

KALGOORLIE CONSOLIDATED GOLD MINES PTY LTD

# **GIDJI ROASTER**

***GEOCHEMICAL CHARACTERISATION  
OF PROFILE-SAMPLES FROM TAILINGS-BED  
(‘STATIC-TESTWORK’)***

**Implications for Process-Tailings Management**

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## 1.0 INTRODUCTION

Kalgoorlie Consolidated Gold Mines Pty Ltd (KCGM) operates the Gidgi Roaster located near Kalgoorlie, Western Australia.

Prior to early-2001, a Calcine stream was discharged to an engineered, tailings-storage facility (TSF). Between early-2001 and early-2002, the process-tailings stream discharged to the TSF comprised a mixture of Calcine, Slimes and UFG-Solids.<sup>1</sup> Since early-2002, the process-tailings stream has comprised Calcine and UFG-Solids.<sup>2</sup> The active-lifetime of the TSF will likely extend until c. 2008.

As shown in the GCA (2001) study, the UFG-Solids contain (finely-ground) pyrite as a major component, so that the process-tailings-solids deposited in the TSF have the potential to acidify. However, since the UFG-Solids contain accessory amounts of "ankerites", the pH will not immediately drop below 6 following tailings-deposition in the TSF. The relative rates of sulphide-oxidation, and dissolution of "ankerites", as influenced by the semi-arid conditions of the Gidgi site, will govern the tailings-bed geochemistry.

Since deposition of UFG-Solids had been in progress for c. 1-2 years, it was deemed timely to assess the geochemical nature (especially pH-status) of the tailings-bed in the TSF.<sup>3</sup> Information of this kind will have a direct bearing on strategies for TSF management (including decommissioning).

Graeme Campbell & Associates Pty Ltd (GCA) was commissioned to carry out geochemical testwork on tailings-solids samples from the tailings-bed profile in the

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<sup>1</sup> UFG = Ultra-Fine Grinding (see GCA [2001] report).

<sup>2</sup> The production-schedules for the Calcine-Solids, and UFG-Solids, are c. 30 dry-tonne per hour (tph) and c. 10 tph, respectively (Mr Trevor Tyson, pers. commun.).

<sup>3</sup> It was also recommended in the GCA (2001) investigation that the geochemistry of the tailings-bed in the TSF is monitored on a regular basis to facilitate planning for TSF-closure.

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TSF.<sup>4</sup> A sample of 100%-Calcine was also tested, since this "end-member-stream" was not included in the samples characterised in the GCA (2001) study.

The main aim of the 'Static-Testwork' Programme was to determine the pH-status of the upper 4 metres (nominal) of the tailings-bed, and thereby infer the likely pH-regime of the "shandy" of seepage-water/groundwater beneath the TSF.<sup>5</sup> Additionally, the sulphide-oxidation rate (SOR) during the "dormant-phase" of c. 3-4 months between tailings-deposition-cycles, is estimated. However, due to the occurrence of "trace-gypsum" (chiefly associated with the Calcine-Solids), the estimated SOR is very approximate. A 'Kinetic-Testwork' Programme (viz. Weathering-Columns) would be needed to better estimate the SOR, depletion rate of alkalinity forms (chiefly "ankerites"), and ensuring pH-Buffering behaviour.

The testwork results are presented and discussed in this report, and implications for TSF management highlighted.

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<sup>4</sup> It should be noted that the term tailings-solids is employed generically herein, and depending on the depth of sampling within the tailings-bed, the tailings-solids may be a mixture of either UFG/Calcine/Slimes, or UFG/Calcine (GCA 2001).

<sup>5</sup> A 'Static-Testwork' Programme comprises "whole-rock" analyses and tests.

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## 2.0 STUDY APPROACH

Details of the sampling and testwork programmes are presented and discussed in the following sections.

### 2.1 Samples

The 100%-Calcine sample (c. 10 kgs) was provided in a 10-L, opaque-plastic-pail. The (moist) calcine-solids were removed, homogenised by hand-mixing, and passed through a 5-mm-nylon sieve.<sup>6</sup>

The tailings-solids samples (c. 3-5 kgs each) from the tailings-bed-profile in the TSF were provided in sealed-plastic-bags packed into 20-L, tin-drums. The tailings-solids samples were collected from depths down to c. 4 m from a Beach-Head site, since:

- (a) Tailings-solids are typically excavated from depths down to c. 3-4 m during the raising of the TSF-embankments by the upstream-construction method, and so the geochemical character of such tailings-solids has implications for TSF-decommissioning; and,
- (b) A drained/unsaturated state is more-developed at the Beach-Heads where coarser-particles mainly reside (c.f. Beach-Toes), and where sulphide-oxidation (as governed by O<sub>2</sub>-diffusive supply) is promoted.<sup>7</sup>

Prior to sampling, the Beach-Head area had been dormant for c. 4 months, and during this period, minimal rainfall occurred (Mr Trevor Tyson, pers. commun.).

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<sup>6</sup> The 100%-Calcine sample emitted an NH<sub>3(g)</sub> odour, and reflects break-down of cyanide forms.

<sup>7</sup> Although the SOR of mine-waste materials in "humid" environments is invariably controlled by the O<sub>2</sub>-supply (e.g. O<sub>2</sub>-diffusion), research and field work is increasingly showing that the supply of water, as governed by the frequency of "flushing-episodes", is the driving control on the SOR in semi-arid/arid environments (Campbell, unpublished results).

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Due to leakage of tailings-porewater from the plastic-bags, all samples were saturated, so that the moisture-content (MC) of each sample could not be meaningfully determined. Accordingly, all samples were oven-dried at c. 40 °C, prior to crushing (nominal 2 mm), and pulverising (nominal 75 µm), for specific tests. Due to their hypersaline-state, the tailings-solids samples were slow to dry at 40 °C, and when sub-samples were dried at 105 °C, the "residual-MCs" ranged up to c. 5-6 % (w/w).<sup>8</sup>

## 2.2 Testwork

The testwork methods employed in this study are based on recognised procedures for the geochemical characterisation of mine-waste materials (e.g. Morin and Hutt 1997; Smith 1992; Coastech Research 1991; BC AMD Task Force 1989).

Details of the testwork methods are presented in Appendix A, and are similar to those employed in the GCA (2001) study.

Part of the testwork was carried out by Australian Environmental Laboratories [AEL] (Welshpool). The analyses performed by AEL have NATA endorsement.<sup>9</sup>

Specialised testing (viz. auto-titrations) were performed by Dr. Graeme Campbell in the GCA testing-Laboratory (Bridgetown).

Copies of the laboratory reports are presented in Appendix B.

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<sup>8</sup> Such "residual-MCs" attest to the strong water-retention of process-tailings-solids that contain appreciable amounts of (hygroscopic) salts (William 2002).

<sup>9</sup> NATA = National Association of Testing Authorities.

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### 3.0 ACID-BASE CHEMISTRY AND SALINITY OF TAILINGS-SOLIDS AND 100%-CALCINE SAMPLES

The testwork results on the acid-base chemistry of the tailings-solids and 100%-Calcine samples are presented in Tables 3.1 and 3.2, and shown on Figures 1 and 2. These results are discussed in the following sections.

#### 3.1 pH and Salinity

The samples had pH-(1:2) and EC-(1:2) values within the range 8.7-10.0, and 16-56 mS/cm, respectively (Table 3.1).<sup>10</sup>

Although somewhat variable, the EC-(1:2) values indicate accumulation of salts (chiefly halite) within the top *c.* 0.1 m of the tailings-bed.<sup>11</sup> This finding accords with that usually observed in TSFs at local gold-mines where solute-concentration (through capillary-rise/evaporation processes) within the Surface-Zone, is operative at "spatial-scales" of centimetres-to-decimetres.

*The testwork results indicate that the tailings-solids and 100%-Calcine samples were all alkaline (viz. pH 9-10), and hypersaline.*

*Accordingly, any tailings-porewater that seeps from the top 4 m (nominal) of the tailings-bed should be alkaline, and not acidic.*

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<sup>10</sup> EC = Electrical-Conductivity. The pH-(1:2) and EC-(1:2) Tests are described in Appendix A.

### 3.2 Sulphur Forms

The 100%-Calcine sample had Total-S and SO<sub>4</sub>-S values of 2.3 %, and 2.0-2.4 %, respectively (Table 3.1). The Sulphide-S content of the 100%-Calcine sample was therefore less than 0.1 %, and the SO<sub>4</sub>-S should occur chiefly as gypsum (CaSO<sub>4</sub>.2H<sub>2</sub>O).

The tailings-solids samples had Total-S and SO<sub>4</sub>-S values of 7.7-23.2 %, and 0.75-2.2 %, respectively (Table 3.1). The Sulphide-S contents of the tailings-solids samples were therefore 6.1-23.0 %, and should occur chiefly in the form of pyrite (GCA 2001). If the SO<sub>4</sub>-S content of the Calcine is c. 2.0 %, and if the overall mass-ratio (w/w, dry-solids) of UFG:Calcine is c. 1:3, then the SO<sub>4</sub>-S content of the "UFG/Calcine-Solids" in the TSF should be c. 1.5 %.<sup>12</sup> This estimated-SO<sub>4</sub> content is near the measured-SO<sub>4</sub> contents of the tailings-solids samples, and suggests that the SO<sub>4</sub>-S produced through sulphide-oxidation *in situ* is well within the sub-percent range.

