- glass;



- (b) polytetrafluoroethylene (PTFE);
- (c) High Density Polyethylene (HDPE) for Per- and Polyfluoro Alkyl Substances (PFAS) determinations (or other fluorine free plastics);
- (d) stainless steel Type 316; or
- (e) polyethylene, polypropylene or polyvinyl chloride when only inorganic analytes will be determined.

These components shall be cleaned so that they do not contribute significantly to the level of analyte being determined.

## 6.2 Agitation apparatus

Agitation apparatus shall be capable of rotating the leaching vessel(s) in an end-over-end fashion at  $30 \pm 2$  r/min.

## 6.3 Filters

Filter discs to suit devices specified in [Clause 6.6](#) and [Clause 6.7](#) and made of borosilicate glass fibres, not containing binder materials and with an effective pore size of  $0.6\mu\text{m}$  to  $0.8\mu\text{m}$  or equivalent, are suitable.

NOTE The filters used should be assessed for the potential retention of PFAS analytes (due to chemical adherence). Where  $> 10\%$  retention is suspected, then an alternative method is to centrifuge the resulting leachate and analyse the supernatant. This deviation should be recorded on the analytical report.

## 6.4 Glassware

Glassware used shall be as follows:

- (a) Graduated measuring cylinders shall conform with ISO 4788.
- (b) Volumetric glassware shall conform with AS 2162.1.

## 6.5 Laboratory balance

A laboratory balance shall be accurate to  $\pm 0.1$  g.

## 6.6 Leaching fluid reservoir

When pressurized by a gas cylinder or mechanical means, the leaching fluid reservoir shall be capable of transferring a measured mass of leaching fluid to the ZHE leaching vessel while maintaining zero headspace within the vessel.

