Manual of geochemical leaching trials 2010–2014

Brukunga Mine Remediation Program Phase 3

June 2015
Report Book 2015/00019

Mine Completion Program

Government of South Australia
Department of State Development

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Phase 3

Mine Completion Program
Mining Regulation and Rehabilitation Branch
Resources and Energy Group

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Resources and Energy Group 2015. *Manual of geochemical leaching trials 2010-2014 Brukunga Mine Remediation Program Phase 3*, Report Book 2015/00019. Department of State Development, South Australia, Adelaide.

Cover photo

Geochemical testing at the Brukunga lab. (Photo 414502)

Job number 204638

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EXECUTIVE SUMMARY

The Brukunga mine site is a former open pit iron sulfide mine that continues to contribute to Acid and Metalliferous Drainage (AMD) to downstream waterways. Sources of AMD include the waste rock dumps and tailings storage facility.

In 2007 the Brukunga Mine Remediation Program commenced an assessment of long-term remediation options to rehabilitate the mine site. The current design involves the subaqueous disposal of co-disposed waste rock and tailings with a cover system to prevent ongoing oxidation of sulfidic materials.

To confirm the geochemical performance of the proposed design, a series of kinetic and geochemical tests were conducted to test various waste composition scenarios. The trials consisted of combining, compacting and saturating waste materials and limestone in various proportions at various scales and testing leachate and water cover chemistry over time.

The trials attempt to de-risk the remediation options and demonstrate its effectiveness plus identify any issues arising during the trials that would require further testwork. The aim of this manual is to report on the construction of leachate trials conducted at Brukunga between 2010 and 2014, when the following testwork was carried out:

- Bench test column leach trials
- Bin scale column leach testwork
- Field based geochemical test cell

The appendices provide further background to the monitoring, sampling and testing procedure carried out during the trial work. The many personnel involved over time are also acknowledged and include:

- Technical Advisory Group (TAG – geochemical)
- Brukunga mine site personnel
- Laboratory services
- Department of State Development (DSD) Brukunga team
- Vacation students

1. INTRODUCTION

1.1 BACKGROUND

The Brukunga mine site (located 4 km north of Nairne in the Mt Lofty Ranges) represents an ongoing source of acid and metalliferous contamination to downstream waterways. Open pit mining of iron sulfide minerals (pyrite and pyrrhotite) between 1955 and 1972 has left a legacy of acid and metalliferous drainage (AMD) largely associated with two large waste rock dumps (WRD) and a tailings storage facility (TSF).

The South Australian Government took responsibility for the site in 1977 and commissioned a water treatment plant in 1980 to improve water quality in Dawesley Creek. Further remediation has included diverting the creek in an attempt to contain and treat acid drainage on the mine site and increasing the capacity of the water treatment plant.

1.2 REMEDIATION STRATEGY

The Brukunga Remediation Project Technical Advisory Group (TAG) is a group of national and international AMD specialists who have proposed a long-term remediation plan for the mine site (TAG 2009). The design involves placement of co-disposed waste rock, tailings and limestone into two pits on the mine bench behind an engineered cut-off wall. Permanent saturation of the compacted co-disposed waste materials is designed to eliminate acidity generation caused by sulfide oxidation. The incorporated limestone aims to neutralise stored acid salts within the pore spaces.

1.3 GEOCHEMICAL TESTWORK AT BRUKUNGA

Various tests, trials, research and investigation have been carried out at Brukunga since 2007, focusing on the geochemistry of mine waste at the Brukunga mine site (Fig. 1). Geochemical tests and trials were designed to demonstrate and refine the saturated co-disposed mine waste model proposed for the Brukunga remediation design. Trials carried out prior to 2010 included waste rock pile testwork and sulfate leach / oxygen consumption testwork.

The purpose of this manual is to document the methodology for construction, running and monitoring of geochemical leach trials at various scales that were carried out at Brukunga from 2010 to 2014. This can assist with future planning of repeat trials for characterising the geochemistry of mine waste remediation. Testwork was scaled from laboratory/bench tests to field-base/pilot testing.

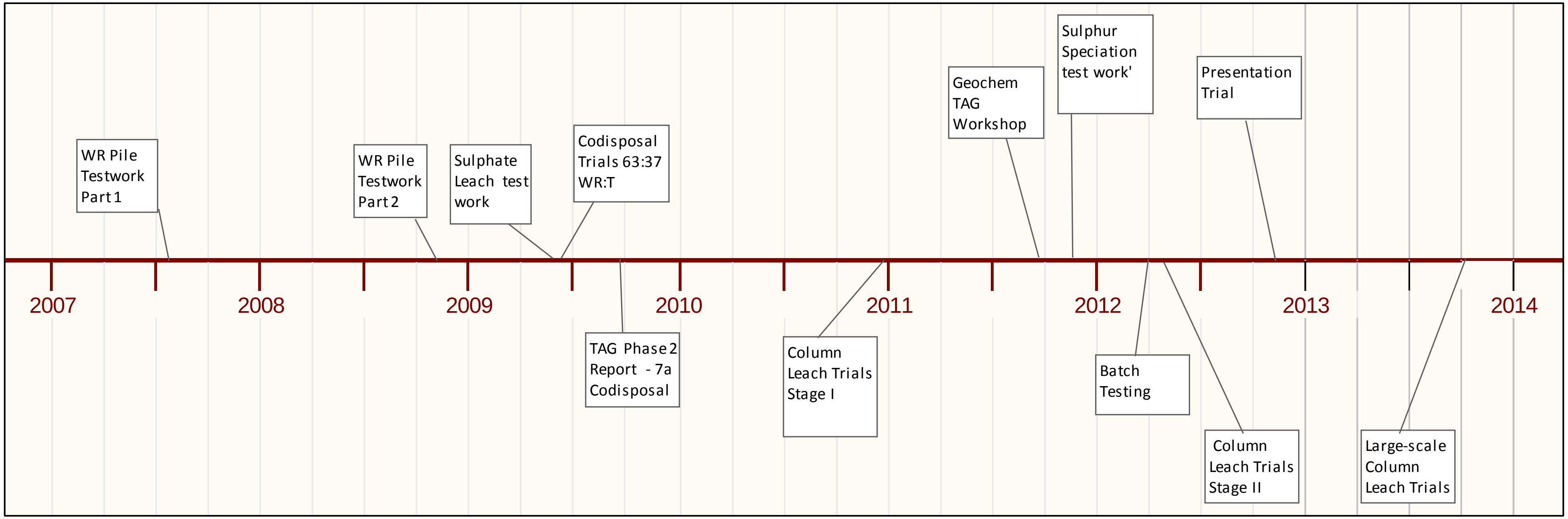


Figure 1. Timeline of geochemistry testwork at Brukunga 2007–2014. WR = waste rock; T = tailings; TAG = Technical Advisory Group.

2. PRELIMINARY WORK

2.1 COLLECTION OF WASTE MATERIALS FOR TRIALS

Collection of waste materials for geochemical kinetic testwork conducted at the Brukunga mine site between 2010 and 2014 was supervised by the Brukunga Technical Advisory Group (TAG). The following testwork (from smaller to larger scale) was carried out during this period:

- Bench test column leach trials (~25 kg solids in 200 x 1000 mm columns)
- Large-scale column leach trial (~400 kg solids in 200 litre bins)
- Presentation trial (100 tonne geochemical cell)

This represents an up-scaling from laboratory to pilot testing (field scale) studies. A larger scale trial was conducted by the TAG prior to 2010 and involved the construction of seven test piles (pyramids) each of ~1000 tonnes of waste rock material (Note: An even larger scale 10 000 tonne geotechnical trial has also been carried out in 2014).

Potential Acid Forming (PAF) materials at the Brukunga mine site include waste rock from the waste rock dump (WRD) and tailings from the Tailings Storage Facility (TSF). PAF materials for the geochemical trial testwork were excavated from preferred sites based on previous test drilling and geochemical analysis. Tailings were excavated (Fig. 2) from a 2 m depth pit located at groundwater monitoring site T01 in the TSF. Tailings included a mix of oxidised (pale brown to orange) and less oxidised (grey) materials below 1 m depth.

Figure 2. Excavation of tailings in February 2012 showing pale yellow brown oxidised tailings and grey un-oxidised materials below 1 m depth. (Photo 414503)

Waste rock was sourced from the Waste Rock Test Pile #3 (pyramid #3) on the bench below the mine wall (Fig. 3). This material was originally sourced from the northern side of the Southern WRD as being reasonably representative of waste rock on the mine site. Test Pile #3 was a site of previous geochemical trial testwork (see Appendix 6.10) that had not been subjected to any alkaline treatment and was effectively a control for the earlier set of trials.

Sub-samples were collected for sulfur speciation test work which was undertaken to characterise PAF materials. A methodology for excavation and collection of waste materials including a sampling plan for sulfur speciation of waste rock and tailings was provided by Scott (2012).

Figure 3. Excavation of waste rock from Pyramid #3 with collection of sub-samples for sulfur speciation testwork. (Photo 414504)

2.2 GEOCHEMICAL CHARACTERISATION

Sulphur speciation testwork (geochemical static testing) was carried out in 2011 to fully characterise the sulphur speciation of drillhole materials in mine wastes (tailings and waste rock). The recommended lab for analysis was Levay & Co. Environmental Services.

Samples of waste rock (<25 mm grain-size) and tailings materials were characterised for the following parameters:

- Gravimetric moisture content
- Total sulfur
- Chromium reducible sulfur
- KCl-extractible sulfur
- HCl-extractible sulfur

- Acid Neutralisation Capacity (ANC)
- Net Acid Generation (NAG)

Sulphur speciation testwork was also done to characterise all materials to be used in the geochemical trials (batch extraction testing, stage I and II column leach trials, 100 t presentation trial).