*The testwork results indicate that, as expected, the 100%-Calcine sample contained negligible amounts of sulphide-minerals, corresponding to a Sulphide-S content less than 0.1 %. The tailings-solids samples contain pyrite as a minor-to-major component.*

*Although difficult to estimate accurately, when due account is taken of the SO<sub>4</sub>-S content of the Calcine in the "UFG/Calcine-Solids" overall, the indications are that SO<sub>4</sub>-S produced through sulphide-oxidation in situ is well within the sub-percent range, and probably 'of-the-order' 0.1 % (as S). On this basis, the amount of acid produced through sulphide-oxidation following tailings-discharge to the TSF is likely less than approximately 5 kg H<sub>2</sub>SO<sub>4</sub>/tonne.<sup>13</sup>*

<sup>11</sup> The greater EC-(1:2) values within the top 0.1 m of the tailings-bed reflect mainly chlorides [Table 3.2].

<sup>12</sup> The "fresh/unoxidised" sample of the UFG/Calcine/Slimes tested in the GCA (2001) study had a SO<sub>4</sub>-S value of 0.97-1.2 %, and is consistent with the SO<sub>4</sub>-S content of the 100%-Calcine sample tested herein.

<sup>13</sup> The complete-oxidation of c. 0.15 % of Pyrite-S would produce c. 4.6 kg H<sub>2</sub>SO<sub>4</sub>/tonne (Appendix A).

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### 3.3 Acid-Consuming Properties

#### 3.3.1 Acid-Neutralisation Capacity

The 100%-Calcine sample had an Acid-Neutralisation-Capacity (ANC) value of 26 kg H<sub>2</sub>SO<sub>4</sub>/tonne, and a CO<sub>3</sub>-C value of 0.25 % (Table 3.1).<sup>14</sup> If all of the CO<sub>3</sub>-C occurred as "CaCO<sub>3</sub>", then the "Carbonate-ANC" value would be *c.* 20 kg H<sub>2</sub>SO<sub>4</sub>/tonne. Although the carbonate-mineral in the 100%-Calcine sample was not characterised in this study, weakly/moderately-ferroan varieties (viz. "ankerites") should predominate. The occurrence of reactive-carbonates was evident from the effervescence (i.e. 'fizzing') produced upon the addition ('in-the-cold') of HCl in the ANC Tests.

The tailings-solids samples had ANC values of 66-130 kg H<sub>2</sub>SO<sub>4</sub>/tonne, and CO<sub>3</sub>-C values of 0.46-1.4 % (Table 3.1).<sup>15</sup> If all of the CO<sub>3</sub>-C occurs as "CaCO<sub>3</sub>", then the "Carbonate-ANC" values would be *c.* 38-120 kg H<sub>2</sub>SO<sub>4</sub>/tonne. However, "ankerites" should dominate the carbonate-mineral suite (GCA 2001). The occurrence of ferroan-carbonates is indicated by the drop in pH to *c.* 3-4 upon the dropwise-addition of H<sub>2</sub>O<sub>2</sub> as the pH=7 end-point was approached in the ANC Tests.

*The testwork results indicate that the 100%-Calcine sample had a moderate capacity to consume acid, due to trace-to-accessory amounts of carbonates (e.g. "ankerites").*

*The tailings-solids samples had a high capacity to consume acid, due to accessory-to-minor amounts of "ankerites" (GCA 2001).*

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<sup>14</sup> An ANC value of 26 kg H<sub>2</sub>SO<sub>4</sub> is equivalent to *c.* 2.6 % (as CaCO<sub>3</sub>).

<sup>15</sup> ANC values of 66-130 kg H<sub>2</sub>SO<sub>4</sub>/tonne are equivalent to *c.* 6.6-13 % (as "CaCO<sub>3</sub>").

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### 3.3.2 pH-Buffering Properties

The pH-Buffering properties of selected samples were determined using 0.05 M-H<sub>2</sub>SO<sub>4</sub>, and an auto-titrator (see Appendix A). The H<sub>2</sub>SO<sub>4</sub>-addition rates employed during the auto-titrations ranged up to *c.* 5-10 x 10<sup>4</sup> kg H<sub>2</sub>SO<sub>4</sub>/tonne/year, and so correspond to SORs, at circum-neutral-pH, that are very-fast.<sup>16</sup>

The pH-Buffering curves for all samples exhibited a more-or-less steady decrease in pH with progressive addition of acid (Figures 1 and 2), and the shapes of the curves are similar to those generally observed where "ankerites" contribute appreciably to the Total-ANC (Campbell 2002a,b).

At the rapid rate of H<sub>2</sub>SO<sub>4</sub>-addition employed, the auto-titrations indicate that the tailings-solids samples (*viz.* GCA4758 and GCA4759) from the Surface-Zone (*viz.* 0.0-0.1 m) of the tailings-bed are capable of consuming *c.* 40 kg H<sub>2</sub>SO<sub>4</sub>/tonne, before the pH drops below 6. Although difficult to quantify *a priori*, at the slower SORs expected for the Surface-Zone-Tailings at circum-neutral-pH, acid-consumption should be considerably greater than 40 kg H<sub>2</sub>SO<sub>4</sub>/tonne, before the pH falls below 6 *in situ*.<sup>17</sup> Since the amount of acid produced through sulphide-oxidation is estimated to be less than *c.* 5 kg H<sub>2</sub>SO<sub>4</sub>/tonne (see Section 3.2), it follows that after an exposure-period of *c.* 4 months, the "reserve" of alkalinity-forms within the Surface-Zone-Tailings has been depleted only marginally. The indications are that the duration of the "lag-phase" (*i.e.* the period during which sulphide-oxidation occurs, but acidic conditions do not

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<sup>16</sup> As an approximate guide, it would be atypical for process-tailings-solids that are "sulphide-rich" (e.g. Sulphide-S contents of *c.* 10-20 %) to be characterised by SORs at circum-neutral-pH that are much above 50 kg H<sub>2</sub>SO<sub>4</sub>/tonne/year (as determined through 'kinetic' testing where samples are subjected to weekly weathering-cycles).

<sup>17</sup> At circum-neutral-pH, the SOR of the UFG/Calcine-Solids is unlikely to be faster than *c.* 1,000 mg SO<sub>4</sub>/kg/week, as determined from weekly weathering-cycles in 'kinetic' testing (= *c.* 50 kg SO<sub>4</sub>/tonne/year). A programme of 'kinetic' testing would be needed to confirm (ore refine) this estimated SOR for the UFG/Calcine-Solids in the TSF.

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develop) may amount to at least 2-3 years. However, 'kinetic' testing would be needed to better estimate the duration of the "lag-phase".

*The testwork results indicate that the "ankerites" in the tailings-solids samples should be capable of maintaining the pH above 6 until at least 40-50 kg H<sub>2</sub>SO<sub>4</sub>/tonne of acid has been consumed. Such pH-Buffering properties are consistent with "ankerites" that are only weakly/moderately-ferroan.*

*Although approximate, and subject to confirmation/refinement through 'kinetic' testing, the indications are that, under the semi-arid conditions of the mine-site, the "lag-phase" to acidification of exposed Surface-Zone-Tailings may amount to years (c.f. weeks-to-months). Such inferred dynamics of weathering-processes within the Surface-Zone-Tailings reflect the "flushing-frequency" dependence of sulphide-oxidation in semi-arid/arid environments, as typically encountered at local gold-mines.*

*The estimated duration of the "lag-phase" is therefore long, compared with the dormant-phase ranging up to 3-4 months between tailings-deposition-cycles to the TSF (Mr Trevor Tyson, pers. commun.). However, the Surface-Zone-Tailings on the outer-surface of the TSF-embankments raised via the upstream-construction method will likely be exposed for periods considerably longer than 3-4 months. Further information (especially 'kinetic' testing) is needed to indicate the timing for, and form of, the capping-layer to be placed over the outer-surfaces of the TSF-embankments, as part of TSF-closure.*

### **3.4 Acid-Formation Potential**

The 100%-Calcine sample had a Net-Acid-Producing-Potential (NAPP) value of -22 kg H<sub>2</sub>SO<sub>4</sub>/tonne (Table 3.1), and reflects the occurrence of "trace-carbonates", and negligible amounts of sulphides.

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## 4.0 CONCLUSIONS

Based on the testwork results obtained in this study, it is concluded that:

- Within the operating-TSF, the SORs are sufficiently slow, that neutral-to-alkaline conditions should prevail within the tailings-bed throughout the active-lifetime of the TSF (scheduled to be complete by *c.* 2008).<sup>18</sup> Although subject to confirmation/refinement through 'kinetic' testing (viz. Weathering-Columns), the "ankerites" are believed to be weakly/moderately-ferroan varieties that are capable of maintaining the pH above 6 until *c.* 1-2 % (as S) of Pyrite-S has been oxidised.

As a "first-pass" estimate, the SORs (at circum-neutral-pH) may be 'of-the-order' 10 kg H<sub>2</sub>SO<sub>4</sub>/tonne/year, as governed by the (infrequent!) "flushing-episodes" arising from sizeable (e.g. greater than 10 mm over a 24-hr period) rainfall-events. On this basis, the duration of the "lag-phase" (i.e. the period during which pyrite-oxidation occurs, but acidic conditions do not develop), may amount to years (c.f. weeks-to-months). The estimated "lag-phase" is therefore long, compared with the *c.* 3-4 months that typically elapse between deposition-cycles on the tailings-beaches in the TSF.

- Tailings-porewater that seeps from the top 4 m (nominal) of the tailings-bed where the UFG-Solids reside should be neutral-to-alkaline.<sup>19</sup> Accordingly, the "shandy" of seepage-water/groundwater beneath the TSF should also be neutral-to-alkaline.

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<sup>18</sup> TSF = Tailings-Storage Facility; SOR = Sulphide-Oxidation Rate.

