

## **LABORATORY PROTOCOLS FOR TESTING DRILLING WASTES AND RECEIVING SOILS**

John Ashworth<sup>1</sup> and Ha Du<sup>2</sup>

### **ABSTRACT**

We suggest procedures for bench-scale mixing of samples of soils and waste materials from oil and gas drilling leases, intended to replicate on-site volume mix-ratios. We also discuss methods for testing coloured drilling waste liquids using the Microtox<sup>TM</sup> bioassay. In each case our aim is to reduce inter-laboratory variability in test results.

### **INTRODUCTION**

Under the current G-50 guidelines (AEUB, 1996) that govern disposal of drilling wastes in Alberta, any waste disposed of on-site must for soil stability purposes be mixed with at least 3 times its volume of soil. Mix-ratios wider than 3:1 are sometimes required in order to meet guideline thresholds for post-disposal electrical conductivity (EC) and sodium adsorption ratio (SAR) of the treated soil.

Drilling waste disposal contractors often ask testing laboratories to prepare and analyze waste:soil mixtures at a series of volume ratios, thus introducing the problem of replicating field practices on a bench scale. At the request of an AEUB sub-committee charged with updating the salinity portion of G-50, we undertook to devise a rugged lab mixing protocol.

Under certain waste disposal conditions, drilling fluids or waste extracts must pass the Microtox bioassay. The dark brown waste liquids which are often involved present a challenge for colour-correction methods used to estimate the contribution of colour absorbance, as distinct from toxicity, to loss of light output from the test organism. Again in the interests of improving inter-lab data agreement, we investigated ways to circumvent this difficulty.

### **MATERIALS AND METHODS**

Various drilling wastes and receiving soils were submitted to ALS Environmental from well sites in the vicinity of Fort St. John, BC and Grande Prairie AB by drilling waste disposal contractors. The range of materials was typical of samples generated using current field practices. The EC and SAR of individual soils and soil-waste mixtures were measured by analyzing the filtrate from a saturated paste (USDA 1954; Carter 1993).

Sump liquids or 1:1 extracts of sump solids were subjected to the Microtox bioassay after high speed centrifugation and pH adjustment, as prescribed in G-50. One of us (H.D.) investigated the effect of the order of doing these procedures in preparing dark brown fluids for the Microtox bioassay.

<sup>1</sup> Senior Soil Scientist, ALS Environmental, 9936-67 Ave., Edmonton AB, T6E 0P5  
john.ashworth@alsenviro.com

<sup>2</sup> Inorganic Section Leader, ALS Environmental, 9505-111 St. Grande Prairie AB, T8V 5W1

### **Soil-waste mixing protocol**

Soil samples delivered to a lab are almost always disturbed material. In any case, if waste:soil mixing is required, for quality control purposes the soil sample must be homogenized. Moist-sieving through 5 mm, followed by hand mixing, yields a substrate that is suitable for preparing the mixture, but the bulk density of the homogenized soil is then significantly less than on-site. Rather than attempt to mix soil and waste on a volume basis, therefore, enough homogenized moist soil for a saturated paste can be weighed into a suitable container, then the appropriate volume of liquid waste added.

The volume of waste to use is given by:

$$\text{Volume (mL)} = \text{soil weight (g)} \times (100 - \% \text{ moi.}) / \text{Factor} \quad \dots (1)$$

in which % moi. (obtained by drying a separate sub-sample at 105 C) =  $100 \times (\text{loss on drying}) / (\text{moist weight})$ .

The Factor in Eqn. (1) =  $100 \times R \times D$  where R is the mix ratio and D is the field soil's undisturbed dry bulk density. It is possible to select a value of D based on texture, but choosing the correct texture could be another source of inter-lab disagreement. Using a default bulk density of 1.54 kg/L values of the Factor are 460, 770 and 1,080 for a 3:1, 5:1 and 7:1 volume mix ratio, respectively. The default bulk density was the mean value obtained for 14 subsoils, from drilling leases in the region, whose density ranged from 1.33 to 1.79 kg/L (J.A., unpublished, 2004). It is close to the mean value obtained in another recent study of Alberta soils (D. Keyes, private communication).

Rather than use a measuring cylinder for the waste volume, a useful procedure is to weigh the required volume of deionized water into a disposable clear plastic cup, mark the level, discard the water and fill to the mark with the waste, which is then transferred and rinsed into the container holding the soil sample.