- Tailings samples were submitted as two separate sub-samples (0–1 m and 1–2 m) from a pit excavated to 2 m in the TSF.
- Waste rock samples with particle size distribution (PSD) -200 mm taken from pyramid #3, were to be used for the 100 tonne demonstration trial. Waste rock samples to be used for the Stage II column leach trial were screened to PSD -25 mm.
- NAF material (concrete sand, small river pebbles and mixed river pebbles) for the 100 tonne presentation trial were also tested. The small river pebbles were rejected as suitable NAF material due to a NAG pH of 3.9.

Measurement of PSD for source materials was also undertaken. Finer materials will be biased towards greater concentrations of AMD salts and acidity, reactivity, and surface area — on a per unit mass basis. Hence, small scale column tests (which cannot accommodate larger particle sizes) will tend to over-estimate the acidity per kg. Knowledge of PSDs are needed to convert the acidity per unit mass of a finer fraction that is tested to the acidity per unit of total mass of interest.

Analytical methods used for determining static geochemical parameters (sulphur speciation) is provided in a handbook (Amira International 2002) and summarised in Earth Systems (2013a and b).

2.3 BULK LEACH / BATCH EXTRACTION TESTWORK

Bulk leach (bottle-roll) testwork was carried out in March 2012 by On Site Technology Pty Ltd. The aim being to quantify the amount of limestone necessary to neutralise the total mass of acidity that jarosite could potentially generate within the co-disposed mix. The outcome of this testwork identified an optimum ratio to effectively neutralise acidity in the co-disposed mix (Earth Systems 2013a,b).

Bottle-roll tests (Fig. 4), provide an ideal continuous mixing environment for the limestone – co-disposed waste mixtures. The advantage of this method is that it speeds up the chemical reactions (especially dissolution of jarosite), hence the optimum amount of limestone addition required to fully neutralise the jarosite can be estimated. The process represents an ideal fully mixed system.

Figure 4. Bottle-roll leach testwork carried out at On Site Technology Pty Ltd. (Photo 414505)

Waste materials used for this leach testwork were taken from the following samples as recommended by the TAG (EGi, 2012):

- Waste Rock (Drillhole W03, 12.5–13.3 m and -6.7 mm sieved fraction only)
- Tailings (Drillhole T01, 14–15.5 m)
- Limestone (Penrice Ag Lime)

A 63:37 waste rock:tailings mix screened to 5 mm was added to each PVC tube with five treatments — limestone weight percentages of 1, 2.5, 4, 6 and 8 per cent were trialled. De-ionised water was used for the mixing and extracting solution and two leaching rates were used (10 and 30 L/kg/day). This gave a total of 5 x 2 = 10 bulk leach tests conducted and was run over 35 days. Five large and five small diameter PVC tubes were used as tumblers (Fig. 4). Tubes were filled from a third to half full of solid material and are therefore highly agitated, even at a tumbler speed of 10 rpm.

There was approximately 3 litres of water in the large diameter tubes and 1.5 L in the small diameter tubes with leachate being extracted daily and tested for the following parameters:

- pH, EC and TDS
- Acidity
- Hydroxide alkalinity
- Carbonate alkalinity
- Bicarbonate alkalinity
- Total alkalinity
- Chloride and sulphate

3. LABORATORY-SCALE COLUMN LEACH TRIALS

3.1 INTRODUCTION

Kinetic geochemical testwork involves measuring chemical parameters over time and is used to identify the likely rate of acidity or metals release over time due to oxidation of sulfide minerals and assess likely leachate chemistry (Davis et al. 2014). For example, column leach trials provide a laboratory-scale approach to assessing the effectiveness of acidity neutralisation while assessing the effects of variables such as grain size and concentration of jarosite and limestone. Test columns can simulate a fully saturated co-disposed waste materials with an:

- Overlying water cover / supernatant (providing a guide to surface water quality).
- Under-drain system (providing a guide to pore water quality and to indicate potential groundwater contamination issues).

Leachate trials can be conducted at various scales ranging from bench test trials conducted in the laboratory to pilot scale field trials. Larger column sizes can be utilised to examine scale-up factors and further up-scaled to field-based test pile trials. Kinetic leach tests need to operate for at least six months but can extend to one or two years before sufficient data are available to interpret AMD characteristic of materials (DITR 2007).

The construction of 200 mm leach columns using blended waste materials was conducted over a number of years at Brukunga. Stage I column testwork commenced in November 2010 and stage II commenced in April 2012.

STAGE I COLUMN LEACH TESTWORK (NOV 2010 TO APRIL 2012)

This initial testwork was designed to investigate the effectiveness of a permanent water cover on preventing sulfide oxidation and to investigate the effectiveness of 1 wt.% limestone in neutralising acidity in the co-disposed mix.

While the water cover was able to prevent further sulfide oxidation, it also caused the jarosite (a secondary acid salt) to dissolve and acidify both the leachate and the water cover. Further testwork was considered necessary to resolve the jarosite issue via greater additions of limestone to the co-disposed mix.

STAGE II COLUMN LEACH TESTWORK (APRIL 2012 TO AUGUST 2012)

Bulk leach (bottle-roll) testwork (Section 2.3) was carried out to quantify the amount of limestone necessary to neutralise the total mass of acidity that jarosite could potentially generate. This testwork identified an optimum ratio of 60 wt.% waste rock, 36 wt.% tailings and 4 wt.% $CaCO_3$ to effectively neutralise acidity in the co-disposed mix. Stage II column leach tests were then carried out to determine whether the ideal optimum ratio would effectively neutralise leachate and supernatant acidity. A full report is provided by Earth Systems (2013a and b).

3.2 LEACH COLUMN CONSTRUCTION METHODOLOGY

Stages involve sample preparation, column set up and loading followed by field testing.

The following materials are required:

- Perspex tubing (190 mm ID)
- Waste rock, tailings (characterised and assayed for sulfur forms, ANC and NAG)
- Limestone (Penrice Ag lime)
- Filter sand (Rocla glass sand)
- Creek water (from Dawesley Creek)
- Fittings for columns (taps, tubing, collection bags)
- Perspex lids for base and top of columns

- Weighing balance
- Clean plastic buckets
- Plastic wheelbarrow tub
- Spades and trowels

SAMPLE PREPARATION

The weight of solids to be loaded into each column needs to be determined. This involves measuring the wet and dry moisture content of a sub-sample of the materials. To obtain a dry weight, samples need to be oven dried. Soil moisture data is entered into a spreadsheet (see example in Appendix 6.6) to calculate the weight of solids in the correct ratio. The methodology for mixing waste materials with other additives can be done as follows:

- Determine the total dry mass required to achieve 1800 kg/m^3 target compaction, within the volume of the tube that needs to be filled.
- Calculate the dry mass of crushed limestone required to achieve the required wt.% of $CaCO_3$, (correcting for moisture and purity).
- Determine the corresponding ‘wet’ mass of limestone to be measured into the mixing bucket, by factoring in the antecedent moisture content.
- Allocate the remaining dry mass in the ratio 63:37 between waste rock and tailings.
- Determine the corresponding ‘wet’ (moisture containing) masses of materials that will be measured into the mixing tub.

Each type of material is placed into a clean plastic bucket, weighed and then tipped into a plastic wheelbarrow tub for thorough mixing using a clean shovel.

COLUMN PREPARATION

The majority of the leachate trails were constructed in a 1 m length of clear acrylic Perspex 190 mm internal diameter (ID) tubing, sealed at the base with a flat piece of Perspex. A tap valve is installed at the base of the column (Fig. 5) to control effluent discharge. It is a good idea to check that the column does not leak by filling it with water and leaving it stand for a day.

Each new column needs to be labelled with a unique ID number and the depth to which materials need to go are marked on the side of the tube:

- 0–5 cm basal filter layer
- 5–50 cm solid materials
- 50–90 cm water cover

The internal base of the columns requires a filter system. This can be done by placing a small diameter (40–50 mm OD) perforated PVC tube on the base aligned with the outlet tap (Fig. 5) and covering the perforated tube with a fabric filter mesh.

COLUMN LOADING

A filter layer of dry inert acid-washed pure quartz sand (Rocla Glass Sand ©) provides a porous and non-reactive collection medium. The filter layer at the base of the column (around 5 cm depth) should just cover the perforated PVC tube (Fig. 5). The mixed material is slowly loaded with minor compaction/consolidation to achieve a dry bulk density close to 1.8 t/m^3 (1800 kg/m^3). This can be achieved by adding the material in 10 cm lifts up to the 50 cm mark on the column and lightly compacting using a ram-rod with a flat end (Fig. 6). The material should be moist (~15%) but not too wet. Any left-over material can be used for additional testing (e.g. bulk density, sulfur speciation, paste pH etc.). Appendix 6.4 provides a standard operation procedure (SOP) for conducting a paste pH test.

Figure 5. Adding a filter layer of sand at base of new column. (Photo 414506)

A 40 cm constant head water cover is then added to saturate the materials in the column. To prevent erosion to the top of the materials, water can be riffled using a cushioning device placed on top of the solid material. Once the water level has stabilised at the 90 cm mark on the column, a loose fitting Perspex plate is placed on the top of the column to prevent evaporative loss.

Tables 1 and 2 provide physical and geochemical specifications for both stage I and stage II column leach testwork.

Table 1. Physical and geochemical specifications for column leach stage I testwork.

