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ECOLOGIA ENVIRONMENT

ON

ACID SULFATE SOIL TESTING BY SUSPENSION PEROXIDE OXIDATION COMBINED ACIDITY AND SULFATE METHOD (SPOCAS)

According to the:
Acid Sulfate Soils Laboratory Methods Guidelines 2004,
Department of Natural Resources and Mines, Queensland

CHEMISTRY CENTRE REPORT NO 06A289
8 DECEMBER 2006

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SAMPLE IDENTIFICATION

Twelve samples of soil at ambient temperature were received for analysis on 28/11/06. On receipt at the Chemistry Centre the samples were identified and allocated a Laboratory Number.

TEST METHODS

The test methods of the Acid Sulfate Soils Laboratory Methods Guidelines manual were used in this work, specifically Method Code 23 – SPOCAS (Suspension Peroxide Oxidation Combined Acidity and Sulfate).

The SPOCAS method is a standardised set of procedures useful in assessing the environmental impact of soils suspected of containing pyrite and other iron sulfides, which might lead to an acid sulfate soil problem if disturbed.

SAMPLE PREPARATION

The samples were dried at 80 ^{o}C for 48 hours. Coarse material >2 mm was removed by sieving. The portion of sample greater than 2mm is indicated in the table below. The <2 mm fraction was ground to <150 microns. Sub samples of the milled homogenised material were then subjected to chemical tests. All results are reported on a dry weight basis.

Lab No	Client ID	>2mm
		%
06A289/001	Koolanooka 15-16 KNRC 469	62
06A289/002	Koolanooka 3-4 KNRC 472	21
06A289/003	Koolanooka- Waste dump material	46
06A289/004	Koolanooka- Waste dump material	43
06A289/005	Mungada East 40-41 BHRC 354	23
06A289/006	Mungada East 20-21 BHRC 339	15
06A289/007	Mungada East 20-21 BHRC 354	41
06A289/008	Mungada East 10-11 BHRC 339	10
06A289/009	Mungada West 70 BHRC 015	26
06A289/010	Mungada West 42 BHRC 013	63
06A289/011	Mungada West- pit sample 1	66
06A289/012	Mungada West- pit sample 2	60

TEST PROCEDURES

SPOCAS METHOD

Step 1: Determination of Potassium Chloride Extractable Sulfur (S_{KCl} %) and Total Actual Acidity (TAA)

In this procedure the sample is extracted with potassium chloride solution. The extraction with potassium chloride is used to determine soluble and absorbed sulfur

(non-sulfidic sulfur) and the titratable actual acidity of the sample (**TAA**). The sulfur is determined using inductively coupled plasma atomic emission spectrometry (ICPAES).

The pH, acidity, and sulfur content of the resultant solution are reported as **pH_{KCl}, TAA_{KCl},** and **S_{KCl}** respectively.

Step 2: Determination of the Peroxide Oxidation Sulfur (S_P%), and Titratable Peroxide Acidity (TPA).

This step involves oxidation of the sample with hydrogen peroxide to produce maximum acidity from any reduced sulfidic material. The sulfur content (**S_P%**), the Titratable Peroxide Acidity (**TPA**), and pH (**pHox**) of the oxidised solution are determined. **S_P%** will include the soluble, absorbed, and sulfide, sulfur species.

Step 3: Determination of Retained Acidity

Existing acidity in acid sulfate soils includes **'actual'** acidity (TAA) and **'retained'** acidity (acidity stored in largely insoluble iron and aluminium sulfate minerals). A dilute HCl extraction performed on the washed soil residue after peroxide digestion will give the residual acid soluble sulfur (**S_{RAS}**) from which the retained acidity can be calculated. This acidity is expressed as **a-S_{RAS}**. For soil samples with pH_{KCl}<4.5 the S_{RAS} **must** be determined.

Step 4: Determination of the Excess Acid Neutralising Capacity (ANCe)

This determination is optional depending on the peroxide solution pH.
If the solution pH after the peroxide step is >6.5, the material may have an acid neutralization capacity. The fine grinding of the sample for analysis will lead to an over estimation of the effective acid neutralising capacity and an appropriate safety factor must be applied.

Step 5: Peroxide Oxidizable Sulfur (S_{POS})

This step involves calculating the differences between the extracts from Step 2 and Step 1. The peroxide oxidizable sulfur is used to predict the potential acid risk from non-oxidised sulfur compounds.

Peroxide oxidizable sulfur: $S_{POS} = (S_P - S_{KCl})\%$

RESULTS

See attached spreadsheet 06A289 SPOCAS

ACID BASE ACCOUNTING (ABA)

The acid base accounting approach is used to predict net acidity from the oxidation of sulfidic material. The SPOCAS method is in essence a self-contained ABA. The **TPA**

result represents a measure of the net acidity, effectively equivalent to the sum of the soil's potential sulfidic activity and actual acidity **(TAA)** less any neutralising capacity of the sample. Where the pH_{KCl} is <4.5 then the residual acid soluble sulfur (**S_{RAS}**) component of SPOCAS should be done, since the TPA does not measure **retained acidity**. In soils that are self neutralising (i.e. TPA=0), then the HCL titration step in SPOCAS allows calculation of the **excess acid neutralising capacity** (**ANC_E**)

GUIDE TO INTERPRETATION OF SPOCAS TESTWORK

Interpretation of results from SPOCAS test methods involves determination of action criteria and comparison of the test results with the criteria. The NSW ASSMAC has published Action Criteria as follows:

Type of Material		**Action Criteria, <1,000 tonnes**		**Action Criteria, >1,000 tonnes**	
Texture	**Approx Clay Content (%<0.002 mm)**	**Sulfur Trail S_{POS} %**	**Acid Trail TPA mole H^+/t**	**Sulfur Trail S_{POS} %**	**Acid Trail TPA mole H^+/t**
Coarse e.g. sands	≤5	0.03	18	0.03	18
Medium e.g. loams/light clays	5 – 40	0.06	36	0.03	18
Fine clays/silts	≥40	0.1	62	0.03	18

According to the NSW ASSMAC, exceedance of these criteria indicates risk of an acid sulfate soil issue and the need for an acid sulfate soil management plan with development approval.

INTERPRETATION OF RESULTS OF SPOCAS TESTS FROM THIS WORK

No single method, including SPOCAS, will provide all the answers to the complex chemistry involved in reactions of acid sulfate soils. However results from SPOCAS test procedures will provide guidance to identification of potential ASS issues.

All samples are within guideline values. The fine grinding of the samples for analysis will result in a higher ANCe value (and a lower TPA value) than would measured under field conditions.

RECOMMENDATIONS

No sample treatment is indicated.

REFERENCES

1 **Guidelines for Sampling and Analysis of Lowland Acid Sulfate Soils (ASS) in Queensland 1998** Queensland Acid Sulfate Soils Investigation Team (QASSIT) October 1998, C.R.Ahern, M.R. Ahern, and B Powell.

2. **Acid Sulfate Soils Laboratory Methods Guidelines 2004**, Department of Natural Resources and Mines, Queensland, 2004.

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8 December 2006