In cases where the drilling waste supplied is a sump solids sample which does not pour easily, a weight of homogenized waste can be used; the weight (g) of waste required = (above calculated Volume)  $\times$  (waste SG), where SG is the specific gravity of the waste, obtained for example with a mud balance.

In all cases, having added the required quantity of waste to the soil, deionized water is then blended in and a saturated paste made, equilibrated and filtered, as usual.

### **Microtox bioassay of dark brown liquids**

The procedure for the Microtox bioassay described in G-50 is essentially the 82 % ISA procedure described by the Western Canadian Microtox Users Committee (WCMUC 1994). Sample preparation usually involves centrifugation and pH adjustment (range 6.0 - 8.8). In this bioassay, light emitted by aliquots of the luminescent test bacterium, treated with four dilutions of the test liquid, is measured and plotted against liquid concentration on a log-log plot. An EC50(15) value is reported, which is the value of the intercept of a least squares fit to the data, on the axis where light output has fallen by 50 % after 15 minutes (log "Gamma" = 0).

Colour correction of the EC50(15) value is required for reddish-brown samples because they absorb light of the wavelength emitted by the bacteria. If light absorbance by the test liquid is intense, the mathematics of the colour correction breaks down, and corrected light readings fall erratically on the log-log plot. This failure can occur whether

colour correction is made by the double cuvette method (still used in on-site measurements) or by the spectrometer method now used in most testing laboratories.

## RESULTS AND DISCUSSION

### Soil-waste mixing protocol

If the EC or SAR of the 3:1 mixture exceeds post-disposal criteria, the wider mix ratio  $R_w$  needed to meet criteria is given by:

$$R_w = \{ 4 \times (p_{3:1} - p_{soil}) / (p_{crit} - p_{soil}) \} - 1 \quad \dots (2)$$

in which  $p$  is the value of the EC or SAR parameter either in the 3:1 mix, the soil, or in the AEUB guideline ( $p_{crit}$ ). Equation (2) is based on the observation that plots of mix EC or SAR against the % of waste in the mix are in effect linear when the waste percentage is 25 % (3:1 ratio) or less (wider ratios). Predicting a required wider mix ratio in this way may be preferable to actually analyzing soil:waste mixtures made at multiple mix ratios, from the points of view of both analytical consistency and expense. Previous work (Ashworth & Webster 2004) showed good agreement between predicted and actual EC and SAR data at mix ratios other than 3:1. Subsequent testing of a pilot scale mix made on-site can be done to confirm that required post-disposal criteria are being met at that ratio.

### Microtox bioassay of dark brown liquids

In many cases, dark brown drilling waste liquids also have high pH. We find that, if such samples are pH-adjusted and then centrifuged, the resulting supernatant is often clear and far less coloured. Whereas if an identical sample is instead first centrifuged and then pH-adjusted, it tends to remain strongly coloured and thus likely to cause the math of the colour correction procedure to break down, as described above. Microtox testing labs are, therefore, strongly recommended to do pH adjustment and colour correction, in that order. No set order for these steps is prescribed in the Microtox literature.

If sufficient colour removal cannot be achieved, a possible procedure to follow is to dilute enough of the colour out, so as to obtain a plot in which the 4 colour-corrected points fall reasonably close ( $r^2 > 0.9$ ) to a straight line, or to a gentle curve that can be fitted by a quadratic. If the toxicity is also diluted out, an extrapolated EC50(15) value can be obtained; we recommend that the length of the extrapolated portion of the log-log plot should be less than the distance covered by the 4 experimental points.

The extrapolated EC50(15) value for the diluted, colour-corrected liquid (which may be as high as several hundred percent) must of course be divided by the dilution factor used which, if there is actual toxicity present in addition to the interfering colour, will then result in a reportable EC50(15) value below the accepted 75 % pass threshold (i.e. a Fail).

## CONCLUSIONS

In order to meet post-disposal EC and SAR criteria in the revised D-50, drilling waste contractors may have to rely on achieving a suitably wide mix-ratio. Accurate prediction of the required mix-ratio will depend on submitting good samples, and careful mixing in the laboratory, preferably by following the procedure described above. Rather than try several mix-ratios, predicting the required ratio from data obtained at one default ratio (e.g. 3:1), using Eqn. 2 above, may also help avoid inter-lab discrepancies.

Procedures have also been suggested for improving interlab agreement on results of Microtox bioassays for dark brown liquids.

### **ACKNOWLEDGEMENTS**

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### **LITERATURE REFERENCES**

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