<sup>19</sup> UFG = Ultra-Fine Grinding.

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- Since the outer-surfaces of the TSF-embankments raised via the upstream-construction method will comprise PAF-Tailings, placement of a capping-layer as part of TSF-closure must occur before the Surface-Zone-Tailings acidify.<sup>20</sup>

*In brief, the expectation that the PAF-tailings in the TSF at the Gidji site would not acidify "immediately" (GCA 2001), has been confirmed by the findings of this study. Since completion of the GCA (2001) investigation, it has become very clear through both research- and field-work (Campbell, unpublished results) that, within semi-arid environments, sulphide-oxidation in tailings-beds is **not** a continuous process (and controlled by O<sub>2</sub>-diffusive supply), as commonly encountered in "humid" environments where rainfall is plentiful. Instead, the SOR in semi-arid environments is directly linked to the frequency of "flushing-episodes", as governed by sporadic rainfall-events. When taken over the annual hydrological-cycle, weathering of PAF-Tailings in the semi-arid regions of Western Australia is best viewed as a "stop-start" process wherein the "oxidation-cycle" is largely complete within days following a "flushing-episode". During the extended duration (often months) that typically elapses between successive "flushing-episodes", sulphide-oxidation essentially "shuts-down". Such extended periods between inundations also mean that slowly-dissolving-gangue phases (e.g. ferroan-carbonates and primary-silicates) are more effective in buffering near pH=7 (c.f. "humid" environments).*

*It is recommended that 'kinetic' testing is undertaken to confirm/refine the estimated SORs and "lag-phase" duration presented in this study, since these estimates are "first-pass" only. Better estimation of the "lag-phase" duration will facilitate development of a TSF-closure strategy (e.g. use of the "store-and-release" approach for capping-layers over PAF-Tailings in semi-arid/arid environments) [Wilson 2000,2002].*

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<sup>20</sup> PAF = Potentially-Acid Forming.

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## 5.0 REFERENCES

British Columbia Acid Mine Drainage Task Force Report, 1989, "Draft Acid Rock Drainage Technical Guide. Volume 1".

Campbell GD, 2002a, "Geochemistry and Management of Pyritic Mine-Wastes: I. Characterisation", in Proceedings of Workshop on "Soil Technology - Contaminated Land", February 2002, Centre for Land Rehabilitation, University of Western Australia.

Campbell GD, 2002b, "Geochemistry and Management of Pyritic Mine-Wastes: II. Weathering Behaviour and Arsenic Solubility", in Proceedings of Workshop on "Soil Technology - Contaminated Land", February 2002, Centre for Land Rehabilitation, University of Western Australia.

Coastech Research Inc., 1991, "Acid Rock Drainage Prediction Manual".

Graeme Campbell & Associates Pty Ltd, 2001, "Gidgi Roaster & Fimiston Mill: Geochemical Characterisation of Process Tailings Samples ('Static-Testwork') - Implications for Tailings Management", Unpublished report prepared for Kalgoorlie Consolidated Gold Mines Pty Ltd.

Jambor JL, Dutrizac JE and Chen TT, 2000, "Contribution of Specific Minerals to the Neutralization Potential in Static Tests", pp. 551-565 in "Proceedings from the Fifth International Conference on Acid Rock Drainage", Volume I, Denver.

Lenahan WC and Murray-Smith R de L, 1986, "Assay and Analytical Practice in the South African Mining Industry", The South African Institute of Mining and Metallurgy Monograph Series M6, Johannesburg.

- 
- Morin KA and Hutt NM, 1997, "Environmental Geochemistry of Minesite Drainage: Practical Theory and Case Studies", MDAG Publishing, Vancouver.
- Smith A, 1992, "Prediction of Acid Generation Potential", in Hutchison IPG and Ellison RD (eds), "Mine Waste Management", Lewis Publishers, Michigan.
- Sobek AA, Schuller WA, Freeman JR and Smith RM, 1978, "Field and Laboratory Methods Applicable to Overburdens and Minesoils", EPA-600/2-78-054.
- Stevens RE and Carron MK, 1948, "Simple Field Test for Distinguishing Minerals by Abrasion pH", *American Mineralogist*, 33:31-49.
- White AF and Brantley SL (eds.), 1995, "Chemical Weathering Rates of Silicate Minerals", Reviews in Mineralogy, Volume 31, Mineralogical Society of America, Washington, D.C.
- Wilson GW, 2000, "Appropriate Concepts and Criteria for the Design and Construction of Mine Waste Cover Systems", pp. 81-90 in Grundon NJ and Bell LC (eds.), "Proceedings of Fourth Australian Workshop on Acid Mine Drainage", Australian Centre for Mining Environmental Research.
- Wilson GW, 2002, "Concepts for Soil Cover Design", in "Short Course on Design of Covers for Saline, Sodic and Sulfidic Wastes", Newcastle, 10th November 2002, Australian Centre for Mining Environmental Research.
- Williams D, 2002, "Case Study: Trial Covers on Saline Tailings", in "Short Course on Design of Covers for Saline, Sodic and Sulfidic Wastes", Newcastle, 10th November 2002, Australian Centre for Mining Environmental Research.

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**TABLES**

**Table 3.1: Acid-Base-Analysis and Salinity Results for Tailings-Solids Samples from TSF-Beach-Head Site and 100%-Calcine Sample**

GCA-SAMPLE NO.	SAMPLE DEPTH (m)	pH-(1:2)	EC-(1:2) [mS/cm]	TOTAL-S (%)	SO <sub>4</sub> -S (%)	Sulphide-S (%)	CO <sub>3</sub> -C (%)	ANC		ANC/MPA RATIO	AFP CATEGORY
								(kg H <sub>2</sub> SO <sub>4</sub> /tonne)	NAPP		
<b>PROFILE-SAMPLES FROM BEACH-HEAD SITE</b>											
GCA4758	0.00-0.05	9.0	56	23.2	1.2	23.0	0.55	83*	630	<1	PAF
GCA4759	0.1	9.0	49	20.4	1.4	19.0	0.46	75*	510	<1	PAF
GCA4760	0.2	8.9	39	21.0	1.2	18.8	0.48	66*	510	<1	PAF
GCA4761	0.3	8.9	44	11.6	2.2	9.4	0.78	90*	200	<1	PAF
GCA4762	0.5	8.8	37	7.7	1.6	6.1	nm	54* (54*)	140	<1	PAF
GCA4763	1.0	10.0	42	15.7	1.2	14.5	nm	130*	320	<1	PAF
GCA4764	2.0	9.5	32	9.5	1.5	8.0	nm	73*	180	<1	PAF
GCA4765	3.0	9.0	18	11.1	1.6	9.5	nm	110*	190	<1	PAF
GCA4766	4.0	8.7 (8.7)	16 (16)	14.9	0.78 (0.75)	14.1	1.4	100*	340	<1	PAF
<b>100%-CALCINE SAMPLE</b>											
GCA4668	-	8.7 (8.7)	34 (32)	2.3	2.0 (2.4)	<0.1	0.25	26	-22	8.4	NAF

**Notes:**

EC = Electrical Conductivity; ANC = Acid-Neutralisation Capacity; NAPP = Net-Acid-Producing Potential; AFP = Acid-Formation Potential; PAF = Potentially-Acid Forming; NAF = Non-Acid Forming; nm = not measured.  
 pH-(1:2) and EC-(1:2) values correspond to sample slurries prepared using deionised-water, and a solid:solution ratio of c. 1:2 (w/w).  
 ANC values labelled with an asterisk signify that the pH values dropped to c. 3-4 upon the dropwise-addition of H<sub>2</sub>O<sub>2</sub>, as the pH=7 end-point was approached in the ANC Tests (see Section 3.3.1).  
 All results expressed on a dry-weight basis, except for pH-(1:2) and EC-(1:2).  
 Values in parentheses represent duplicates.

**Table 3.2: Chloride and Sulphate-S Contents of Tailings-Solids Samples from TSF-Beach-Head Site**

GCA-SAMPLE NO.	SAMPLE DEPTH (m)	WATER-EXTRACTABLE Cl (% w/w)	WATER-EXTRACTABLE-SO <sub>4</sub> -S (% w/w)	Na <sub>2</sub> CO <sub>3</sub> -EXTRACTABLE-SO <sub>4</sub> -S (% w/w)
GCA4758	0.00-0.05	4.7	0.79	1.2
GCA4759	0.1	3.6	0.76	1.4
GCA4760	0.2	3.0	0.78	1.2
GCA4761	0.3	3.4	0.73	2.2
GCA4762	0.5	2.7	0.72	1.6
GCA4763	1.0	3.0	0.85	1.2
GCA4764	2.0	2.3	0.69	1.5
GCA4765	3.0	2.0	0.71	1.6
GCA4766	4.0	1.5	0.68	0.78 (0.75)