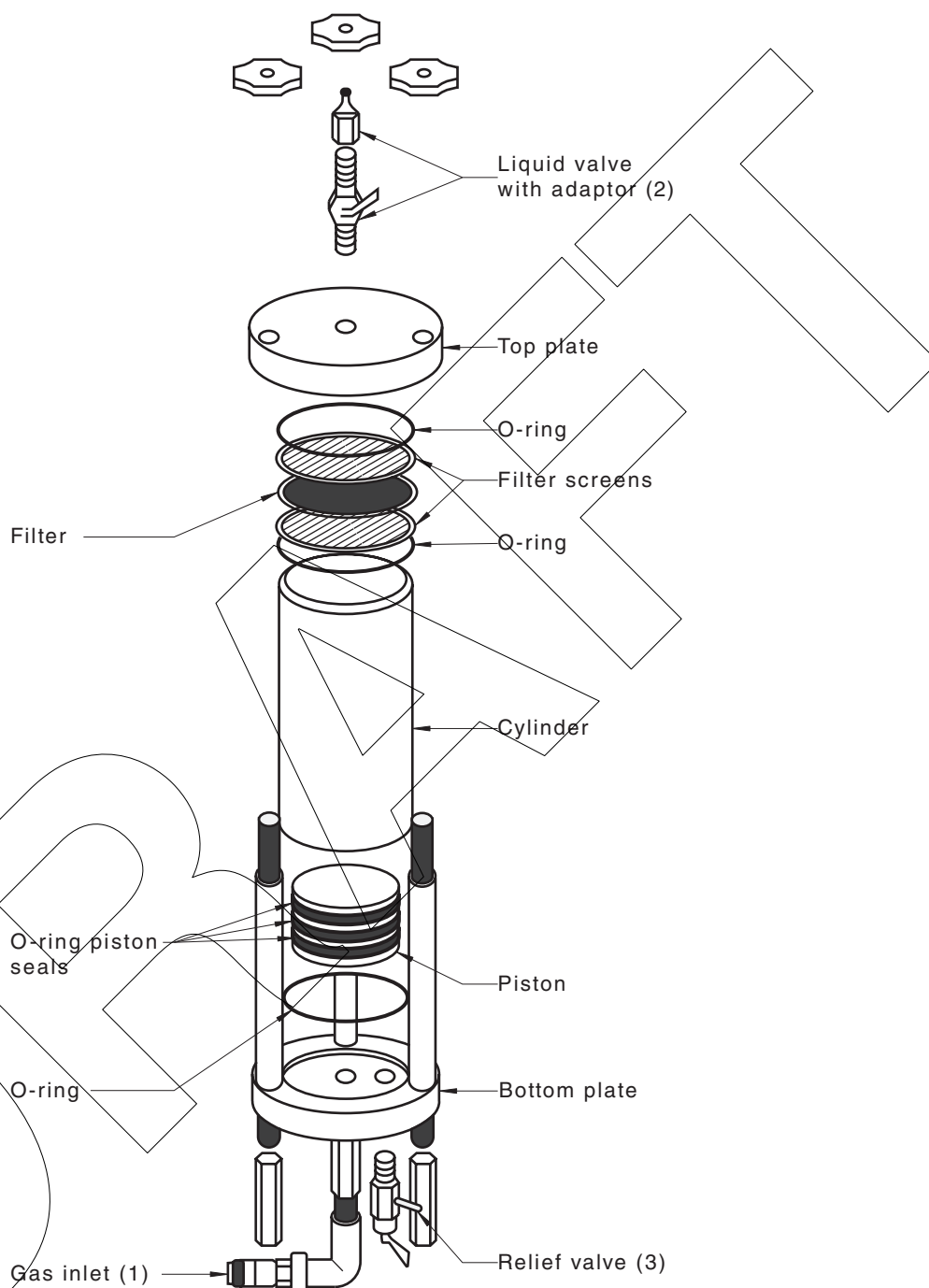
## 6.7 Leaching vessel

A leaching vessel shall be as follows:

- (a) Constructed of 316 grade stainless steel and PTFE.
- (b) An internal volume of 500 mL to 600 mL.
- (c) Capable of accommodating a filter.
- (d) A zero headspace extraction (ZHE) design, i.e. shall provide for variable internal volume by means of a moveable piston activated by gas pressure to allow nil internal headspace above the enclosed liquid.

- (d) Piston shall provide a means of removal of the enclosed liquid without exposure to the external atmosphere, via a filter with retention of solids within the vessel.

NOTE An example of a leaching vessel of suitable design is shown in [Figure 1](#).



**Figure 1 — Example of a zero headspace extractor**

## 6.8 Liquid collection devices

Sample liquid and solids leachate shall be collected in one of the following containers:

- (a) Polyvinyl fluoride bags fitted with appropriate sample introduction and outlet valves.

- (b) Gastight glass, PTFE or stainless steel syringes, with a capacity sufficient to hold a volume of leachate sufficient to support the analyses required, including any replicate analysis necessary.

NOTE Lubricants should not be used.

## 6.9 Mortar and pestle

The mortar and pestle shall be prechilled to less than 4 °C.

NOTE Other less aggressive apparatus may be selected to obtain particle size reduction provided the apparatus selected minimizes the loss of volatile components.

## 6.10 Plastic push rod

Plastic rod of the same or slightly narrower diameter to the stainless steel cylinder of the leaching vessel.

## 6.11 pH meter

The pH meter shall be accurate to  $\pm 0.05$  pH units at 25 °C.

## 6.12 Pressure filtration device

A pressure filtration device shall be as follows:

- (a) Any filter holder capable of supporting a 0.6 m to 0.8 m glass fibre filter membrane and of at least 47 mm diameter, and able to withstand a pressure of 350 kPa or more.
- (b) Devices shall have a capacity of at least 300 mL.

NOTE 1 An example of a suitable pressure filtration device is shown in [Figure 2](#).

NOTE 2 The recommended device has a capacity of 1.5 L and 142 mm diameter.