Focus	Column ID	Description
Standard design	ST1, ST2 ST3	Standard waste rock and tails mix with 1wt.% $CaCO_3$ 3 x replicates
Limestone quantity	L1 L2	Standard waste rock and tails mix with 0.5wt.% $CaCO_3$ Standard waste rock and tails mix, no added limestone
Ratios of waste materials	R1 R2	Waste rock with 1wt.% added $CaCO_3$ Tails with 1wt.% added $CaCO_3$
High sulfur	S1 S2	High sulfur waste rock and tails mix with 1wt.% $CaCO_3$ 2 x replicates
Oxidised tailings	O1	Standard waste rock and oxidised tails mix with 1wt.% $CaCO_3$
Staged build	SB	Standard waste rock and tails mix with 1wt.% $CaCO_3$ and built in 3 x ⅓ layers at intervals of 2–3 months
Treatment mix water	T1 T2	Standard waste rock and tails mix with 1wt.% $CaCO_3$ and adding treatment plant alkaline discharge water to the mix
Wet lime addition	WL1 WL2	Standard waste rock and tails mix with a pre-mixed slurry of 1wt.% $CaCO_3$
Surface water head	H1 H2 H3	1 cm surface water head 10 cm surface water head 100 cm surface water head (2 m tall column)
Acid groundwater added	A1	Standard waste rock and tails mix with 1wt.% $CaCO_3$ with injection of acidic water
Surface drying	D1	Standard waste rock and tails mix with 1wt.% $CaCO_3$ and built in 3 x ⅓ layers and subject to drying of surfaces
Worst case construction	WC	50:50 waste rock and tails mix, no added limestone
Excess flushing	Tall1,2,3	Rapid flushing of multiple pore volumes

Table 2. Physical and geochemical specifications for column leach stage II testwork.

No.	Column ID	Description
1	Mix-0	Standard waste rock and tails mix, no added limestone
2	Mix-2	Standard waste rock and tails mix with 2wt.% $CaCO_3$
3	Mix-4	Standard waste rock and tails mix with 4wt.% $CaCO_3$
4	Mix-4A	Standard waste rock and tails mix with 4wt.% $CaCO_3$ (with mid-level irrigation pipe)
5	Mix-6A	Standard waste rock and tails mix with 6wt.% $CaCO_3$ (with mid-level irrigation pipe)
6	WR-2	Waste rock (<25 mm) with 2wt.% added $CaCO_3$
7	WR-4	Waste rock (<25 mm) with 4wt.% added $CaCO_3$
8	WR(4)-2	Waste rock (<4 mm) with 2wt.% added $CaCO_3$
9	WR(4)-4	Waste rock (<4 mm) with 4wt.% added $CaCO_3$
10	WR(1.2)-2	Waste rock (<1.2 mm) with 2wt.% added $CaCO_3$
11	WR(1.2)-4	Waste rock (<1.2 mm) with 4wt.% added $CaCO_3$
12	T-2	Tails with 2wt.% added $CaCO_3$
13	T-4	Tails with 4wt.% added $CaCO_3$

Figure 6. Loading column and compacting solid material. (Photo 414507)

3.3 MONITORING AND SAMPLING

Columns should be allowed to stand for at least one week to allow secondary acid salts (e.g. jarosite) to be fully neutralised by the limestone before any testing of leachate commences.

For the purpose of mimicking groundwater conditions (with minimal exposure to air), leachate was collected in sealed medical drainage bags with a built-in tap for leachate sampling (Fig. 7). Leachate samples are allowed to freely drain from the column until 500 to 1000 mL are collected in the bags. Depending on permeability (which may be a function of compaction), the time taken to collect a sample may vary from less than an hour to half a day.

Two 90-degree pipe fittings were installed after the tap (at the base of the columns) for the purpose of raising the discharge water level in order to keep the bottom sand layer saturated. If this feature was not installed, leachate would drain more quickly from the sand layer than what could permeate through the column, resulting in the potential for sulfide oxidation at the base of the column.

Pore water chemistry is determined by both field testing and laboratory analysis. Water analysis includes a standard suite of metal assays, major cations and anions, pH, redox, EC and temperature measurements to enable geochemical modelling. PIRSA (2011) determined that this procedure would apply to all extraction and leaching tests (including batch testing, column leach trials and 100 t presentation trial for both co-mix and cover material).

FIELD TESTING

Field testing involves in situ field measurements and subjective visual observations of colour and odor for both the leachate and the tube surface water cover.

Sampling of the tube surface water involves collecting a sample from the near surface using a syringe. The syringe should be rinsed with the surface water three times and then withdraw 20 mL of the supernatant. Measurement of field water quality parameters (see Appendix 6.2 for standard operation procedures) is then taken as soon as possible and all volume consumptions should be recorded.

Leachate sampling requires leachate to be collected into drainage bags. Leachate is allowed to drain freely from the column until a desired amount (500 to 1000 mL) is collected in the bag. Once the leachate is discharged into a beaker, measurement of field parameters need to be taken as soon as possible with all volume consumptions being recorded.

It is recommended to use dedicated syringes for each column and separate ones for surface water and leachate water and each should be appropriately labelled. The following parameters are tested on a weekly basis:

- pH
- ORP
- EC
- Acidity

Waters, Pape and Taylor (2014) note that calculation of Total Acidity may be more accurate than laboratory-derived acidity measurements since metal concentrations are usually measured to a much greater accuracy than Total Acidity.

Upon completion of testing and sampling, each column is irrigated with a volume of creek water equivalent to the volume of water removed for sampling of supernatant and leachate. This should return the depth of water back up to the 90 cm mark on the column.

Figure 7. **Collecting leachate from stage I columns into drainage bags. (Photo 414508)**

LABORATORY ANALYSIS

Water samples are collected for laboratory analysis at a NATA accredited laboratory and analysed for the following parameters.

- pH
- EC
- Acidity and alkalinity
- Cations: Ca, K, Mg and Na
- Anions: Cl, SO_4, F
- Dissolved metals: Al, As, Cd, Cr, Cu, Fe, Pb, Mn, Ni and Zn

Sample bottles are supplied by the laboratory and need to be clearly labelled along with a Chain of Custody (COC) form to be filled out prior to delivery. Generally two plastic bottles are provided for sampling and includes a 500 mL bottle for physical parameters and a 60 mL bottle for total or dissolved metal analysis. Samples need to be kept chilled and are stored in an esky with ice or ice bricks. The turn-around-time for delivery needs to be observed for parameters analysed.

Two sets of duplicate samples are required from each parent sample to satisfy QA/QC requirements. This includes:

- Intra-laboratory duplicate – a field duplicate analysed as a ‘blind’ sample by the main laboratory performing the analysis and is done each time.
- Intra-laboratory duplicate - a field duplicate sent to a second laboratory as an independent check and done monthly if sampling weekly.

3.4 RECOMMENDATIONS

Leaks occasionally develop around tap seals and on a few occasions the drainage bags have failed – usually because taps have been left on for too long and the bags have over-filled. A maximum volume of 1.5 L is recommended for collection in drainage bags.

The collection of leachate in some columns was very slow indicating that they may have been over-compacted in some cases. Only light tamping should be done when compacting the solid material and it should not be too moist.

Supernatant and leachate acidity were determined by the following methods:

- Field-based acidity titration of water sample following collection.
- Laboratory analysis of leachate sample by titration.
- Estimated acidity calculations based on pH and dissolved metal concentrations.

Earth Systems (2013) observed that significant discrepancies were apparent between the three methods of determining acidity. Field and laboratory acidity values were substantially elevated above the actual calculated value which was put down to interference from Ca and Mg ions. They determined that calculated acidity represented the best reflection of actual values.

An important issue identified by the TAG was that field testing of stage I and stage II columns commenced too early. Enough time should be allowed for the lime to effectively neutralise jarosite in the waste rock material. Early sampling resulted in a first flush of high acidity and high salinity leachate.

4. LARGE-SCALE COLUMN LEACH TESTWORK

4.1 INTRODUCTION

This test work up-scales from the 200 mm Perspex columns holding around 25 kg of solid material to 200 litre heavy-duty plastic bins holding around 400–500 kg of solid material.

This trial was preceded by the 100 Tonne Presentation Trial (which had a design flaw), whereby reductive dissolution of jarosite [$KFe(OH)SO_4$] and ferrihydrite [$Fe(OH)_3$] resulted in leachate with significantly elevated iron (Fe) and sulfate (SO_4) concentrations. The large-scale column leach tests were set up to see if leachate quality could be improved in the presence of dissolved organic carbon (Earth Systems 2013c).

The benefits of adding organic matter to co-disposed waste are that it helps to form reducing systems whereby metals and Fe are removed by the process of ‘sulfidisation’ and to encourage pyrite (FeS) precipitation to occur. Clogging and armouring of carbonate will be less of an issue if Fe is kept soluble. Outcomes would therefore provide assessment of the risk of using construction materials containing organic carbon.

4.2 METHODOLOGY

Five heavy duty plastic bins (240 L) were used as large-scale columns and set up as indicated in Table 3.

Table 3. Large-scale column leach trial configuration.

Bin No.	Configuration
1	Baseline (PAF + NAF sourced from 100T Presentation Trial)
2	Baseline + uniformly distributed organic matter in PAF
3	Baseline + basal layer of organic matter
4	Baseline (PAF sourced from pyramid 3 and TSF)
5	Control (NAF only)

Baseline = 600 mm of PAF overlain by 350 mm of NAF within the large columns (bins).
PAF = Potential Acid Forming materials comprising 60 wt.% waste rock (WR) and 36 wt.% tailings (T).
NAF = Non Acid Forming materials comprising 60% concrete sand (0.1–0.5 mm) and 40% mixed river pebbles (50–60 mm).
TSF = Tailings Storage Facility.