**Notes:**

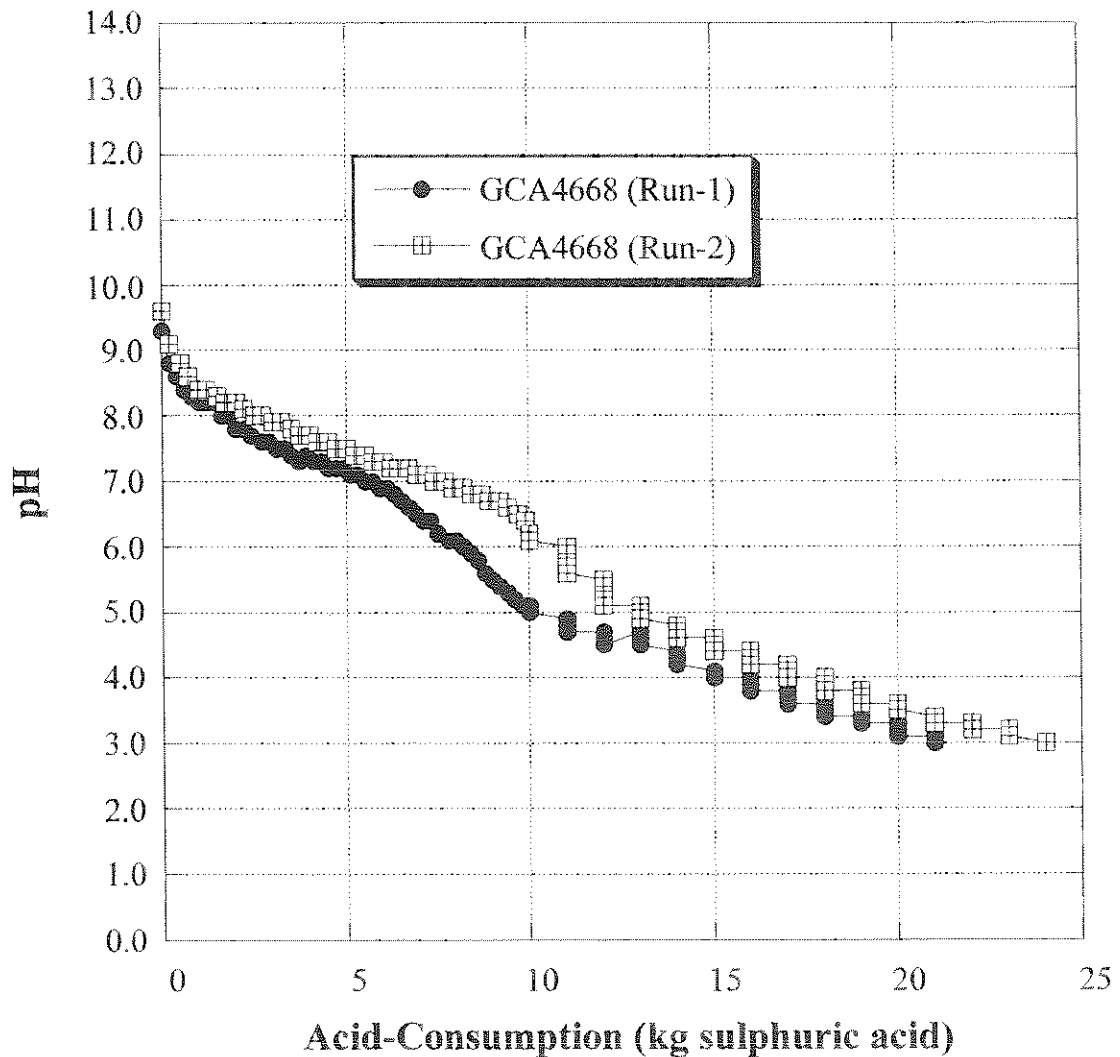
Water-Extraction Testwork employed sample-slurries corresponding to a solid:solution ratio of c. 1:10 (w/w). The sample-slurries were bottle-rolled for c. 24 hrs before being left to stand prior to vacuum-filtering of the supernatants for analysis. All results expressed on a dry-weight basis. Values in parentheses represent duplicates.

---

**FIGURES**

Figure 1

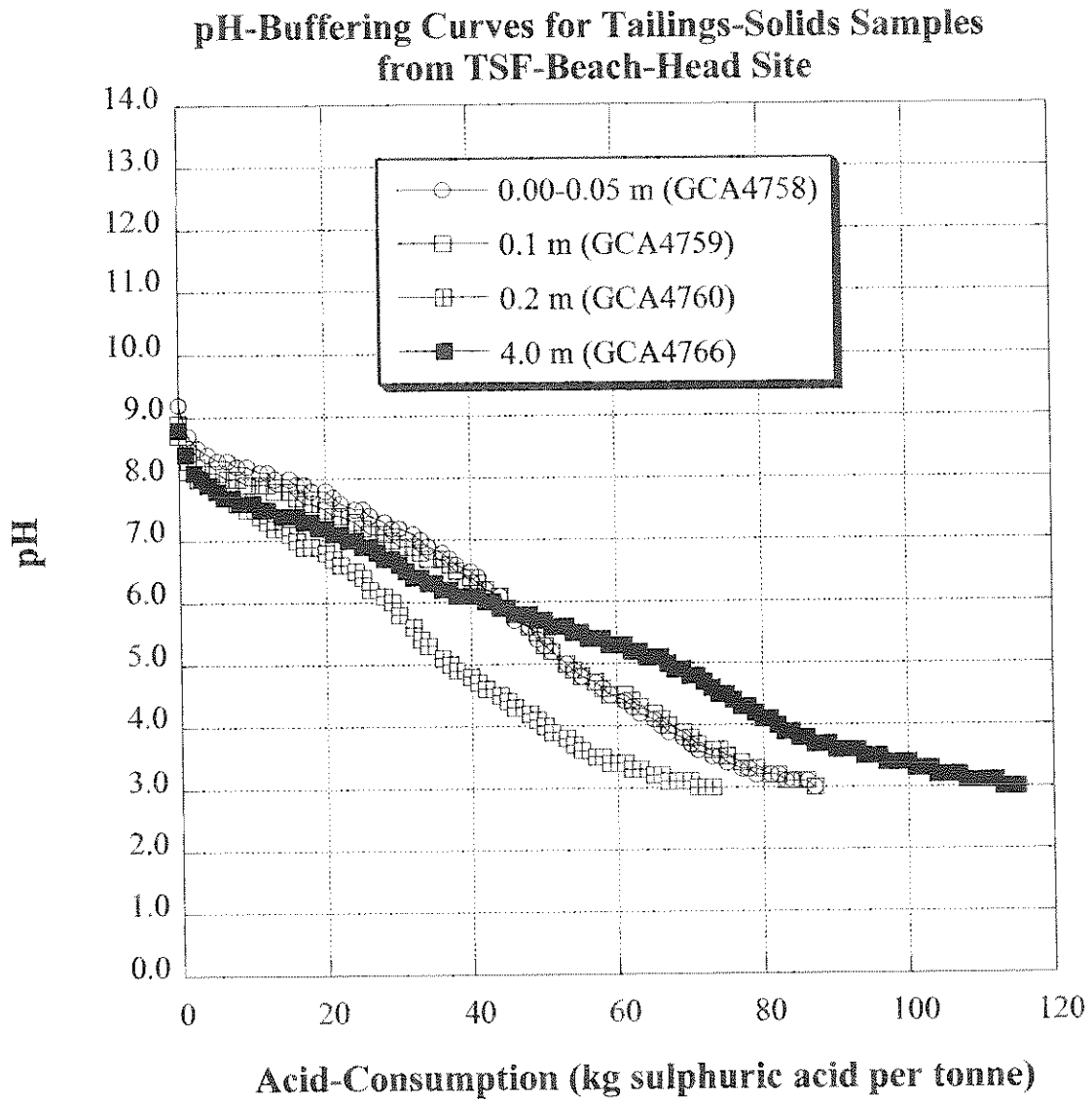
pH-Buffering Curve for 100%-Calcine Sample



Note: The  $H_2SO_4$ -addition rates employed in the auto-titrations correspond to sulphide-oxidation (SOR) rates of *c.*  $1-2 \times 10^5$  mg  $SO_4$ /kg/week (= *c.*  $5-10 \times 10^3$  kg  $H_2SO_4$ /tonne/year).

These SORs are therefore up to  $10^4$ - $10^5$  faster than those typical for the weathering (at least at circum-neutral-pH) of mine-waste materials that contain "trace-sulphides".

Figure 2



Note: The  $H_2SO_4$ -addition rates employed in the auto-titrations correspond to sulphide-oxidation (SOR) rates of *c.*  $1-2 \times 10^6$  mg  $SO_4$ /kg/week (= *c.*  $5-10 \times 10^4$  kg  $H_2SO_4$ /tonne/year).

These SORs are therefore up to  $10^3-10^4$  faster than those typical for the weathering (at least at circum-neutral-pH) of mine-waste materials that are "sulphide-rich".

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**APPENDIX A**

**TESTWORK METHODS**

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## APPENDIX A

### TESTWORK METHODS

#### A1.0 ACID-BASE-CHEMISTRY AND SALINITY TESTWORK

The acid-base chemistry and salinity of the tailings-solids and 100%-Calcine samples was assessed by determining:

- pH and Electrical-Conductivity (EC) on sample slurries.
- Total-Sulphur (Total-S) and Sulphate-Sulphur (SO<sub>4</sub>-S).
- Acid-Neutralisation Capacity (ANC), Carbonate-Carbon (CO<sub>3</sub>-C), and pH-Buffering properties.
- Net-Acid-Producing Potential (NAPP).

Relevant details of the testwork methods employed are discussed briefly below. Further details are presented in the laboratory reports (see Appendix B).

#### A1.1 pH-(1:2) and EC-(1:2) Tests

Measurements of pH and EC were performed on slurries prepared using deionised-water, and a solid:water ratio of *c.* 1:2 (w/w). The sample slurries were allowed to age in contact with the air for *c.* 24 hours, prior to measuring pH and EC.<sup>1</sup>

The resulting pH-(1:2) and EC-(1:2) values provide a measure of the inherent acidity/alkalinity and salinity of the samples.<sup>2</sup>

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<sup>1</sup> The sample slurries were stirred at the beginning of the testwork, and once again immediately prior to measuring pH and EC. The tailings-solids samples had been oven-dried at *c.* 40 °C, prior to preparing the sample slurries.