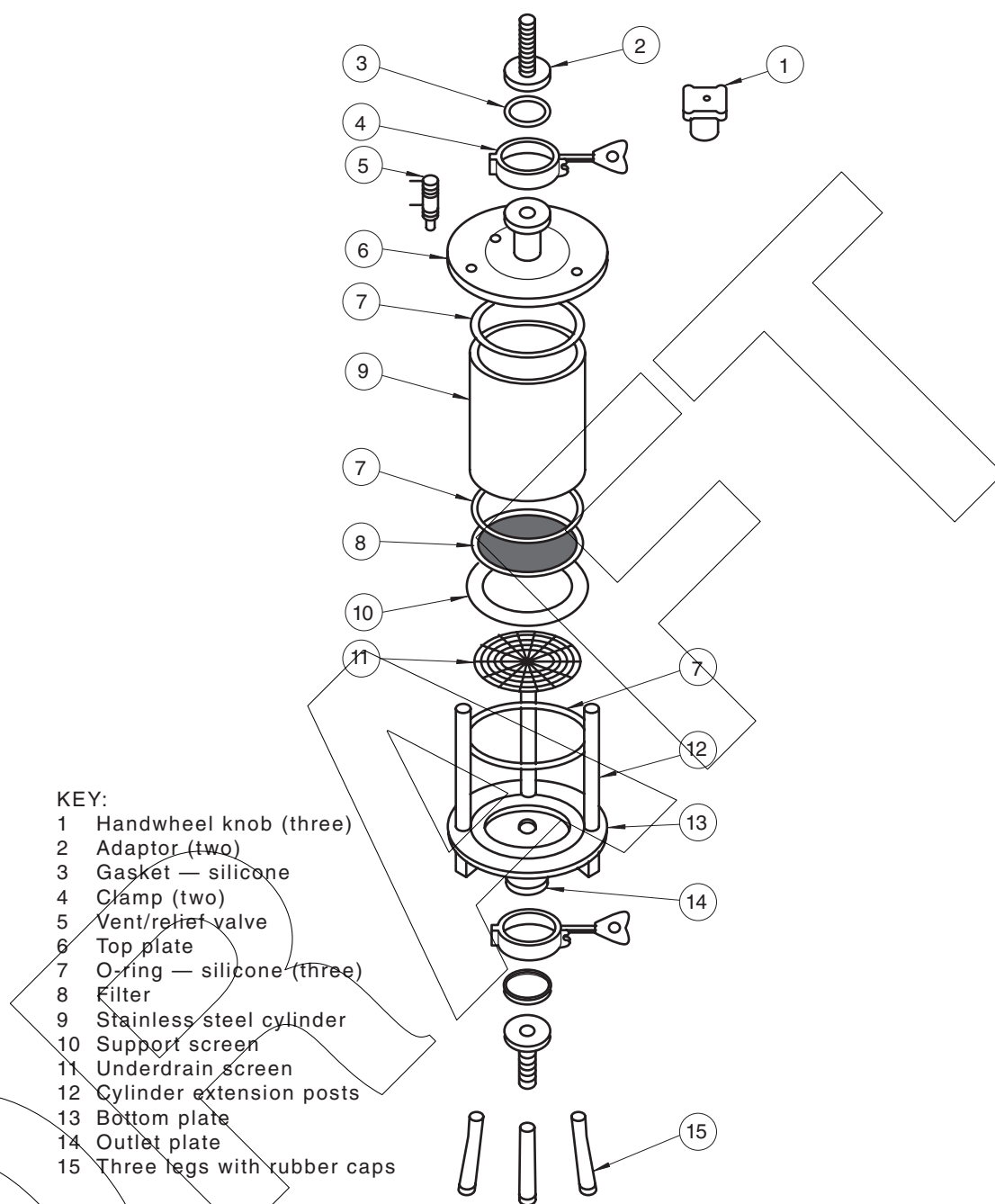


Figure 2 — Example of pressure filtration device

## 7 Preparation of test samples

### 7.1 Collection and storage

Samples shall be large enough to support all the requirements of this method. There shall be sufficient sample to perform, where required, the following:

- (a) Preliminary determination of total analytes if necessary.
- (b) Preliminary evaluation of the solids content.
- (c) Leaching of solids for volatile organic compounds.

- (d) Leaching of solids for metals if required.
- (e) Leaching of solids for semi-volatile compounds if required.
- (f) Any repeat analysis.

**WARNING — SAFETY PRECAUTIONS AS DEFINED IN AS 2243.1, AS 2243.2 AND AS 2243.3 SHOULD BE TAKEN.**

Sufficient sample solids shall be present to provide enough leachate to support each of the necessary analyses, taking into account the limits of detection of the various analytical methods used. Additionally, enough sample shall be collected to support any repeat analyses which may be required.

Leaching shall be carried out as soon as possible after receipt of the samples. The leaching shall be completed within 7 days.

NOTE Samples should be refrigerated until testing is completed unless refrigeration results in irreversible physical change.

Where precipitation occurs, the entire sample, including the precipitate, shall be extracted. Preservatives shall not be added to samples before leaching.

Samples shall be collected and stored in a manner to prevent loss of volatile components, i.e. in polytetrafluoroethylene (PTFE) lined screw-capped glass containers, filled to capacity, and opened only immediately prior to preparation for leaching.

## 7.2 Reduction of sample particle size

Due to the size of the leaching vessel, the possibility of damage to its inner wall, and in order to minimize sampling error, the particle size of the test sample (consisting of at least 250 g), should be reduced to 2 mm to 3 mm. This may be confirmed visually by comparison with a ruled scale, or by screening the material.

Fragments larger than 4 mm may be removed before particle size reduction if they —

- (a) comprise less than 10 % by mass of the sample;
- (b) are appreciably different in character from the bulk of the sample (e.g. stones, fragments of twigs); and
- (c) are not expected to contribute significantly to the leaching of analytes.