The heavy duty plastic bins have the following specifications:

- Nominal volume = 240 L
- Internal height = 990 mm (0.99 m)
- Basal dimensions = 460 x 430 mm (0.46 x 0.43 m)
- Top dimensions = 570 x 510 mm (0.57 x 0.51 m)

PIPES AND FITTINGS

A basal drainage pipe was fitted to each of the five bins. The pipe comprised of 25 mm PN18 pressure pipe with two rows of 2 mm slots spaced at 60–70 mm. The pipe was placed as near as possible to the base of the bins. A filter mesh sleeve of 100 µm nylon was securely stitched with nylon thread around the slotted drainage pipe (Fig. 8). A tap and pipe fittings were attached on the exterior of the bin to allow leachate to be readily collected and field tested in-situ.

Figure 8. Drainage pipe in large-scale column, wrapped in 100 µm filter mesh. (Photo 414509)

The five bins were mounted on wooden pallets within an old greenhouse, thereby protected them from extremes of heat by the shade cloth. Each bin has a lid which remains closed except when irrigating from the nearby water tank. The bins are angled on a slight gradient to prevent a water sump collecting at the base of the bins (Fig. 9).

LOADING THE COLUMNS

PAF materials

As indicated in Table 3, five bins were loaded with co-disposed waste materials sourced from the dissembled 100T Presentation Trial as well as new waste materials sourced from pyramid 3 and the TSF.

For Bins 1, 2 and 3, PAF material was excavated from the centre of the discontinued Presentation Trial and stockpiled into a trailer. The wet weight of PAF material added to 600 mm depth was measured to be approximately 215 kg and was not compacted during loading. Larger stones (>150 mm) were excluded from the bins.

Figure 9. Loading PAF material into a large-scale column (plastic bin). (Photo 414510)

For Bin #4, new PAF material was sourced from pyramid 3 (see Section 2.1) and from the TSF. The new PAF material was mixed as follows in a wheelbarrow with plastic tub:

- 8 x shovels of WR sourced from pyramid 3.
- 6 x shovels of tailings sourced from the TSF.
- 1 x shovel of Penrice Aglime.

This approximates to a mixing ratio of 60 wt.% WR : 36 wt.% T : 4 wt.% $CaCO_3$. The mixing procedure was repeated until the bin was filled to the 600 mm mark. The material was not compacted or wetted during loading into the bin.

Organic Matter

Organic matter was added to bins 2 and 3. The selected material to be used was Spent Mushroom Compost sourced from Adelaide Mushrooms Nominees Pty Ltd based at Monarto near Murray Bridge. A Material Safety Data Sheet (MSDS) is available and includes a Typical Analysis.

For Bin #2, mushroom compost (5 wt.%) was mixed evenly into the PAF material. This was done by measuring out 10.7 kg of compost and 215 kg of PAF. The compost and the PAF were mixed in a wheelbarrow before loading into the bin.

For Bin #3, mushroom compost was compacted as a layer at the base of the bin with enough to cover the drainage pipe (Fig. 10). This amounted to approximately 100 mm depth of compost and required a little over a 30 L bag of compost (~13 kg).

Figure 10. Compacting organic material in large-scale column. (Photo 414511)

NAF material

The NAF material for each bin was originally sourced from Pooraka Sand and Metal Landscape Supplies and included:

- Mixed river pebbles (50–60 mm).
- Concrete sand (0.1–0.5 mm).

The materials were well mixed in a ratio of 60% sand to 40% pebbles.

Each bin was filled to within 40 mm to the top. The depth of NAF material amounted to 350 mm and had a total weight of around 200 kg.

Bin #5, acted as a control column and the entire bin was filled with NAF material.

SATURATION OF MATERIALS

Based on the dimension of the bins, the total volume occupied by PAF materials is 0.135 m^3 and the total volume occupied by NAF materials is 0.095 m^3. With an estimated porosity of 35% for PAF materials and 45% for NAF materials, the total volume of water required to fully saturate the materials is 90 L.

Each bin was fully saturated to the top of the NAF with water sourced from Dawesley Creek. A constant head water cover of 5–20 mm was maintained on top of the NAF.

4.3 MONITORING AND SAMPLING

Field testing (pH, ORP and EC) commenced in November 2013 and continued on a weekly basis. Leachate was tested immediately following extraction from the outlet port tap. The volume of leachate extracted for testing was 100 to 300 mL.

Field testing of acidity was not carried out as Waters et al. (2014) note that calculation of Total Acidity provides a more accurate and reliable indication of acidity than laboratory-derived acidity measurements.

Measurement of field parameters (see Appendix 6.2 for standard operation procedures) is then taken as soon as possible after sampling. The colour and odour (e.g. H_2S odour) of the leachate are also noted. An example of a field data monitoring sheet is shown in Appendix 6.8.

Upon completion of testing and sampling, each bin is irrigated with a volume of creek water equivalent to the volume of water removed for sampling.

4.4 RECOMMENDATIONS

Two of the bins developed leaks some weeks into the sampling program. The leaks were occurring at the base of the bins. It was unclear if this was an inherent flaw in the bins or if it a stress related crack resulted from the weight of the materials.

Bins should be tested for water tightness before loading materials. The materials (especially if containing stones and larger pebbles) should be carefully placed into the bins/columns.

5. 100 TONNE PRESENTATION TRIAL

5.1 INTRODUCTION

The construction of a 100 tonne geochemical trial at Brukunga was undertaken to validate the geochemical processes of the co-disposed mix by scaling-up from lab-columns to a full scale field trial constructed in a cement brick cell. The building of a besser-block field cell to house the waste materials took place in November 2011 and the trial was set up almost one year later in October 2012. The besser-block cell has internal dimensions of 12.13 m x 2.235 m x 1.58 m. Construction details were provided by Earth Systems (2012).

5.2 METHODOLOGY

Prior to construction, the interior of the cement brick cell was washed and cleaned. Because the walls of the cell had been constructed about one year previously, there were some visible cracks evident in the fibreglass resin and these were sealed with silicone cement. The inherent structural weakness was to become a major issue which severely compromised the outcomes of the trial.

Three 50 mm PVC tubes (with industrial slotting and wrapped in a 250 micron filter mesh) were laid lengthways along the base of the shell. It is recommended that Teflon tape be used on all threaded joins and 100 micron filter mesh is preferable. The tubes were bedded in an 80 mm layer of sand/gravel (fully characterised NAF material was used). In hindsight it would have been preferable to place an envelope of sand/gravel around each individual pipe (Jeff Taylor, Earth Systems pers comm.).

The bottom half of the structure was filled with PAF material and the top half with NAF material.

ADDITION OF PAF MATERIAL

A small excavator was used to excavate and mix PAF materials on the mixing pad adjacent to the stockpiles (Fig. 11).

Figure 11. Mixing waste rock, tailings and lime for 100 t presentation trial. (Photo 414512)

The source of the limestone used was Penrice Ag lime which has a purity of 93–97% $CaCO_3$. The mixing ratio was 60.5WR : 35.5T : 4L wt.% which equated to 8 x excavator buckets of waste rock, 6 x buckets of tailings and 1 x bucket of limestone. A large front end loader then transported the mixed material to be tipped into the test-shell (Fig. 12). Larger rocks (>250 mm) were excluded.

Figure 12. Dumping PAF mix into the geochemical test-shell, composed of besser blocks. (Photo 414513)

Ten loads of PAF mix were tipped into the structure to an internal depth of 0.80 m. Earth Systems (2012) indicated that around 39 tonnes of PAF material (assuming an average bulk density of 1.8 g/cm^3) would need to be added to bring it to the required height.

After each load of PAF material was dumped into the test-cell, it was evenly spread out using rakes and shovels. The material was wetted up (Fig. 13) by spraying with a hose connected to the 23 kL tank containing creek water and a pressure pump to aid delivery. The material was sprayed until the surface was glistening with moisture.

Figure 13. Wetting up and compacting the PAF mix within the geochemical test-cell. (Photo 414514)

A plate vibrator was run across the surface with just one pass and moving at a slow pace. Material was continued to be added and the wetting and compacting process repeated for each 10 cm lift.

ADDITION OF NAF MATERIAL

This material was sourced from Pooraka Sand and Metal Landscape Supplies and included:

- Mixed river pebbles (50–60 mm).
- Concrete sand (0.1–0.5 mm).

The materials were to be well mixed in a ratio of 60% sand : 40% pebbles prior to delivery. Because the material was stockpiled on the ground, care was required to avoid contamination when scooping up the material to be placed in the test-cell. It was estimated that over 40 tonne of NAF material was tipped into the enclosure to bring the NAF layer almost level to the top of the besser-brick wall. The NAF layer was wetted and compacted in the same manner as the PAF layer.

PIEZOMETER INSTALLATION

Following on from the three slotted pipes laid lengthways along the base of the test-cell, three series of piezometers were installed horizontally cross-ways at either end of the test-cell (Fig. 14). They were installed at the following depths:

- Middle of PAF layer (0.40 m from base).
- Interface of PAF and NAF (0.80 m from base).
- Middle of NAF layer (1.20 m from base).

Figure 14. Piezometers placed horizontally across the width of the test-cell. (Photo 414515)

Piezometer specifications were 50 mm PN18 PVC, machine slotted and wrapped in a 250 micron filter mesh. A 100 micron filter was recommended by the TAG but was unavailable from the supplier. The PVC tubes were levelled with a spirit level and were surrounded by a ~20 cm envelope of sand/gravel filter pack (using fully characterised NAF material). The nine slotted pipes formed the nine water collection ports for pore water analysis.

A channel to simulate Days Creek was formed at the surface of the NAF layer (Fig. 15). The channel was lined with slotted 150 mm diameter stormwater PVC pipe cut in half lengthwise through its circular cross section. Upon full saturation of the NAF layer, the depth of water in the channel was maintained at ~40 cm. A cover lid was placed on top of the shell to prevent lime dust from the treatment plant contaminating the materials.