<sup>2</sup> The pH-(1:2) values approximate the "Abrasion-pH" values employed for identifying minerals in the field (Stevens and Carron 1948).

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## A1.2 Total-S and SO<sub>4</sub>-S Tests

The Total-S values were measured by Leco combustion (@ 1300 °C) with detection of evolved SO<sub>2(g)</sub> by infra-red spectroscopy.

The SO<sub>4</sub>-S values were determined by the Na<sub>2</sub>CO<sub>3</sub>-Extraction Method (Lenahan and Murray-Smith 1986).<sup>3</sup> The difference between the Total-S and SO<sub>4</sub>-S values indicates the Sulphide-S (strictly Non-Sulphate-S) content.

It was assumed that the sulphide-mineral suite in the tailings-solids samples is dominated by pyrite (GCA 2001).

## A1.3 Acid-Consuming Properties

### A1.3.1 ANC Tests

The ANC values of the samples were determined by a procedure based on that of Sobek *et al.* (1978). This procedure is essentially the "standard" method employed for estimating the ANC values of mine-waste materials (Morin and Hutt 1997; BC AMD Task Force 1989).

The samples were reacted with dilute HCl for *c.* 2 hours at 80-90 °C, followed by back-titration with NaOH to a pH=7 end-point to determine the amount of acid consumed.<sup>4</sup> The simmering step for *c.* 2 hours differs slightly from the heating treatment of the Sobek *et al.* procedure wherein the test mixtures are heated to near boiling until reaction is deemed to be complete (*viz.* gas evolution not visually apparent), followed by boiling

---

<sup>3</sup> The Na<sub>2</sub>CO<sub>3</sub>-reagent extracts SO<sub>4</sub>-S which occurs as soluble sulphates, and calcium sulphates (e.g. gypsum and anhydrite). It also extracts SO<sub>4</sub> sorbed to the surfaces of sesquioxides, clays and silicates. However, SO<sub>4</sub> present as barytes (BaSO<sub>4</sub>) is not extracted, and SO<sub>4</sub> associated with jarositic-type and alunitic-type compounds are incompletely extracted.

<sup>4</sup> Two drops of 30 % (w/w) H<sub>2</sub>O<sub>2</sub> were added to the test mixtures as the pH=7 end-point was approached, so that any Fe(II) forms released by the acid-attack of ferroan-carbonates and -silicates are oxidised to Fe(III) forms (which then hydrolyse to "Fe(OH)<sub>3</sub>"). This step ensures that the resulting ANC values are not unduly biased "on-the-high-side", due to the release of Fe(II) during the acidification/digestion step. Such potential bias in ANC values may be marked for mine-waste samples in which "Fe-rich" ferroan-carbonates (e.g. siderite) dominate acid consumption. The addition of the H<sub>2</sub>O<sub>2</sub> reagent is not part of the methodology described by Sobek *et al.* (1978).

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for one minute. In terms of dissolution of carbonate, primary-silicate and oxyhydroxide minerals, this variation to the Sobek *et al.* method is inconsequential.

The Sobek *et al.* (1978) procedure exposes mine-waste samples to both strongly-acidic conditions (e.g. pH of 1-2), and a near-boiling temperature. Provided excess acid is added, this method ensures that carbonate-minerals (including ferroan and manganoan varieties) are dissolved quantitatively, and that at least traces of ferro-magnesian silicates (e.g. amphiboles, pyroxenes, chlorites, micas, etc.), and feldspars, are dissolved. However, under circum-neutral (*viz.* pH 6-8) conditions required for mine-waste and environmental management, the dissolution of ferro-magnesian silicates is kinetically extremely slow (e.g. see review-monograph by White and Brantley [1995]). Near pH=7, the dissolution rates (under 'steady-state' conditions, and in the absence of inhibiting alteration-rims) of mafic-silicates and feldspars generally correspond to H<sub>2</sub>SO<sub>4</sub>-consumption rates 'of-the-order' 10<sup>-11</sup>/10<sup>-12</sup> moles/m<sup>2</sup>/s (White and Brantley 1995). As a guide, for minerals of sub-mm grading, such silicate-dissolution rates correspond to Sulphide-Oxidation Rates (SORs) ranging up to 'of-the-order' 1-10 mg SO<sub>4</sub>/kg/week (= 0.05-0.5 kg SO<sub>4</sub>/tonne/year).<sup>5</sup> Maintenance of circum-neutral-pH through dissolution/hydrolysis of primary-silicates is therefore restricted to both "mineral-fines", and slow rates of pyrite weathering.

Despite the aggressive-digestion conditions employed, the ANC values determined by the Sobek *et al.* (1978) method allow an informed, initial "screening" of mine-waste materials in terms of acid-consuming and pH-buffering properties, especially when due account is taken of gangue mineralogy (Morin and Hutt 1997). Jambor *et al.* (2000) have presented a compendium of 'Sobek-ANC' values for specific classes of primary-silicates, and assists interpretation of the ANC values recorded for mine-waste materials of varying mineralogy.

#### A1.3.2 CO<sub>3</sub>-C Values

The CO<sub>3</sub>-C value is the difference between the Total-C and Total-Organic-C (TOC) values.

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<sup>5</sup> SORs of this magnitude (at circum-neutral-pH) would typically only be recorded for the oxidation of "trace" sulphides (e.g. Sulphide-S contents less than 0.5 %).

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The Total-C was measured by Leco combustion (@ 1300 °C) with detection of evolved CO<sub>2(g)</sub> by infra-red spectroscopy. The TOC was determined by Leco combustion on a sub-sample which had been treated with strong HCl to decompose carbonate-minerals.

It is assumed that the carbonate-mineral suite in the tailings-solids samples was dominated by "ankerites" (GCA 2001). Although not determined as part of the present study, the "ankerites" within the primary-ores produced from the Golden Mile are believed to be weakly/moderately-ferroan varieties, and not "Fe-rich".<sup>6</sup>

### A1.3.3 pH-Buffering Properties

The pH-Buffering properties of selected samples were determined via a Metrohm® 736 Titrino auto-titrator, and 0.05 M-H<sub>2</sub>SO<sub>4</sub>.

The auto-titrations comprised regular addition of the H<sub>2</sub>SO<sub>4</sub> reagent to monotonically decrease the pH of the test-suspensions to 3.0.<sup>7</sup> The Start-pH values of the suspensions were *c.* 9. Under the testwork conditions employed, the H<sub>2</sub>SO<sub>4</sub>-addition rates correspond to SORs 'of-the-order' 10<sup>5</sup>-10<sup>6</sup> mg SO<sub>4</sub>/kg/week (i.e. 'of-the-order' 5,000-50,000 kg H<sub>2</sub>SO<sub>4</sub>/tonne/year), and so represent very-rapid rates of acid addition.

Further details of the auto-titrations are presented in the laboratory reports (Appendix B).

### A1.4 NAPP Calculations

The NAPP values of the samples were calculated from the corresponding Total-S, SO<sub>4</sub>-S and ANC values, assuming that all of the Non-Sulphate-S occurs in the form of

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<sup>6</sup> This statement is based on Dr. Graeme Campbell's experience with numerous local gold-mines where "ankerites" occur, and their composition determined via electron-microprobe analyses.

<sup>7</sup> It should be noted that, in titrating to a pH=3.0 end-point, any Fe(II) released through acid attack of ferroan-silicates and -carbonates is not quantitatively oxidised to Fe(III). Furthermore, under the conditions employed in the auto-titration, the equivalent of *c.* 0.5 kg H<sub>2</sub>SO<sub>4</sub>/tonne was required to decrease the pH of the "solution-only" (i.e. without tailings-solids sample) to pH=3.0. No correction was made for such "electrolyte-consumption" of the 0.05 M-H<sub>2</sub>SO<sub>4</sub> titrant.

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pyrite. The NAPP calculations serve as a starting point in the assessment of the acid-formation potential of sulphide-bearing materials.

The complete oxidation of pyrite may be described by:



If the Sulphide-S (in %S) occurs only as pyrite, then the amount of acid (in kg H<sub>2</sub>SO<sub>4</sub>/tonne) produced through the complete oxidation of pyrite is given by 30.6 x %S.

Note: The above treatment of oxidation-reaction stoichiometry is restricted to oxidation by 'atmospheric-O<sub>2</sub>' which is the dominant oxidant at circum-neutral-pH. A different oxidation-stoichiometry applies under acidic conditions (e.g. pH less than 3-4) where soluble-Fe(III) forms prevail, and then function as the chief oxidant.

## **A2.0 WATER-EXTRACTION TESTWORK**

The tailings-solids samples were subjected to Water-Extraction Tests by preparing sample-slurries using deionised-water, and a solid:solution ratio of 1:10 (w/w).

The sample-slurries were "bottle-rolled" for *c.* 1 day, and after leaving to stand to allow "fines" to settle, the slurry-waters were vacuum-filtered (0.45-µm-membrane), and analysed for Cl and SO<sub>4</sub>.

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**APPENDIX B**

**LABORATORY REPORTS**



16 December, 2002

Graeme Campbell & Associates Pty Ltd  
 Attn: Dr G Campbell  
 PO Box 247  
 BRIDGETOWN WA 6255

Our Reference: 68676  
 Your Reference: GCA0233  
 NATA Accreditation: 2562(1705)

Dear Sir

On the 15<sup>th</sup> of November 2002 you forwarded testwork instructions for nine (9) tailings solids samples (GCA4758-GCA4766) which were received on the 19<sup>th</sup> of November at our laboratory. Additional instructions were also received on the 3<sup>rd</sup> of December

The samples were received as 1-2 kg of moist tailings solids which were initially oven dried at 40°C prior to crushing to a nominal 2mm particle size. A sub sample of the crushed material was then dried at 105°C prior to pulping to a nominal -75µm particle size for total sulphur analysis. The loss in weight of the 40°C dried sample at 105°C is reported as the moisture content. The crushed sample not retained for testwork was forwarded to the GCA Testing Laboratory as requested.