If the sample consists mainly of large particles, or if the larger particles are not significantly different to the bulk of the material, then the whole test portion should be reduced to a maximum particle size of 2 mm to 3 mm in such a way as to minimize any loss of analytes of interest.

The sample, mortar and pestle (see [Clause 6.11](#)) shall be chilled to less than 4 °C prior to the particle size reduction. Care shall be taken during the particle size reduction to prevent the generation of heat. The particle size reduction should be performed as quickly as possible to reduce the loss of analytes.

## 7.3 Determination of percentage solids

The determination of percentage solids shall be required —

- (a) where the sample is free flowing with visible free liquid then percentage solids is determined as below; *OR*
- (b) where the sample is > 20 % moisture (moisture may be determined using AS 1289.2.1.1 or Section 11.2 of USEPA SW846 1313, 2017) then pressure filtration (i.e. percentage solids determined as below) is required; *UNLESS*
- (c) the sample has significant clay content that inevitably would not give up liquid under pressure.

If the percentage solids is not determined, assume that the percentage solids = 100 % for the purposes of the calculation in [Clause 8.5\(a\)](#).

Where percentage solids determination is required the percentage solids in the sample shall be determined as follows:

- (i) Weigh the vessel that is to receive the filtrate ( $m_1$ ).
- (ii) Weigh approximately 100 g of the test sample into a beaker or similar vessel, and record the combined weight.
- (iii) Rapidly transfer as much of the test sample as possible to the pressure filtration device (see [Clause 6.6](#)), fitted with a filter (see [Clause 6.5](#)). Weigh the beaker and calculate the mass of waste transferred ( $m_2$ ) by difference.

**WARNING — SUITABLE CONTAINMENT PROCEDURES SHALL BE IN PLACE TO ISOLATE FLYING PARTS IN THE EVENT OF OVERPRESSURE EXPLODING THE DEVICE.**

- (iv) Apply a pressure of 50 kPa to the pressure filtration device, and maintain it until liquid ceases to pass through the filter. Gradually increase the pressure, in 50 kPa increments, to 350 kPa.
- (v) When filtration is complete (i.e. when no liquid has passed in the previous 1 min period), weigh the receiving vessel plus filtrate ( $m_3$ ).

NOTE If the sample liquid contains volatile solvents, then either the receiving vessel is designed to prevent loss of volatiles (e.g. narrow neck flask) or the pressure filtration device is weighed before and after to determine the mass of liquid collected.

- (vi) Calculate the percentage solids from the following [Equation 1](#):

$$\text{Percentage solids} = 100 - \left[ \frac{m_3 - m_1}{m_2} \times 100 \right]$$