Figure 15. **NAF cover surface with gutter to simulate a creek-line (note: 23 kL water storage tank on left and cover lid frame to the right). (Photo 414516)**

SATURATION PROCEDURE

Water for the trial was obtained from Dawesley Creek and stored in a 23 000 L tank with pressure pump (Fig. 15). The recommended strategy was to slowly wet up the mix to prevent oxidation from occurring. The NAF layer was initially wetted to just below field capacity to prevent oxygen from getting down into the PAF material. After 3–4 weeks, the shell was then flooded to its full capacity. This would give time for the residual acidity in the PAF layer to be neutralised by the limestone (expected to take ~4 weeks). The NAF layer would remain unsaturated until all the jarosite in the PAF had been neutralised. The NAF layer would then be saturated and this process would simulate ‘real-world’ rehabilitation.

Water was added to the shell at a rate of 11 L/min. The NAF material is fully saturated when free water appears in the surface channel. During wetting up it was noted that most of the piezometers made water during and immediately following the additions of water, but then ‘dried up’ a few days later. A number of irrigations was therefore required to maintain a constant head of water in the NAF.

At an estimated porosity of 25% in the PAF material it was considered that it would take 5000 to 6000 litres of water to fully saturate the PAF material. At an estimated porosity of 50% in the NAF material it would take around 11 000 litres of water to fully saturate the NAF layer. The total amount of water required to fully saturate the test-cell was calculated to be 16 000 litres, but in reality 20 000 litres was required.

Although not done in this trial it was suggested that for more accurate monitoring of the watertable, a pressure transducer (vibrating wire piezometer) should be used in preference to an open borehole (which could allow oxygen to enter the PAF layer).

5.3 MONITORING AND SAMPLING

Field testing for chemistry commenced around 2 weeks after construction to allow the limestone to neutralise the acidity of PAF materials in the test-cell. Laboratory sampling commenced a month after construction.

FIELD TESTING

Field testing involves in situ field measurements and subjective visual observations of colour and odor for the leachate collected from the sampling ports. Leachate samples are allowed to drain freely from the ports until a desired amount is collected (e.g. 100 mL). Taps need to be opened and closed quickly to prevent oxygen from entering the test-cell. Ports were not purged prior to taking a sample. The following parameters were tested on a weekly basis:

- pH
- ORP
- EC

Field testing of acidity was not carried out upon the recommendation of Earth Systems (J Taylor pers comm.). The calculation of Total Acidity provides a more accurate and reliable indication of acidity than laboratory-derived acidity measurements (Waters et al. 2014).

LABORATORY ANALYSIS

Water samples (500–1000 mL) are collected from selected ports for laboratory analysis at a NATA accredited laboratory. Field filtering was required for dissolved metals analysis. Samples were analysed for the following parameters:

- pH
- EC
- Acidity and alkalinity
- Cations: Ca, K, Mg and Na
- Anions: SO_4, Cl, F and NO_3
- Total N, TKN, Total P
- COD, TOC and DOC
- Dissolved metals: Al, As, Cd, Cr, Cu, Fe, Mn, Ni, Pb and Zn.

Upon completion of testing and sampling, the test cell is flooded to its full capacity with creek water. This was done by placing a delivery hose from the tank into the surface drain and pumping in water at a rate of ~10 L/minute.

HYDROGEOLOGICAL TESTING

Hydrogeological tests were conducted on the co-disposed waste materials to help establish baseline estimates of geotechnical parameters such as hydraulic conductivity. This involved flushing the porous base layer initially and then allowing leachate to drain freely from the middle basal port.

The test-cell was continually irrigated to maintain a hydrostatic head in the co-disposed materials. The discharge flow rate was measured at regular intervals along with field parameters of effluent leachate.

5.4 RECOMMENDATIONS

It soon became apparent that the besser brick structure/enclosure was not entirely waterproof but became increasingly leaky over time. The more water that was added to the shell the worse that the leaks became. Salt efflorescences and iron crusting appeared on the outside walls over the period of the trial and despite many attempts to seal the leaks, the problem continued to worsen.

The swelling and expansion of co-disposed waste materials when wetting up to saturation requires a robust design for the housing of these materials during experimental trials. The use of a heavy duty plastic tank-liner is recommended for containing leaks emanating from large brick geochemical test-shells.

6. APPENDICES

The following appendices are provided as a supplement to the geochemical trial work at Brukunga.

6.1 SAFETY PROCEDURES FOR FIELD AND LABORATORY LEACH TRIALS (ADAPTED FROM THE PIRSA OHS MANUAL 2010)

INTRODUCTION

The purpose of this document is to provide details on the safety procedures used in leachate trials at Brukunga mine field laboratory. This includes outlining the responsibilities of supervisors, employees, vacation students and other relative individuals while working for the program. This manual conforms and supports all OH&S directives and policies of the Primary Industries and Resources South Australia (now Department of State Development). All individuals involved in field column trials are required to be familiar with this procedures and policies outlined in this document.

GENERAL FIELD TRIALS SAFETY

Individuals must become familiar with the location of safety showers, washing facilities and first aid equipment in the field. Everyone in the field facility has a responsibility to protect the safety of themselves and for others working together. A hazards checklist of the field work should be checked prior to commencing any experimental work.

i. PPE (Personal Protective Equipment)

a. Safety boots must be worn at all the time in the field. Open shoes or sandals are NOT allowed. Closed laces up shoes are preferred
b. Safety glasses must be worn all the time, person wearing prescription glasses must have toughened impact resistant lenses; otherwise safety glasses must be worn
c. Clothing must be able to cover significant parts of body (e.g. long pants and shirt)
d. Wear appropriate gloves for protection all the time while handing hazardous chemicals.

ii. General rules

e. Keep the field laboratory tidy and clean at all times
f. Report to the project manager if you are the last person to use a solution and reagent, make sure it refilled for the next experiment
g. Report to the project manager if you break any apparatus (including glassware)
h. Walkway and floor space must be clear
i. Never smoke, eat, drink in the area where chemicals are placed and experiments are in progress
j. Don't leave experiments or samples unlabeled and unattended
k. Wash your hands thoroughly after the experiment.

CHEMICAL SAFETY

This section conforms and supports all OH&S directives and policies of the Primary Industries and Resources South Australia (PIRSA) : PIRSAFE Procedure No: 11 Hazardous Substances Management. A collection of Material Safety Data sheet (MSDS) provides information on hazardous chemicals and correct handing protocols and procedures to minimise risk. MSDS are available via ChemGold III database (PIRSA inside website). Before using any chemical the user must check with the database to confirm if the chemical has any hazardous properties and follow the handing methods listed in the database. For leachate testwork, there are three chemicals involved in the field experiment: Sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2) and Phenolphthalein ($C_{20}H_{14}O_4$) - see classification hazardous substance.

iii. General rules

a. All chemicals should be treated as being potentially hazardous
b. Refer to MSDS regularly and read the warning labels on unfamiliar chemicals
c. Label all containers
d. Unlabelled container will be considered waste and disposed if unclaimed at the end of the working day.

iv. Storage of chemicals

a. All chemicals shall be stored away from direct sunlight as sunlight can have detrimental effect on the integrity of some plastic
b. All chemicals shall be stored away from heating source
c. All quantities of chemicals stored within the acidity test kit are to be kept an absolute minimum for 2 months operation. (depend on the effective time of standard solution suggested by suppliers)
d. All chemicals should be sealed after every use, minimise the time exposing to air.

DISPOSAL OF EXPERIMENTAL WASTE

a. Small quantities of acid or alkali can be washed carefully down sink with excess volume of continuous flowing cold water
b. Waste acid water can be drained directly in the channel of wastewater treatment outside the field laboratory
c. Aqueous chemicals should be disposed in special bin provided in field laboratory
d. Broken glass should be disposed of in the designated glass bin
e. All other general waste should be disposed into the general bin in the field laboratory.

Chemical hazardous

- Material safety data sheets available and current
- Hazardous substance labelled
- Suitable containers
- Inventory of hazardous substances available and up to date
- All chemicals have been sealed and not leaking
- Volume of chemicals not excessive.

Waste storage

- Designated paper and plastic bin
- Designated bin for broken glass
- Chemical wastes segregated
- Regular waste disposal organised.

CLASSIFICATION HAZARDOUS SUBSTANCES AND CHEMICAL LABELS

The classification of chemicals is based on PIRSAFE Procedure No. 11 Hazardous Substances Management.

Name of chemcial	Class	Description
Sodium hydroxide	Low risk	See MSDS
Hydrogen peroxide	Moderate risk	See MSDS
Phenolphthalein	High risk	See MSDS

6.2 STANDARD OPERATION PROCEDURE — FIELD WATER QUALITY PARAMETERS

PURPOSE AND SCOPE

This Standard Operation Procedure (SOP) describes the measurement of field parameters for rapid testing of water quality in the field. A multi-parameter water quality meter is used to measure pH, Redox potential (ORP), electrical conductivity (EC), total dissolved salts (TDS) and temperature. Field measurements should represent as closely as possible the natural condition of the water at the time of sampling.

MATERIALS/EQUIPMENT/CHEMICALS

The following materials are equipment are required:

- paper towels, safety glasses and latex gloves
- distilled water / wash bottles
- beakers
- instruments (TPS and Myron Ultrameter)
- standard calibration solutions and buffer solutions

CALIBRATION OF INSTRUMENTS

Multi-parameter water quality meters are used for measuring field parameters at Brukunga. Parameters to be read are temperature, pH, ORP and Conductivity. It is recommended that TDS not be read as this is a calculated value and the factor varies with the quality of the water being measured.

Instruments in use at Brukunga are the TPS and Myron Ultrameter water quality meters. A calibration procedure is described below for both instruments.