Results of all testwork performed follow:

Sample Number	Moisture @ 105°C (%w/w)	pH (pH Units)	Conductivity (1:2) (µS/cm)	Total Carbon (% w/w)	Total Organic Carbon (% w/w)
GCA4758	0.43	9.0	56000	0.61	0.07
GCA4759	5.47	9.0	49000	0.52	0.06
GCA4760	6.54	8.9	39000	0.53	0.06
GCA4761	4.00	8.9	44000	0.85	0.08
GCA4762	1.90	8.8	37000	NA	-
GCA4763	2.61	10.0	42000	NA	-
GCA4764	1.77	9.5	32000	NA	-
GCA4765	3.87	9.0	18000	NA	-
GCA4766	4.79	8.7(8.7)	16000(16000)	1.6	0.20

Sample Number	Water Soluble Chloride, Cl (%w/w)	Water Soluble Sulphate-Sulphur, SO <sub>4</sub> -S (%w/w)	Total Sulphur S (% w/w)	Sulphate Sulphur SO <sub>4</sub> -S (Na <sub>2</sub> CO <sub>3</sub> ) (% w/w)	Carbonate Carbon CO <sub>3</sub> -C (% w/w)
GCA4758	4.7	0.79	23.2	1.2	0.55
GCA4759	3.6	0.76	20.4	1.4	0.46
GCA4760	3.0	0.78	21.0	1.2	0.48
GCA4761	3.4	0.73	11.6	2.2	0.78
GCA4762	2.7	0.72	7.7	1.6	-
GCA4763	3.0	0.85	15.7	1.2	-
GCA4764	2.3	0.69	9.5	1.5	-
GCA4765	2.0	0.71	11.1	1.6	-
GCA4766	1.5	0.68	14.9	0.78(0.75)	1.4

CLIENT: Graeme Campbell & Associates Pty Ltd      OUR REFERENCE: 68676  
 PROJECT NO: GCA0233

**NOTES:**

1. Moisture content was determined on the 40°C dried sample as loss in weight after 24 hours drying at 105°C and is reported on that basis. Bracketed results are from duplicate analysis.
2. pH and conductivity were determined on a 1:2 w/w 40°C dried crushed sample to deionised water extract after 24 hours ambient aging.
3. Sulphate sulphur was determined on 40°C dried crushed sample by Na<sub>2</sub>CO<sub>3</sub> extraction. BaSO<sub>4</sub> precipitation with results reported back to the 105°C dried sample basis. Bracketed result from duplicate analysis.
4. Total sulphur, total carbon and total organic carbon (noncarbonate or acid insoluble carbon) were determined on dried pulped sample by LECO induction furnace, IR detection, and is reported on that basis. This testwork was performed by SGS Analabs, Welshpool, report numbers WM067281 and WM067542.
5. Water soluble sulphate and chloride were determined from a 1:10w/w 40°C dried crushed sample to deionised water extract after overnight bottle rolling and standing 24 hours and are reported back to the 105°C dried sample basis.

**Acid Neutralisation Capacity (ANC):**

Sample Number	Fizz Rating	Sample Weight (g)	Titre NaOH (mL)	Normality HCl/NaOH (N)	Initial Effervescence	Effervescence on Warming	ANC Solution pH	ANC (kg H <sub>2</sub> SO <sub>4</sub> /tonne)
GCA4758	0	2.0010	18.10	0.5	Slight	Nil	0.8	83*
GCA4759	0	2.0106	19.00	0.5	Slight	Nil	0.8	75*
GCA4760	1	2.0027	19.80	0.5	Slight	Nil	0.8	66*
GCA4761	2-3	2.0031	17.80	0.5	Moderate	Nil	0.9	90*
GCA4762	2-3	2.0083	20.50	0.5	Moderate	Nil	0.8	54*
GCA4762 Rpt	2-3	2.0026	20.50	0.5	Moderate	Nil	0.9	54*
GCA4763	3	2.0193	14.60	0.5	Moderate	Nil	1.0	130*
GCA4764	3	2.0092	19.00	0.5	Moderate	Nil	0.8	73*
GCA4765	3	2.0070	16.10	0.5	Moderate	Nil	0.9	110*
GCA4766	3	2.0097	16.70	0.5	Moderate	Nil	0.8	100*
ANC Std200	-	1.0086	17.10	0.5	-	-	0.6	189

**NOTES:**

1. Acid neutralisation capacity was determined on 40°C dried crushed sample with results corrected back to the 105°C sample weight basis. Unless otherwise stated, 25mL of HCl is used. Reagent blank titre of 0.5N NaOH was 24.80mL.
2. Samples marked with an asterisk (\*) gave a pH drop to approximately 3 to 4 on addition of the hydrogen peroxide. Two drops of hydrogen peroxide are added to each sample as the pH=7 endpoint is approached to oxidise any ferrous iron.
3. ANC Std200 is an internally produced standard of CaCO<sub>3</sub> and quartz pulped to a nominal 75µm particle size which has a nominal ANC of 200kg of H<sub>2</sub>SO<sub>4</sub>/tonne.
4. This procedure is based on Sobek et al, 1978.

CLIENT: Graeme Campbell & Associates Pty Ltd      OUR REFERENCE: 68676  
PROJECT NO: GCA0233

It is noted that the Na<sub>2</sub>CO<sub>3</sub> extraction procedure is not covered by our terms of NATA accreditation.

Yours faithfully,

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PETER BAMFORD  
Manager Laboratory Services

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JANICE VENNING  
Manager, Perth

*This report supersedes our preliminary results sent by facsimile on the 11/12/2002.*



20 December, 2002

Graeme Campbell & Associates Pty Ltd  
Attn: Dr G Campbell  
PO Box 247  
BRIDGETOWN WA 6255

Our Reference: 68916  
Your Reference: GCA0233  
NATA Accreditation: 2562(1705)

Dear Sir

On the 3<sup>rd</sup> of December 2002 you forwarded testwork instructions for a tailings solid sample (GCA4668) previously received on the 1<sup>st</sup> of October 2002 at our laboratory. Due to the time since receipt of the sample, the as received sample was re tested for moisture content the result of which is reported below. Results of all testwork performed follow:

Sample Number	pH (pH Units)	Conductivity ( $\mu$ S/cm)	Water Soluble Chloride (%w/w)	Water Soluble Sulphate Sulphur $\text{SO}_4\text{-S}$ (%w/w)	Total Sulphur S (% w/w)	Sulphate Sulphur $\text{SO}_4\text{-S}$ ( $\text{Na}_2\text{CO}_3$ ) (% w/w)
GCA4668	8.7	34000	1.6	1.1	2.3	2.0
GCA4668 Rpt	8.7	32000	-	-	-	2.4

**NOTES:**

1. *Moisture content was determined on the as received sample as loss in weight after 24 hours drying at 105°C and is reported on that basis.*
2. *pH and conductivity were determined on a 1:2 w/w dry weight corrected as received sample to deionised water extract after 24 hours ambient aging.*
3. *Total sulphur was determined on the original dried pulped sample by LECO induction furnace, IR detection, and is reported on that basis. This testwork was performed by SGS Analabs, Welshpool, report number WM067625.*
4. *Sulphate sulphur was determined on an as received sample by  $\text{Na}_2\text{CO}_3$  extraction,  $\text{BaSO}_4$  precipitation with results reported back to the 105°C dried sample basis.*
5. *Water soluble chloride and sulphate were determined from a 1:10 as received sample to deionised water extract (bottle roll for 24 hours) and are reported back to the 105°C dried sample basis.*

CLIENT: Graeme Campbell & Associates Pty Ltd      OUR REFERENCE: 68916  
PROJECT NO: GCA0233

The Na<sub>2</sub>CO<sub>3</sub> extraction procedure and the bottle rolling procedure are not covered by our terms of NATA accreditation.

Yours faithfully,

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PETER BAMFORD  
Manager Laboratory Services

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JANICE VENNING  
Manager, Perth

*This report supersedes our preliminary results sent by facsimile on the 11 December 2002.*

## pH-BUFFERING TESTWORK (GCA4758)

Cumulative Volume of Acid Added (mL)	Cumulative Acid Consumption (kg H <sub>2</sub> SO <sub>4</sub> /tonne)	pH	Cumulative Volume of Acid Added (mL)	Cumulative Acid Consumption (kg H <sub>2</sub> SO <sub>4</sub> /tonne)	pH
0.00	0.0	9.2	16.40	54	4.9
0.40	1.3	8.7	16.80	55	4.8
0.80	2.6	8.5	17.20	57	4.7
1.20	4.0	8.4	17.60	58	4.6
1.60	5.3	8.3	18.00	59	4.5
2.00	6.6	8.3	18.40	61	4.4
2.40	7.9	8.2	18.80	62	4.3
2.80	9.2	8.2	19.20	63	4.2
3.20	11	8.1	19.60	65	4.1
3.60	12	8.1	20.00	66	4.0
4.00	13	8.0	20.40	67	3.9
4.40	15	8.0	20.80	69	3.8
4.80	16	7.9	21.20	70	3.7
5.20	17	7.9	21.60	71	3.6
5.60	18	7.8	22.00	73	3.5
6.00	20	7.8	22.40	74	3.5
6.40	21	7.7	22.80	75	3.4
6.80	22	7.6	23.20	77	3.3
7.20	24	7.5	23.60	78	3.3
7.60	25	7.5	24.00	79	3.2
8.00	26	7.4	24.40	81	3.2
8.40	28	7.3	24.80	82	3.2
8.80	29	7.2	25.20	83	3.1
9.20	30	7.2	25.60	84	3.1
9.60	32	7.1	26.00	86	3.1
10.00	33	7.0	26.40	87	3.0
10.40	34	6.9			
10.80	36	6.8			
11.20	37	6.7			
11.60	38	6.6			
12.00	40	6.5			
12.40	41	6.4			
12.80	42	6.2			
13.20	44	6.1			
13.60	45	5.9			
14.00	46	5.7			
14.40	48	5.6			
14.80	49	5.4			
15.20	50	5.3			
15.60	51	5.2			
16.00	53	5.0			

Note: Titration performed using a Metrohm® 736 Titrino auto-titrator, and 0.05 M-H<sub>2</sub>SO<sub>4</sub>. Equilibration time between titrant additions was 15 minutes. 1.5 g of moist sample (= 1.5 g dry-solids) initially dispersed in 150 mL of deionised-water. Test mixture in contact with air, at ambient temperature, and continuously stirred.