where

$m_3$  = mass of the receiving vessel plus filtrate, in grams

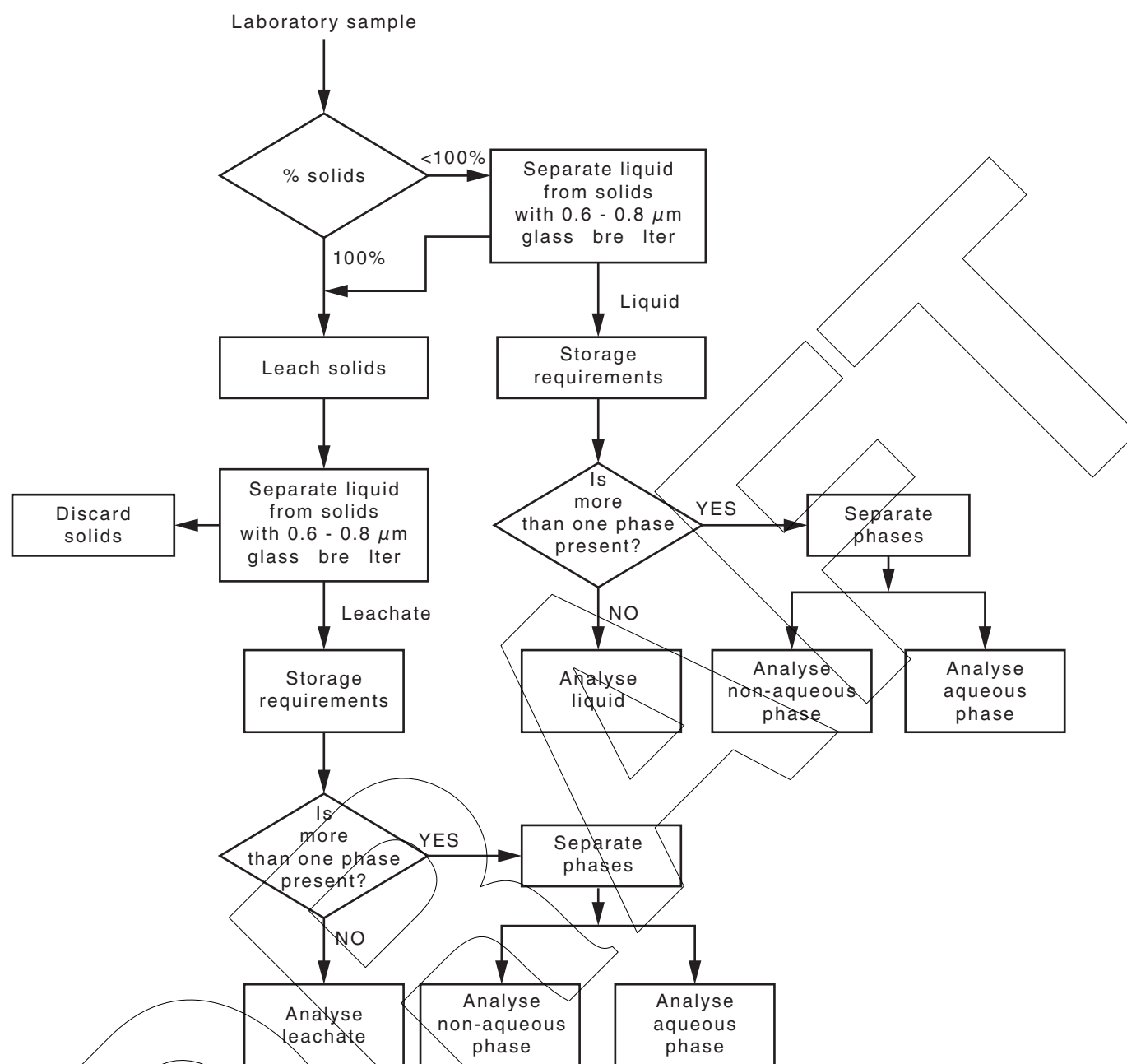
$m_1$  = mass of the empty receiving vessel, in grams

$m_2$  = mass of sample transferred, in grams

## 8 Procedure

### 8.1 Introduction

A flowsheet outlining the leaching procedure is shown in [Figure 3](#) below.



**Figure 3 — Leaching procedure flowsheet**

## 8.2 Selection of leaching fluid

The leaching fluid selected should relate to the appropriate landfill category (see [Table 1](#)).



**Table 1 — Selection of leaching fluids**

Class	Landfill category	Leaching fluid
Class 1	<i>In situ</i> — to be left undisturbed at the site	Reagent water
Class 2	Monofilled	
	2a Putrescible material	Acetate buffer pH 5
	2b Non-putrescible material	Reagent water
Class 3	Co-disposed with	
	3a Putrescible material	Acetate buffer pH 5
	3b Non-putrescible material	Acetate buffer pH 5 and tetraborate buffer pH 9.2 (i.e.: two elutions)
Class 4	Disposed of without confinement, e.g. dispersed over land	Reagent water

NOTE Other appropriate leaching fluids, such as local groundwater or seawater, may be used in place of those above. Use of groundwater as a leaching fluid may prove difficult as it would need to be maintained in the same chemical, physical and biological environment from which it was obtained up to, and during, the extraction process. It may be difficult to interpret analytical data if the complete chemical composition of the groundwater is not known due to analytical interferences and chemical speciation/precipitation reactions.

### 8.3 Number of determinations

A single test portion from each test sample shall be leached, using the procedure specified in [Clause 8.5](#). In addition, duplicate test portions shall be leached from selected test samples at a rate not less than one duplicate leach in 10 test portions or one duplicate leach for each batch of samples, whichever is the greater rate (where sufficient sample mass is supplied).

### 8.4 Blank test

A blank test shall be run with each batch or at a rate of at least one in every 20 samples processed through each ZHE unit. The blank test shall be run in parallel using the same procedure as the analysis with the reagents in the same quantity but omitting the sample.

NOTE A blank test should also be determined where there is a possibility of contamination.

If the contribution by the ZHE unit to the analyte blank is greater than 20 % of the appropriate limit, the ZHE shall be cleaned, the seals replaced or new reagents prepared, as required.

### 8.5 Preparation of the zero headspace extractor for use

The procedure for preparing a zero headspace extractor (see [Figure 1](#)) shall be as follows:

- Inspect all components of the ZHE apparatus for signs of contamination. Remove O-ring seals and remove all extraneous debris from the O-ring seal grooves, piston walls and other surfaces. Clean only with a mild detergent and soft brushes.