It is a good idea to maintain a calibration tracking record (see Appendix 6.5) to keep a check on the reliability of the meter. Each instrument should have a permanent log book for recording calibration data and instrument repairs and services. Note that calibration is only required occasionally if the electrodes are in good condition. The sensors / electrodes have a limited lifetime and should be replaced at least annually.

TPS METER

Calibration of temperature

- Place a thermometer in a beaker of water and allow to stabilise for a couple of minutes
- Place conductivity / temperature probe in the same beaker
- Press MENU ☛ Press F1 cal ☛ F4 TEMP
- Record temperature on thermometer and TPS (pre-cal value)
- Enter temperature from thermometer as ACTUAL TEMPERATURE, press ENTER

Calibration of pH

- Calibrate probe with pH 4 and 7 standards (2-point calibration)
- Wash probe with distilled water, dry and then immerse probe in pH 4 standard solution
- Press MENU ☛ press F1 cal ☛ F3 pH
- Record pre-cal value, press ENTER, then record post-cal value
- Wash probe with distilled water, dry and repeat above procedure with pH 7 standard.

Calibration of conductivity

- Remove conductivity probe from case and wash with distilled water. Immerse probe in standard solution so that hole at the top of the probe is covered. Allow the reading to stabilise which may take a minute or more
- Press MENU ☛ press F1 cal ☛ F2 conductivity
- Record pre-cal value in mS/cm, press ENTER, then record post-cal value in mS/cm

Rinse probes with distilled water thoroughly before testing sample or before storing the probes. The conductivity probe should be stored dry, while the pH/ORP electrode bulb is stored in electrode storage solution.

MYRON ULTRAMETER II

Myron instruments are factory calibrated with NIST traceable standard solutions and buffers. These are available from Freshwater Systems Pty Ltd.

The following standard solutions and buffers are used for calibration: pH=4, pH=7, TDS=442-3000 (optional), EC=7000.

- Rinse the Ultrameter 3x with distilled water
- Rinse RHS cell with pink buffer pH=4 solution 3x and fill the cell
- OK If reading is between 3.95-4.04, otherwise recalibrate
- Press CAL, then press the up or down keys to change the reading to match the known value, then press CAL again
- Repeat for pH=7 (green buffer solution), but do all the rinses first
- Rinse LHS cell with the TDS (442-3000) solution and press TDS button, OK if reading is between 2980 and 3020, otherwise recalibrate
- Repeat for EC (KCl-7000) and press COND button, OK if reading is between 6980 and 7020, otherwise recalibrate.

The meter should always be stored with the lid cap in place so the pH/ORP cell does not dry out. Before replacing the rubber cap, fill the well of the sensor with Sensor Storage Solution.

TAKING MEASUREMENTS:

Field measurements need to represent as closely as possible the natural condition of the water at the time of sampling. Measurements should therefore be taken immediately following collection of the water sample.

When using the Myron meter (Fig. 16), rinse the sample cell 3x with distilled water and then 3x with the sample to be tested. Fill the sample cells with the tested sample and measure in turn temperature, ORP, pH, EC and TDS. In some cases it may take a minute or so for the reading to stabilise, in which case the trend should be noted along with the initial reading. For ORP measurements it is recommended that the initial reading be recorded along with the trend of the readout for at least one minute.

After use, rinse cells 3x with distilled water. Occasionally squirt in a foaming cleaner (e.g. Windex®) to remove any oily films or scum which may have coated the electrodes and rinse with water.

Figure 16. Testing leachate samples with the Myron ultrameter. (Photo 414517)

LAB SUPPLIES

Laboratory supplies (glassware etc.) are available from:
ASIS SCIENTIFIC PTY LTD
474 Port Road
Hindmarsh SA 5007
Ph: 8340 8444
customerservice@asisscientific.com.au

Chemical supplies are available from:
FRESHWATER SYSTEMS AUSTRALIA PTY LTD
93 Sir Donald Bradman Drive
Hilton SA 5033
Ph: 8351 7800
jh@freshwatersystems.com.au

RELATED DOCUMENTS / REFERENCES

Freshwater Systems. Advice on maintaining your Myron L 6P, 6PII & 6Psi water meters. Fact Sheet produced by Freshwater Systems Australia Pty Ltd.

Manual for field column test – Water collection and field analysis (TAG Factsheet, author(s) unknown).

6.3 STANDARD OPERATION PROCEDURE — TITRATIONS

PURPOSE AND SCOPE

Acidity is a measure of the concentration of H^+ ions in an aqueous solution. It is expressed as a mass of calcium carbonate equivalent per unit volume (mg/L of $CaCO_3$). One method of measuring acidity is by using the titration test-kit supplied by Earth Systems Pty Ltd. The test-kit measures total acidity to a value of pH 8.3.

This Standard Operation Procedure (SOP) describes the use of the acidity test kit at the Brukunga lab.

MATERIALS/EQUIPMENT/CHEMICALS

The following are required to carry out acidity titrations:

- paper towels, safety glasses and latex gloves
- distilled water / wash bottles
- beakers, pipettes, syringes
- acidity test kit with *risatec* ® micro-titrator
- phenolphthalein indicator (1%), hydrogen peroxide (30% H_2O_2)

OHS

Chemicals used for testing are stored in lockable lab cupboards. Refer to Materials Safety Data Sheets (MSDS) sheets for handling, storage and accidental exposure to chemicals/reagents that are used with the acidity test kit.

Wear safety glasses and surgical gloves during the testing procedure.

TESTING PROCEDURE

Use dedicated beakers, drainage bags or syringes to collect water / leachate samples. Turbid samples may need to be filtered as it is difficult to detect end-point colour changes if the solution is not transparent.

Micro-titrator reaction vessels are stored in an acid bath (2% nitric acid) in a lockable chemical cupboard. Remove vessel from the acid bath and rinse 3x with distilled water and dry.

- Select the low range reagent pipette device stored in the acidity test kit and fill titrant dispersing (hypodermic) syringe **slowly** with low range titrant (0.08 NaOH, 44M) so as not to introduce air bubbles; withdraw 100 units (1.0 mL) of titrant
- Rinse micro-titrator reaction vessel 3x with sample to be tested
- Transfer 10 mL or 20 mL of sample into the reaction vessel using a small plastic syringe (pre-rinse 3x with sample to be tested)
- Add 3 drops of phenolphthalein indicator and 5 drops of hydrogen peroxide to the aliquot in the reaction vessel
- Screw micro-titrator onto the reaction vessel and gently squeeze top of syringe to add titrant to the vessel drop by drop watching for a color change while swirling the vessel (Fig. 17)
- The end-point is when the indicator turns pink (this is somewhat subjective as colors can range from pale pink to bright red). Record the volume of titrant added in mL (e.g. 0.16 mL).

The volume of titrant added is multiplied by a conversion factor to give the acidity concentration of the sample in mg/L of $CaCO_3$. There are a range of conversion factors used depending on the size of the sample aliquot and whether low or high range titrant has been used.

Figure 17. **Testing leachate acidity in the Brukunga lab using an Earth Systems micro-titrator. (Photo 414518)**

It is sometimes necessary to carry out a dilution of the sample to be tested. This is done by adding distilled water (DW) to 5 or 10 mL of the sample and then swirling the beaker to thoroughly mix.

1x dilutions: take 10 mL or 20 mL of sample
5x dilutions: take 10 mL of sample and 40 mL of DW to make up 50 mL
10x dilutions: take 10 mL of sample and add 90 mL of DW to make up 100 mL
20x dilutions: take 5 mL of sample and 95 mL of DW to make up 100 mL
40x dilutions: take 5 mL of leachate and 195 mL of DW to make up 200 mL

LAB SUPPLIES

These are available from a local supplier (e.g. Freshwater Systems Australia Pty Ltd)

- Hydrogen Peroxide (30% H_2O_2)
- Nitric Acid (70%)
- Phenolphthalein (1%)
- Sodium Hydroxide (0.08N)
- pH buffers and calibration standard solutions

RELATED DOCUMENTS / REFERENCES

Earth Systems Pty Ltd Acidity Test Kit Manual and instruction card (further advice on use and maintenance of the test kit can be obtained from Earth Systems).

6.4 STANDARD OPERATION PROCEDURE — PASTE pH TEST

PURPOSE AND SCOPE

This Standard Operation Procedure (SOP) describes a method for determining the paste pH and paste conductivity of rock and soil samples. Paste tests are used to evaluate the geochemical behavior of mine waste materials and estimate the pH (and EC) of pore water resulting from dissolution of secondary minerals on oxidised surfaces of weathered rock particles.

MATERIALS/EQUIPMENT/CHEMICALS

The following are required:

- pH and conductivity meter(s)
- Calibration standard solutions / buffer solutions (pH=4 and 7)
- Stirring rod
- narrow diameter glass beaker
- Distilled water
- Mortar and pestle, sieve
- Weighing scales
- Graduated measuring cylinder

OHS

Wear safety glasses, dust mask, and surgical gloves when preparing materials for testing.

PROCEDURE

Stages involve sample preparation, meter calibration and field testing.

Calibrate pH (and EC probes) using standard solutions. Record calibration data on lab test data sheet (i.e. meter readings prior to and following calibration).

Tests should be carried out on both crushed and uncrushed samples as the undisturbed material provides a better indication of the extent of oxidation than the crushed sample.

The paste pH/EC is determined by equilibrating a sample in deionised water at a solid to water ratio of 1:2 (w/w) and then measuring the pH and EC. This gives an indication of the inherent acidity and salinity of the mine waste material when initially exposed in a waste emplacement area.