Calibration of pH-Glass Electrode:

Immediately prior to titration: asymmetry potential = -14 mV (pH=7.00); slope-point = 139 mV (pH=4.00); 97.2 % of Nernstian response for 25 °C.

Immediately following titration: pH=7.00 buffer read pH=7.06 and pH=4.00 buffer read pH=4.07. These discrepancies represent drift in pH-Glass electrode response during course of auto-titration.

Dr GD Campbell

5 December 2002



## pH-BUFFERING TESTWORK (GCA4759)

Cumulative Volume of Acid Added (mL)	Cumulative Acid Consumption (kg H <sub>2</sub> SO <sub>4</sub> /tonne)	pH	Cumulative Volume of Acid Added (mL)	Cumulative Acid Consumption (kg H <sub>2</sub> SO <sub>4</sub> /tonne)	pH
0.00	0.0	8.9	16.40	54	4.9
0.40	1.3	8.5	16.80	55	4.8
0.80	2.6	8.2	17.20	57	4.7
1.20	4.0	8.1	17.60	58	4.6
1.60	5.3	8.1	18.00	59	4.5
2.00	6.6	8.0	18.40	61	4.5
2.40	7.9	8.0	18.80	62	4.4
2.80	9.2	7.9	19.20	63	4.3
3.20	11	7.9	19.60	65	4.2
3.60	12	7.9	20.00	66	4.1
4.00	13	7.8	20.40	67	4.0
4.40	15	7.8	20.80	69	3.9
4.80	16	7.7	21.20	70	3.8
5.20	17	7.6	21.60	71	3.7
5.60	18	7.6	22.00	73	3.6
6.00	20	7.5	22.40	74	3.6
6.40	21	7.4	22.80	75	3.5
6.80	22	7.4	23.20	77	3.4
7.20	24	7.3	23.60	78	3.3
7.60	25	7.2	24.00	79	3.3
8.00	26	7.1	24.40	81	3.2
8.40	28	7.1	24.80	82	3.2
8.80	29	7.0	25.20	83	3.1
9.20	30	6.9	25.60	84	3.1
9.60	32	6.9	26.00	86	3.1
10.00	33	6.8	26.40	87	3.0
10.40	34	6.7			
10.80	36	6.7			
11.20	37	6.6			
11.60	38	6.5			
12.00	40	6.4			
12.40	41	6.3			
12.80	42	6.2			
13.20	44	6.1			
13.60	45	5.9			
14.00	46	5.8			
14.40	48	5.6			
14.80	49	5.5			
15.20	50	5.3			
15.60	51	5.2			
16.00	53	5.0			

Note: Titration performed using a Metrohm® 736 Titrino auto-titrator, and 0.05 M-H<sub>2</sub>SO<sub>4</sub>. Equilibration time between titrant additions was 15 minutes. 1.6 g of moist sample (= 1.5 g dry-solids) initially dispersed in 150 mL of deionised-water. Test mixture in contact with air, at ambient temperature, and continuously stirred.

Calibration of pH-Glass Electrode:

Immediately prior to titration: asymmetry potential = -21 mV (pH=7.00); slope-point = 154 mV (pH=4.00); 98.9 % of Nernstian response for 25 °C.

Immediately following titration: pH=7.00 buffer read pH=7.02 and pH=4.00 buffer read pH=4.03. These discrepancies represent drift in pH-Glass electrode response during course of auto-titration.

Dr GD Campbell

6 December 2002



## pH-BUFFERING TESTWORK (GCA4760)

Cumulative Volume of Acid Added (mL)	Cumulative Acid Consumption (kg H <sub>2</sub> SO <sub>4</sub> /tonne)	pH	Cumulative Volume of Acid Added (mL)	Cumulative Acid Consumption (kg H <sub>2</sub> SO <sub>4</sub> /tonne)	pH
0.00	0.0	8.7	16.40	54	3.7
0.40	1.3	8.3	16.80	55	3.6
0.80	2.6	8.0	17.20	57	3.5
1.20	4.0	7.9	17.60	58	3.5
1.60	5.3	7.8	18.00	59	3.4
2.00	6.6	7.7	18.40	61	3.4
2.40	7.9	7.6	18.80	62	3.3
2.80	9.2	7.5	19.20	63	3.3
3.20	11	7.4	19.60	65	3.2
3.60	12	7.3	20.00	66	3.2
4.00	13	7.2	20.40	67	3.1
4.40	15	7.1	20.80	69	3.1
4.80	16	7.0	21.20	70	3.1
5.20	17	6.9	21.60	71	3.0
5.60	18	6.9	22.00	73	3.0
6.00	20	6.8			
6.40	21	6.7			
6.80	22	6.6			
7.20	24	6.5			
7.60	25	6.4			
8.00	26	6.2			
8.40	28	6.1			
8.80	29	6.0			
9.20	30	5.8			
9.60	32	5.6			
10.00	33	5.4			
10.40	34	5.3			
10.80	36	5.1			
11.20	37	5.0			
11.60	38	4.9			
12.00	40	4.8			
12.40	41	4.7			
12.80	42	4.6			
13.20	44	4.5			
13.60	45	4.4			
14.00	46	4.3			
14.40	48	4.2			
14.80	49	4.1			
15.20	50	4.0			
15.60	51	3.9			
16.00	53	3.8			

**Note:** Titration performed using a Metrohm® 736 Titrino auto-titrator, and 0.05 M-H<sub>2</sub>SO<sub>4</sub>. Equilibration time between titrations was 15 minutes. 1.6 g of moist sample (= 1.5 g dry-solids) initially dispersed in 150 mL of deionised-water. Test mixture in contact with air, at ambient temperature, and continuously stirred.

Calibration of pH-Glass Electrode:

Immediately prior to titration: asymmetry potential = -22 mV (pH=7.00); slope-point = 158 mV (pH=4.00); 101.6 % of Nernstian response for 25 °C.

Immediately following titration: pH=7.00 buffer read pH=7.02 and pH=4.00 buffer read pH=4.04. These discrepancies represent drift in pH-Glass electrode response during course of auto-titration.

Dr GD Campbell

7 December 2002



## pH-BUFFERING TESTWORK (GCA4668)

Cumulative Volume of Acid Added (mL)	Cumulative Acid Consumption (kg H <sub>2</sub> SO <sub>4</sub> /tonne)	pH	Cumulative Volume of Acid Added (mL)	Cumulative Acid Consumption (kg H <sub>2</sub> SO <sub>4</sub> /tonne)	pH
0.00	0.0	9.3	22.00	11	4.8
0.40	0.2	8.8	22.40	11	4.7
0.80	0.4	8.6	22.80	12	4.7
1.20	0.6	8.4	23.20	12	4.6
1.60	0.8	8.3	23.60	12	4.6
2.00	1.0	8.2	24.00	12	4.5
2.40	1.2	8.2	24.40	12	4.5
2.80	1.4	8.2	24.80	13	4.7
3.20	1.6	8.0	25.20	13	4.7
3.60	1.8	8.0	25.60	13	4.6
4.00	2.0	7.8	26.00	13	4.6
4.40	2.2	7.8	26.40	13	4.5
4.80	2.4	7.7	26.80	14	4.4
5.20	2.7	7.6	27.20	14	4.4
5.60	2.9	7.6	27.60	14	4.3
6.00	3.1	7.5	28.00	14	4.2
6.40	3.3	7.5	28.40	14	4.2
6.80	3.5	7.4	28.80	15	4.1
7.20	3.7	7.3	29.20	15	4.1
7.60	3.9	7.4	29.60	15	4.1
8.00	4.1	7.3	30.00	15	4.0
8.40	4.3	7.3	30.40	16	4.0
8.80	4.5	7.2	30.80	16	3.9
9.20	4.7	7.2	31.20	16	3.9
9.60	4.9	7.2	31.60	16	3.8
10.00	5.1	7.1	32.00	16	3.8
10.40	5.3	7.1	32.40	17	3.8
10.80	5.5	7.0	32.80	17	3.7
11.20	5.7	7.0	33.20	17	3.7
11.60	5.9	6.9	33.60	17	3.7
12.00	6.1	6.9	34.00	17	3.6
12.40	6.3	6.8	34.40	18	3.6
12.80	6.5	6.7	34.80	18	3.6
13.20	6.7	6.6	35.20	18	3.5
13.60	6.9	6.5	35.60	18	3.5
14.00	7.1	6.4	36.00	18	3.4
14.40	7.3	6.4	36.40	19	3.4
14.80	7.5	6.2	36.80	19	3.4
15.20	7.8	6.1	37.20	19	3.4
15.60	8.0	6.1	37.60	19	3.3
16.00	8.2	6.0	38.00	19	3.3
16.40	8.4	5.9	38.40	20	3.3
16.80	8.6	5.8	38.80	20	3.2
17.20	8.8	5.6	39.20	20	3.2
17.60	9.0	5.5	39.60	20	3.2
18.00	9.2	5.4	40.00	20	3.1
18.40	9.4	5.3	40.40	21	3.1
18.80	9.6	5.2	40.80	21	3.1
19.20	9.8	5.1	41.20	21	3.0
19.60	10	5.1	41.60	21	3.0
20.00	10	5.0			
20.40	10	5.0			
20.80	11	4.9			
21.20	11	4.8			
21.60	11	4.8			

Note: Titration performed using a Metrohm<sup>®</sup> 736 Titrino auto-titrator, and 0.05 M-H<sub>2</sub>SO<sub>4</sub>. Equilibration time between titrant additions was 15 minutes. 10.6 g of moist sample (=9.7 g dry-solids) initially dispersed in 150 mL of deionised-water. Test mixture in contact with air, at ambient temperature, and continuously stirred.