NOTE Stubborn residues on stainless steel surfaces may be removed with non-chlorinated, non-scratch cleaner. All components capable of fitting into an ultrasonic bath may be ultrasonically cleaned.

- Flush and wash all components with reagent water (see [Clause 5.2](#)) and dry.
- Inspect the seals and O-rings. These components shall be washed in mild detergent and water, rinsed with reagent water (see [Clause 5.2](#)) and air-dried. Where the O-rings and seals are damaged or contaminated in any way they shall be replaced.
- Wet the O-rings and seals with leaching fluid and fit to the piston plunger.

NOTE For the ZHE to be acceptable for use, the piston with the ZHE should be able to be moved with 1 kPa or less. Where it takes more pressure to move the piston, either the O-rings are dry or they should be replaced and the walls of the vessel examined for scores or other damage which may increase the friction between the moving parts.

- (e) Place the stainless steel cylinder in accordance with [Figure 1](#) and [Figure 2](#) on a clean dry surface and press the piston plunger down into the top of the bore using a clean plastic push rod, until the piston plunger is flush with the outside edge of the stainless steel cylinder.
- (f) Rinse the bore with leaching fluid, then fit the cylinder and plunger onto the lower plate assembly, while ensuring that the lower O-ring seal is aligned and correctly seated.
- (g) Insert a glass fibre filter between two stainless steel retaining screens and install in the top plate assembly. Use the top cylinder O-ring to hold the retaining screens in place.

## 8.6 Leaching of samples

The procedure for preparing sample liquid and solids leachate shall be as follows:

- (a) Weigh out a test portion, as determined from [Equation 2](#) below, into a beaker or similar vessel and record the weight:

$$\text{Mass of test portion, in grams} = \frac{25 \times 100}{\text{percentage solids}} \quad 2$$

For materials of low solids content and normal density, the volume of sample with mass calculated above may exceed the capacity of the leaching vessel (approximately 500 mL). A test portion of 500 mL total volume shall be used in this case. However, the sample shall contain more than 5 g of solid to provide a minimum of 100 mL of solids leachate for analysis. Where a 500 mL volume sample will not support all of the required analyses, replicate leaching shall be performed.

- (b) Immediately pour material into a ZHE vessel (see [Clause 6.7](#)) fitted with a filter (see [Clause 6.5](#)).

NOTE Material that adheres to the beaker can be ignored as it has been accounted for in [Equation 2](#). In this process, time is of the essence to minimize the potential for analyte loss due to volatilisation.

- (c) Weigh the beaker and calculate the mass transferred ( $m_4$ ) by difference.
- (d) Place the top plate assembly onto the cylinder and, after ensuring correct alignment, screw down the hand wheel knobs to evenly compress the seal.
- (e) Attach a compressed gas line to the fitting on the gas inlet [see [Figure 1\(1\)](#)].
- (f) Open the liquid valve [see [Figure 1\(2\)](#)] and gradually apply pressure under the piston until the piston begins to move.

NOTE This can be detected by placing a piece of tubing over the liquid valve [see [Figure 1\(2\)](#)] with the free end of the tube under water, and looking for the evolution of gas bubbles.

- (g) Slowly raise the piston by gradually increasing the pressure applied under the piston.
- (h) At the first appearance of liquid at the liquid valve [see [Figure 1\(2\)](#)], shut off the valve.
- (i) Remove the tubing and attach an evacuated, pre-weighed liquid collection device (see [Clause 6.8](#)) to the liquid valve.
- (j) Open the liquid valve [see [Figure 1\(2\)](#)] and the valve on the liquid collection device and apply a pressure of 70 kPa and hold this pressure for 2 min, then increase by 10 kPa increments.
- (k) Continue increasing the pressure until the total pressure reaches 350 kPa.

- (l) Hold the pressure at 350 kPa until no further liquid flow is detected during any 1 min period; then close all valves, shut off the gas pressure and disconnect the liquid collection device from the ZHE and reweigh.
- (m) Calculate the mass of liquid collected ( $m_5$ ) by difference.

- (n) Remove a small portion of the collected sample liquid and measure and record its pH. Store the remaining liquid prior to analysis as described in [Clause 8.5\(ac\)](#) below.

NOTE At this stage, if redox potential is to be measured in accordance with ASTM D1498-14, a small portion of the leachate should be removed for this purpose and the measurement made within 5 min of the portion being withdrawn.