SAMPLE PREPARATION

To prepare the slurry (for crushed samples), the rock or soil needs to be air-dried and pulverised and then sieved to particles <1.0 mm size (i.e. gritty sandy texture). The procedure involves adding 2 x parts of water to 1 x part of solid material.

Weight/Volume method: Weigh the mass of material into a small diameter flask. To make up a 1:2 (w/v) ratio of soil:water, slowly add two parts of distilled water to the material. For example, if the weight of material is 25 g, then need to add 50 g of distilled water.

Weight/Weight method: For example if adding 0.100 kg of material to a beaker, then need to add distilled water up to a weight of 0.300 kg.

Mix thoroughly by stirring continuously while adding the deionised water.

Wait for the sample to equilibrate (one reference suggests leave stand for 10 minutes, while another suggests leave stand for up to 12 hours before testing).

The testing needs to be done in situ (i.e. directly on the slurry). It is a good idea to use a small diameter flask/vessel as there is not very much supernatant to play with.

For the uncrushed rock sample, weigh the rock/particles, add 2 parts of water, lightly shake and leave stand without further disturbance.

MEASUREMENTS

Tip beaker to one side to allow water to collect in the corner and dip pH probe into the slurry and stir supernatant by lightly swirling the electrode.

Don't stir anymore once commencing the measurements. When the reading remains constant, record the pH. Take measurements at 1, 3, 5, 10, 15, 30, 60 minutes, then hourly. Check again in 24 hours and a week later.

Decontaminate the probe by rinsing with distilled water between tests.

Plot the pH versus time to determine when pH readings are reaching a plateau. Readings can then be terminated. Once pH testing is completed, use the Myron Ultrameter to test the supernatant for EC.

INTERPRETATION

Low pH readings indicate that oxidation and acid generation has occurred. Readings taken on uncrushed samples often provide a better indication of the extent of oxidation than for crushed samples.

According to Environment Australia (EA 1997), the criteria for potentially acid forming rock material is for saturated paste pH to measure <4.

Relatively high electrical conductivity (EC) levels indicate a considerable store of contaminated salts, usually sulfates or other metal salts.

QA / QC

The accuracy of measurements can be verified by using standard solutions.

Following testing, place the probe(s) into standard solutions and record readings on the lab data sheet. If readings have drifted by more than 5%, recalibrate the probe and repeat the tests.

RELATED DOCUMENTS / REFERENCES

EA 1997. Environment Australia. Managing sulfidic mine wastes and acid drainage, best environmental practice environmental management in mining.

Price WA 1997. Guidelines and recommended methods for the prediction of metal leaching and acid rock drainage at mine sites in British Columbia.

Sobek AA, Schuller WA, Freeman JR and Smith RM 1978. Field and laboratory methods applicable to overburden and mine soils. EPA Report 600/2-78-054. US National Technical Information Service Report PB-280495.

6.5 INSTRUMENT CALIBRATION TRACKING RECORD SHEET

EXAMPLE OF MAINTAINING CALIBRATION RECORDS FOR FIELD PH, EC AND TDS MEASUREMENT

Description of standard solutions

Standard solution	TDS, ppm	EC, µS/cm	pH
KCl-7000		7000	
442-3000	3000		
pH buffer 1			4.0
pH buffer 2			7.0

TDS

Sample	Certificated value, B	Measured value, A	RPD, %	Comment
442-3000	3000 ppm			

EC

Sample	Certificated value, B	Measured value, A	RPD, %	Comment
KCl-7000	7000 µS			

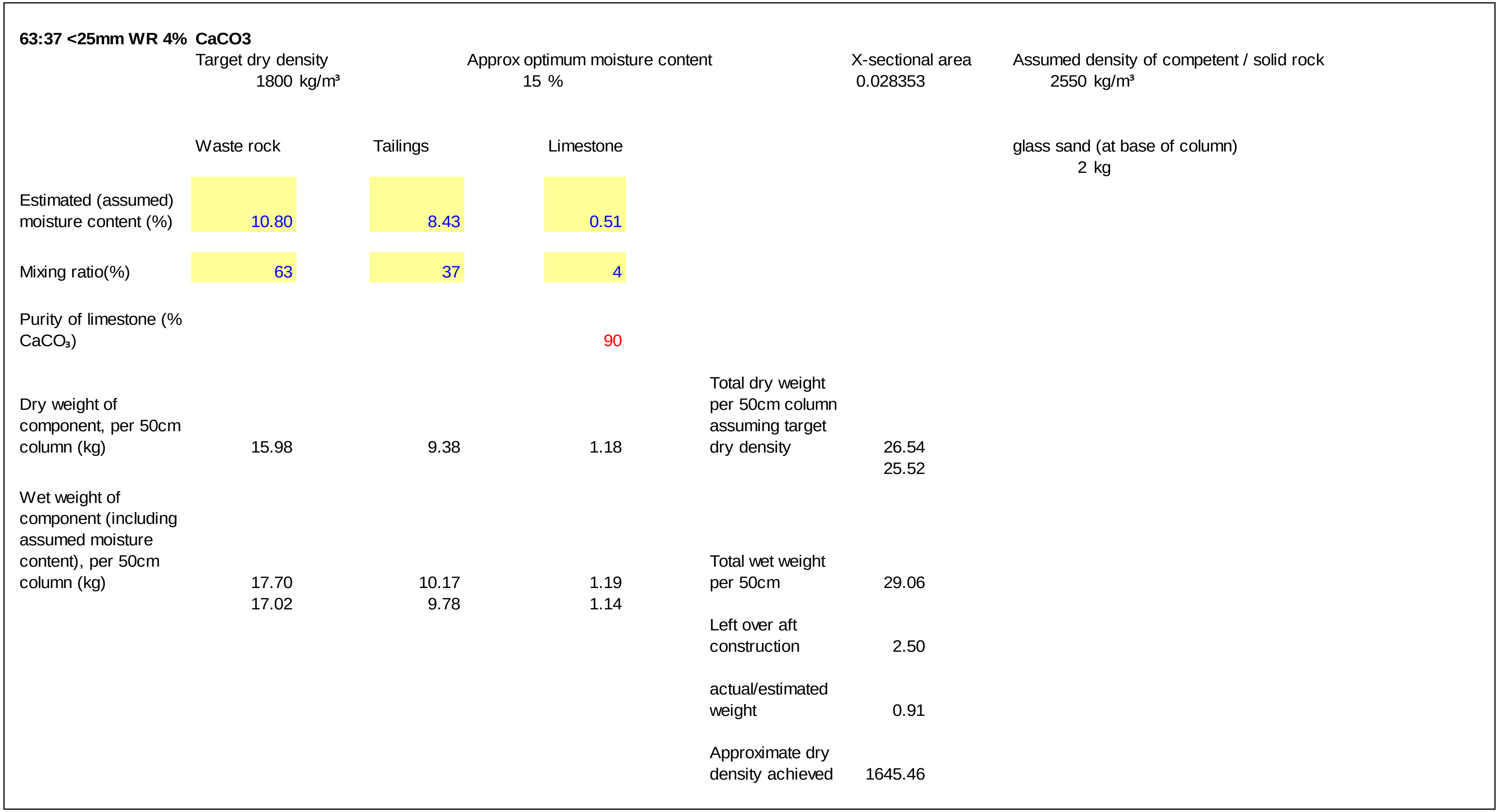
pH

Sample	Certificated value, B	Measured value, A	RPD, %	Comment
pH buffer 1	4.0			
pH buffer 2	7.0			

Recorded by:
Date:
Remarks:

RPD,%=([A-B]/B)*100

6.6 EXAMPLE OF SPREADSHEET FOR CALCULATION OF WEIGHT OF SOLIDS USED IN COLUMN CONSTRUCTION



63:37 <25mm WR 4% CaCO3

Target dry density	Approx optimum moisture content	X-sectional area	Assumed density of competent / solid rock
1800 kg/m³	15 %	0.028353	2550 kg/m³

glass sand (at base of column)
2 kg

	Waste rock	Tailings	Limestone		
Estimated (assumed) moisture content (%)	10.80	8.43	0.51		
Mixing ratio(%)	63	37	4		
Purity of limestone (% $CaCO_3$)			90		
Dry weight of component, per 50cm column (kg)	15.98	9.38	1.18	Total dry weight per 50cm column assuming target dry density	26.54
					25.52
Wet weight of component (including assumed moisture content), per 50cm column (kg)	17.70	10.17	1.19	Total wet weight per 50cm	29.06
	17.02	9.78	1.14		
				Left over aft construction	2.50
				actual/estimated weight	0.91
				Approximate dry density achieved	1645.46

6.7 FIELD TEST SPREADSHEET – LEACH COLUMNS

DATA RECORD FOR FIELD COLUMN TEST

Calibration **pH4:**
pH7: **EC-7000:**

DATE: **RECORDER:**

	Surface water								Leachate											
														Acidity						
Column	pH Field	ORP Field (mV)	EC Field (uS/cm)	Temp Field (deg C)	Vol Samp (mL)	Dil Facto	Volume Titrant (mL)	Acidity surface ng/L CaCO3 per mL titran	Vol Field (mL)	pH Field	ORP Field (mV)	EC Field (uS/cm)	Temp Field (deg C)	Vol Samp (mL)	Dil Facto	Volume Titrant (mL)	Acidity ng/L CaCO3 per mL titrant	Colour	Volume leachate in bags (mL)	Volume irrigation refill (mL)
					20	×1								10	×5					
					20	×1								10	×5					
					20	×1								10	×5					

low range: 0.0786 NM NaOH

FILLED WATER:

pH:

ORP:

EC:
T:

ACIDITY: Unit conversion factors: Surface Water use 197; Leachate use 393

6.8 FIELD TEST SPREADSHEET – LARGE-SCALE COLUMNS

FIELD TESTING DATA SHEET

LARGE-SCALE COLUMN LEACH TESTWORK

Date:

Meter: Myron #6204821

Meter Check against Standard Solns: **pH 7:** **pH4:** **EC 7000:** **ORP 475:**

Bin No:	Configuration	Volume discharged (mL)	Temp (°C)	pH	ORP (mV)	EC (µS/cm)	Color	Odor	Volume irrigation refill (mL)	Comments
1	Baseline PAF + NAF									
2	Baseline O/M in PAF									
3	Baseline basal O/M									
4	Baseline PAF + NAF									
5	NAF only									
Tank	creek water									

Color Descriptions: B=Brown Y=Yellow R=Red p=pale d=dirty/muddy

6.9 EXAMPLE OF QC REPORT FOR LEACHATE SAMPLING

DATE:

Parameter	**Units**	Port 2			
		Lab#1	**Lab#2**	**%difference**	**RPD%**
pH		5.97	5.9	1.17	1.18
EC	µS/cm	12600	11200	11.11	11.76
Acidity	mg/L $CaCO_3$	4050	5630	-39.01	-32.64
Acidity - calculated	mg/L $CaCO_3$	8431	11801		
Alkalinity	mg/L $CaCO_3$	336	421	-25.30	-22.46
Residual alkalinity	mg/L $CaCO_3$	0	0		
Ca	mg/L	445	504	-13.26	-12.43
K	mg/L	547	772	-41.13	-34.12
Mg	mg/L	362	398	-9.94	-9.47
Na	mg/L	596	757	-27.01	-23.80
Sulphate	mg/L	9710	10700	-10.20	-9.70
Chloride	mg/L	308	215	30.19	35.56
Fluoride	mg/L	0.1	0.14	-40.00	-33.33
Nitrate	mg/L	<0.01	<0.06		
Total N	mg/L	21.5	27.1	-26.05	-23.05
TKN	mg/L	21.5	27.0	-25.58	-22.68
Total P	mg/L	0.24	0.668	-178.33	-94.27
COD	mg/L	618	577	6.63	6.86
TOC	mg/L	23	23.2	-0.87	-0.87
DOC	mg/L	20	20.0	0.00	0.00
Al	mg/L	<0.01	6.48		
As	mg/L	0.002	0.011	-450.00	-138.46
Cd	mg/L	0.0002	<0.001		
Cr		<0.001	<0.001		
Cu	mg/L	0.006	<0.001		
Fe	mg/L	3130	4370	-39.62	-33.07
Mn	mg/L	14.7	17.2	-17.01	-15.67
Ni	mg/L	0.003	0.006	-100.00	-66.67
Pb	mg/L	<0.001	<0.001		
Zn	mg/L	0.112	0.124	-10.71	-10.17
Total anions	meq/L	258.0	281.8		
Total cations	meq/L	204.6	268.4		
Ionic balance of analysis	%	11.6	2.4		

6.10 WASTE ROCK 1000 TONNE PILE (PYRAMID) TESTWORK (2007–2009)

A field-scale geochemical trial was designed and implemented by Earth Systems in 2007 to quantify the performance of three AMD management approaches to suppress acidity loads emanating from waste rock dumps:

- The effectiveness of alkaline capping materials
- The benefit of blending limestone with acid generating materials
- The role of soil covers in reducing AMD discharge.

The trials were designed to assess the effect of the following parameters on the minimisation/suppression of acidity loads:

- Amendment grain size
- Waste rock to blended alkaline amendment ratio
- Alkaline amendment solubility and dissolution rates.

The trial involved the construction of seven 1000 tonne waste rock test piles (see Table 4, taken from Struve and Henschke 2012). Waste rock was thoroughly mixed prior to emplacement to ensure compositional homogeneity between each test pile. The test piles were fitted with a surface irrigation system to supplement rainfall and thus accelerate alkalinity dissolution and transport through the test piles. This would enhance the leaching processes, and ensure constant leachate production. Leachate samples were collected monthly for 12 months and analysed for:

- pH, EC, ORP, Cl, F, P, HCO_3 and SO_4
- Major cations
- Key metals: As, Cd, Cr, Cu, Ni, Pb, Zn, Mn, Al, Fe, Sr and Si (ICP-MS).

Follow-up testwork was conducted from November 2008 to August 2009 when pyramids 1 and 2 underwent a design modification to prevent rainfall and irrigation water infiltrating directly into uncapped sulfidic wastes.

Table 4. Waste rock pile / pyramid testwork.

Test Pile/Pyramid No.	Treatment	Variables
1	Alkaline cap	Caustic magnesia selected as the capping material
2	Alkaline cap	Ultra-fine limestone selected as the capping material (<8 µm)
3	Cover material	Impermeable cover applied to isolate waste rock from rainfall
4	None	Test control, representative of baseline conditions
5	Alkaline blending	Blended with 100 tonne of fine limestone (10% mix, grainsize < 2 mm)
6	Alkaline blending	Blended with 20 tonne of ultra-fine grained limestone (2% mix, grainsize <8 µm)
7	Alkaline blending	Blended with 100 tonne of ultra-fine grained limestone (10% mix, grainsize <8 µm)

7. ACKNOWLEDGEMENTS

The following personnel and agencies have provided valuable advice, services, help and assistance throughout the course of the geochemical trial work at Brukunga.

TAG MEMBERS – GEOCHEMICAL
Jeff Taylor (Director, Earth Systems)
Peter Scott (Environmental Earth Sciences)
Stuart Miller (Environmental Geochemistry International)
Mark Logsdon (Geochemica Inc.)

BRUKUNGA MINESITE
Peter Grindley (Brukunga Mine Supervisor)
Paul Wandner (Senior Plant Operator)
Peter Holt (contract services)

LABORATORY SERVICES
Kieren Burns (ALS Environmental)
Russell Schumann (Levay & Co)
Pat Poldervaat (AWQC)
John Waters (On-Site Technology Pty Ltd)

BRUKUNGA TEAM – CURRENT
Raul Mollehuara (Program Manager)
Ross Stevens (Senior Project Manager)
Chris Henschke (Research Officer)

BRUKUNGA TEAM – FORMER
Michael McLeary (former Program Manager)
Naomi Struve (former Research Officer)
Craig Liddicoat (former Research Officer)

VACATION STUDENTS
Wendi Berry
Yuan Tian
Bernice Ng
Gerry Xiu
Farah Ali
James Coley

8. GLOSSARY

Some of the following definitions have been taken from the handbook *Managing acid and metalliferous drainage, Leading Practice Sustainable Development Program for the mining industry* by the Department of Industry Tourism and Resources.

Acid	A measure of hydrogen ion (H^+) concentration; generally expressed as pH. Acid is not equivalent to acidity (see definition below).
Acid drainage	A form of Acid and Metalliferous Drainage (AMD), characterised by low pH, elevated toxic metal concentrations, high sulfate concentrations and high salinity.
Acidity	A measure of hydrogen ion (H^+) concentration and mineral (latent) acidity; generally expressed as mg/L $CaCO_3$ equivalent; measured by titration in a laboratory or estimated from pH and water quality data.
AMD	Acid and Metalliferous Drainage, also known as acid mine drainage or acid rock drainage; AMD can display one or more of the following chemical characteristics: ▪ low pH (typical values range from 1.5 to 4) ▪ high soluble metal concentrations (iron, aluminium, manganese, cadmium, ▪ elevated toxic metal concentrations (copper, lead, zinc, arsenic and mercury) ▪ elevated acidity values (e.g. 50–15 000 mg/L $CaCO_3$ equivalent) ▪ high sulfate (typical sulfate concentrations range from 500–10 000 mg/L) ▪ high salinity (typical EC range from 1000–20 000 µS/cm) ▪ low concentrations of dissolved oxygen (<6 mg/L) ▪ low turbidity or total suspended solids.
EC	Electrical Conductivity is commonly used as a measure of water salinity in units of milli-Siemens per centimetre (mS/cm) or micro-Siemens per centimetre (µS/cm).
Jarosite	Jarosite is a basic hydrous sulfate of potassium and iron ($KFe_3(SO_4)_2(OH)_6$) and is formed by the oxidation of iron sulfides and forms a yellowish or brownish mineral; the presence of Jarosite being an indicator of acidic sulfate rich conditions.
Kinetic test	Procedure used to measure the magnitude and/or effects of dynamic processes, including reaction rates (such as sulfide oxidation and acid generation), material alteration and drainage chemistry and loadings that result from weathering; unlike static tests, kinetic tests measure the behaviour of a sample over time.
NAF	Non-acid forming materials.
O/M	Organic matter.
ORP	Oxidation Reduction Potential.
PAF	Potential acid forming materials.
PIRSA	Primary Industries and Resources, SA (Government of South Australia).
Pyrite	The mineral pyrite, or iron pyrite, is an iron sulfide (FeS_2). Pyrite was mined at Brukunga from 1955–1972.
Static test	Procedure for characterising the physical or chemical status of a geological sample at one point in time. Static tests include measurements of mineral and chemical composition and the analyses required for Acid Base Accounts.
Sulfates	Salts resulting from the chemical action of sufuric acid. In the context of acid sulphate soils and AMD, the term refers to various iron sulfates.
TAG	Technical Advisory Group for the Brukunga remediation design.

Tailings	Finely ground materials from which the desired minerals have been largely extracted.
TSF	Tailings Storage Facility designed for the storage of unsaturated tailings material produced during ore processing. These facilities, unlike tailings dams are not suitable for storage of supernatant water.
Waste rock	Material such as soil or uneconomic mineralised rock that surrounds an orebody and must be removed in order to mine the ore.
Water cover	Layer of surface water or groundwater intended to limit the ingress of oxygen into AMD-generating materials.
WRD	Waste rock dump.

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