Calibration of pH-Glass Electrode:

Immediately prior to titration: asymmetry potential = -13 mV (pH=7.00); slope-point = 160 mV (pH=4.00); 97.9 % of Nernstian response for 25 °C.

Immediately following titration: pH=7.00 buffer read pH=7.03 and pH=4.00 buffer read pH=4.05. These discrepancies represent drift in pH-Glass electrode response during course of auto-titration.

Dr GD Campbell  
25 November 2002



## pH-BUFFERING TESTWORK (GCA4668)

Cumulative Volume of Acid Added (mL)	Cumulative Acid Consumption (kg H <sub>2</sub> SO <sub>4</sub> /tonne)	pH	Cumulative Volume of Acid Added (mL)	Cumulative Acid Consumption (kg H <sub>2</sub> SO <sub>4</sub> /tonne)	pH
0.00	0.0	9.6	19.20	12	5.4
0.40	0.2	9.1	19.60	12	5.2
0.80	0.5	8.8	20.00	12	5.1
1.20	0.7	8.6	20.40	13	5.1
1.60	1.0	8.4	20.80	13	5.0
2.00	1.2	8.4	21.20	13	4.9
2.40	1.5	8.3	21.60	13	4.9
2.80	1.7	8.2	22.00	14	4.8
3.20	2.0	8.2	22.40	14	4.7
3.60	2.2	8.1	22.80	14	4.7
4.00	2.5	8.0	23.20	14	4.6
4.40	2.7	8.0	23.60	15	4.6
4.80	3.0	7.9	24.00	15	4.5
5.20	3.2	7.9	24.40	15	4.5
5.60	3.5	7.8	24.80	15	4.4
6.00	3.7	7.7	25.20	16	4.4
6.40	4.0	7.7	25.60	16	4.3
6.80	4.2	7.6	26.00	16	4.3
7.20	4.5	7.6	26.40	16	4.2
7.60	4.7	7.5	26.80	17	4.2
8.00	5.0	7.5	27.20	17	4.1
8.40	5.2	7.4	27.60	17	4.1
8.80	5.5	7.4	28.00	17	4.0
9.20	5.7	7.3	28.40	18	4.0
9.60	6.0	7.3	28.80	18	3.9
10.00	6.2	7.2	29.20	18	3.9
10.40	6.4	7.2	29.60	18	3.8
10.80	6.7	7.2	30.00	19	3.8
11.20	6.9	7.1	30.40	19	3.7
11.60	7.2	7.1	30.80	19	3.7
12.00	7.4	7.0	31.20	19	3.6
12.40	7.7	7.0	31.60	20	3.6
12.80	7.9	6.9	32.00	20	3.5
13.20	8.2	6.9	32.40	20	3.5
13.60	8.4	6.8	32.80	20	3.5
14.00	8.7	6.8	33.20	21	3.4
14.40	8.9	6.7	33.60	21	3.4
14.80	9.2	6.7	34.00	21	3.3
15.20	9.4	6.6	34.40	21	3.3
15.60	9.7	6.5	34.80	22	3.3
16.00	9.9	6.4	35.20	22	3.3
16.40	10	6.2	35.60	22	3.2
16.80	10	6.1	36.00	22	3.2
17.20	11	6.0	36.40	23	3.2
17.60	11	5.9	36.80	23	3.2
18.00	11	5.7	37.20	23	3.1
18.40	11	5.6	37.60	23	3.1
18.80	12	5.5	38.00	24	3.0

Note: Titration performed using a Metrohm<sup>®</sup> 736 Titrimo auto-titrator, and 0.05 M-H<sub>2</sub>SO<sub>4</sub>. Equilibration time between titrant additions was 15 minutes. 10.4 g of moist sample (=8.1 g dry-solids) initially dispersed in 150 mL of deionised-water. Test mixture in contact with air, at ambient temperature, and continuously stirred.

Calibration of pH-Glass Electrode:

Immediately prior to titration: asymmetry potential = -15 mV (pH=7.00); slope-point = 160 mV (pH=4.00); 98.3 % of Nernstian response for 25 °C.

Immediately following titration: pH=7.00 buffer read pH=7.03 and pH=4.00 buffer read pH=4.05. These discrepancies represent drift in pH-Glass electrode response during course of auto-titration.

Dr GD Campbell

20 November 2002



## pH-BUFFERING TESTWORK (GCA4766)

Cumulative Volume of Acid Added (mL)	Cumulative Acid Consumption (kg H <sub>2</sub> SO <sub>4</sub> /tonne)	pH	Cumulative Volume of Acid Added (mL)	Cumulative Acid Consumption (kg H <sub>2</sub> SO <sub>4</sub> /tonne)	pH
0.00	0.0	8.8	22.40	58	5.4
0.40	1.0	8.4	22.80	59	5.3
0.80	2.1	8.1	23.20	60	5.3
1.20	3.1	8.0	23.60	61	5.3
1.60	4.2	7.9	24.00	62	5.2
2.00	5.2	7.8	24.40	63	5.2
2.40	6.2	7.7	24.80	64	5.1
2.80	7.3	7.7	25.20	66	5.1
3.20	8.3	7.6	25.60	67	5.0
3.60	9.4	7.6	26.00	68	4.9
4.00	10	7.6	26.40	69	4.9
4.40	11	7.5	26.80	70	4.8
4.80	12	7.5	27.20	71	4.8
5.20	14	7.4	27.60	72	4.7
5.60	15	7.4	28.00	73	4.6
6.00	16	7.4	28.40	74	4.5
6.40	17	7.3	28.80	75	4.5
6.80	18	7.3	29.20	76	4.4
7.20	19	7.2	29.60	77	4.3
7.60	20	7.2	30.00	78	4.3
8.00	21	7.1	30.40	79	4.2
8.40	22	7.1	30.80	80	4.1
8.80	23	7.0	31.20	81	4.1
9.20	24	7.0	31.60	82	4.0
9.60	25	6.9	32.00	83	3.9
10.00	26	6.9	32.40	84	3.9
10.40	27	6.8	32.80	85	3.8
10.80	28	6.7	33.20	86	3.8
11.20	29	6.7	33.60	87	3.7
11.60	30	6.6	34.00	88	3.7
12.00	31	6.5	34.40	89	3.7
12.40	32	6.4	34.80	90	3.6
12.80	33	6.4	35.20	92	3.6
13.20	34	6.3	35.60	93	3.6
13.60	35	6.3	36.00	94	3.5
14.00	36	6.2	36.40	95	3.5
14.40	37	6.2	36.80	96	3.5
14.80	38	6.1	37.20	97	3.4
15.20	40	6.1	37.60	98	3.4
15.60	41	6.1	38.00	99	3.4
16.00	42	6.0	38.40	100	3.4
16.40	43	6.0	38.80	101	3.3
16.80	44	5.9	39.20	102	3.3
17.20	45	5.9	39.60	103	3.3
17.60	46	5.8	40.00	104	3.2
18.00	47	5.8	40.40	105	3.2
18.40	48	5.8	40.80	106	3.2
18.80	49	5.7	41.20	107	3.2
19.20	50	5.7	41.60	108	3.1
19.60	51	5.6	42.00	109	3.1
20.00	52	5.6	42.40	110	3.1
20.40	53	5.6	42.80	111	3.1
20.80	54	5.5	43.20	112	3.1
21.20	55	5.5	43.60	113	3.0
21.60	56	5.4	44.00	114	3.0
22.00	57	5.4	44.40	115	3.0

Note: Titration performed using a Metrohm<sup>®</sup> 736 Titrimo auto-titrator, and 0.05 M-H<sub>2</sub>SO<sub>4</sub>. Equilibration time between titrant additions was 15 minutes. 2.0 g of moist sample (= 1.9 g dry-solids) initially dispersed in 150 mL of deionised-water. Test mixture in contact with air, at ambient temperature, and continuously stirred.

Calibration of pH-Glass Electrode:

Immediately prior to titration: asymmetry potential = -20 mV (pH=7.00); slope-point = 158 mV (pH=4.00); 100.0 % of Nernstian response for 25 °C.

Immediately following titration: pH=7.00 buffer read pH=7.04 and pH=4.00 buffer read pH=4.06. These discrepancies represent drift in pH-Glass electrode response during course of auto-titration.

Dr GD Campbell

8 December 2002