- (o) Calculate the mass of leaching fluid required to be added to the solids from the following [Equation 3](#):

$$\text{Mass of leaching fluid, in grams} = 20 \times (m_4 - m_5)$$

where

20 = ratio of leaching solution to final solids

$m_4$  = mass of test sample transferred, in grams

$m_5$  = mass of sample liquid collected, in grams

- (p) Rinse out the leaching fluid reservoir (see [Clause 6.6](#)) with leaching fluid, then refill with the correct mass of leaching fluid [see [Clause 8.5\(o\)](#)]. Pressurize the vessel and flush the transfer line until no air bubbles remain in the line.
- (q) Attach this transfer line to the liquid valve [see [Figure 1\(2\)](#)]. Apply pressure to the transfer vessel, open the liquid valve [see [Figure 1\(2\)](#)] and allow the transfer of the correct mass [see [Clause 8.5\(o\)](#)] of leaching fluid into the ZHE. Open the relief valve [see [Figure 1\(3\)](#)] to allow the piston to be driven down by the incoming leaching fluid.
- (r) Close the liquid valve [see [Figure 1\(2\)](#)] of the ZHE and disconnect the transfer line.
- (s) Check all valves are closed and then rotate the ZHE end-over-end two or three times.
- (t) Connect a gas line to the gas valve of the ZHE and apply 50 kPa pressure.
- (u) Slowly open the liquid valve, allow gas to escape and close the valve immediately when liquid appears. Close the gas valve on the ZHE and disconnect the gas line. Ensure there are no leaks.

NOTE Confirmation of the leaching fluid sample mass ratio can be made by weighing the ZHE before and after filling with leaching fluid.

- (v) Place the ZHE in the rotary agitation apparatus (see [Clause 6.10](#)), counterbalanced by another ZHE or counterbalance weight.

**CAUTION — UNLESS CORRECTLY COUNTERBALANCED, THE ROTATION APPARATUS WILL BE SUBJECT TO SEVERE VIBRATIONS, WHICH WILL CAUSE WEAR OF THE BEARINGS, OVERHEATING OF THE MOTOR, AND MAY CAUSE THE APPARATUS TO MOVE ALONG THE BENCH ON WHICH IT STANDS. IF A SECOND ZHE IS USED AS A COUNTER BALANCE, IT MAY CONTAIN WATER OR EXTRACTION FLUID, WITH OR WITHOUT ANOTHER SAMPLE. HOWEVER, IT IS DESIRABLE TO MATCH THE WEIGHT OF THE ZHE AND ITS COUNTERBALANCE AS CLOSELY AS POSSIBLE, PREFERABLY TO WITHIN ONE GRAM.**

- (w) Rotate the ZHE for  $18 \pm 2$  h at  $30 \pm 2$  r/min at an ambient temperature in the range of  $22 \pm 5$  °C.

- (x) Remove the ZHE from the rotary agitation apparatus (see [Clause 6.10](#)) and check that the ZHE is still pressurized by referring to a pressure gauge, if fitted. Alternatively, place tubing over the relief valve [see [Figure 1\(3\)](#)] with the other end immersed in water. Open the gas valve — there should be bubbling to indicate residual air pressure below the piston.

NOTE If the pressure under the piston has not been maintained then the procedure shall be aborted and the test procedure recommenced using a fresh test portion and after cleaning and changing the seals on the ZHE.

- (y) Evacuate a liquid collection device, close the valve on it and weigh the device.
- (z) Attach the valve section of the liquid collection device to the liquid valve [see [Figure 1\(2\)](#)].
- (aa) Connect a gas line to the gas inlet [see [Figure 1\(1\)](#)].
- (ab) Open the valve on the liquid collection device, the liquid valve [see [Figure 1\(2\)](#)].
- (ac) Apply gas pressure of 50 kPa below the ZHE piston. At 2 min intervals apply a further 50 kPa increments in pressure to a maximum pressure of 350 kPa.
- (ad) Hold at 350 kPa until no further liquid has passed into the liquid collection device in any 1 min period.
- (ae) Close the valves of the ZHE and the liquid collection device.
- (af) Remove the liquid collection device, reweigh and store the collected leachate in the collection device at 4 °C for not more than 7 days before analysis. It should however, be analysed as soon as possible after its preparation.

NOTE Where redox potential is to be measured in accordance with ASTM D1498-14, a small portion for this determination should be removed and the pH and  $E_h$  measured within 5 min of the sample being withdrawn. Store the remaining leachate in the leachate collection device as described above, prior to analysis.

- (ag) Depressurize the ZHE and then disassemble it.
- (ah) Thoroughly clean the ZHE as specified in [Clause 8.4](#).
- (ai) Store until the ZHE is required again.

## 9 Analysis and calculation

All sample liquid and leachate phases shall be analysed separately using appropriate analytical methods for the required analytes. The results shall be reported separately (see [Clause 10](#)).

NOTE With multiphasic samples, more than one phase may result from either the sample liquid(s) or leachate.

A mass averaged value shall also be reported with the separate phase results. For each analyte, this is calculated using [Equation 3](#) below:

$$c_{\text{calc}} = \frac{\sum (c_i \times m_i)}{\sum m_i}$$

3

where

$c_{\text{calc}}$  = calculated mass averaged value for the analyte in sample liquid(s) and leachate, in milligrams per kilogram

$c_i$  = concentration of analyte measured in the sample liquid or leachate phase  $i$ , in milligrams per kilogram

$m_i$  = mass of sample liquid or leachate phase  $i$ , in grams

As an aid to interpretation of results, it is recommended that the  $E_h$  of the extracted sample liquid and the solids leachate be measured and reported.

## 10 Test report

The following information shall be reported:

- (a) Identification of the laboratory sample as received.
- (b) Date of receipt.
- (c) Leaching fluid used.
- (d) pH of the sample liquid and solids leachate.
- (e) Concentration of analytes in the sample liquid (if requested for multiphasic samples) and solids leachate.
- (f) Date of leachate preparation and date of analysis.
- (g) A reference to this Standard, i.e. AS 4439.2.
- (h) Moisture content (as determined by AS 1289.2.1.1 or Section 11.2 of USEPA SW846 1313, 2017 or similar methodology at  $105 \pm 5^\circ\text{C}$ ).

Additionally for multiphasic samples only, include the following:

- (i) Percentage solids in the laboratory sample.
- (ii) Mass of sample liquid expelled.

Further, the laboratory should report the following if variance from [Clause 7.1](#) occurs:

- (A) Details of storage of the laboratory sample.
- (B) Details of any sample preparation carried out.
- (C) Mass of test sample.

Other information that may be reported are as follows:

- (1) Mass of leaching fluid added.
- (2) Mass of solids leachate collected.
- (3)  $E_h$ , if measured, of the sample liquid and solids leachate.
- (4) Any observation, in relation to the sample or the performance of the method, which may affect the correct interpretation of the ensuing analytical results.

NOTE Item (1) to Item (4) are optional unless requested on the sample submission.



## Appendix A (informative)

### Purgable volatile organic compounds

This Appendix lists those volatile organic compounds that may be regarded as significant pollutants. It does not preclude other volatile compounds from being termed the same, however this list may need to be assessed in consultation with the appropriate regulatory authority at the time of implementation of this Standard.

Acetone	Dichlorobenzene <i>o</i> -, <i>m</i> -, <i>p</i> -	Methylene chloride
Acrolein	Dichlorobromomethane	Pyridine
Acrylonitrile	Dichlorodifluoromethane	Styrene
Benzene	1,1-Dichloroethane	1,1,1,2-Tetrachloroethane
Bromoform	1,2-Dichloroethane	1,1,2,2-Tetrachloroethane
Butanone	1,1-Dichloroethylene	Tetrachloroethylene
Carbon tetrachloride	1,2- <i>trans</i> -Dichloroethylene	Toluene
Carbon disulphide	1,2-Dichloropropane	1,2,4-Trichlorobenzene
Chlorobenzene	1,3-Dichloropropane	1,1,1-Trichloroethane
Chlorodibromomethane	Ethyl benzene	Trichlorofluoromethane
Chloroethane	Isobutanol	Vinyl chloride
Chloroethyl vinyl ether	Methyl bromide	Xylenes
Chloroform	Methyl chloride	
<i>bis</i> (Chloromethyl) ether	Methyl ethyl ketone	

## Bibliography

AS 4439-3, *Wastes, sediments and contaminated soils, Part 3: Preparation of leachates – bottle leaching procedure*

AS/NZS 2243.1, *Safety in laboratories, Part 1: Planning and operational aspects*

AS/NZS 2243.2, *Safety in laboratories, Part 2: Chemical aspects*

AS/NZS 2243.3, *Safety in laboratories, Part 3: Microbiological safety and containment*

ASTM D1498-14, *Standard Test Method for Oxidation-Reduction Potential of Water*, ASTM International, West Conshohocken, PA, 2014. Available from: [www.astm.org](http://www.astm.org)



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