Technical Memorandum F

To: Nunavut Water Board
From: Shear Diamonds Ltd.
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Onsite Nitrate and TSS Analysis Methods

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1.0 INTRODUCTION

The onsite analysis methods (OAM) for nitrate and total suspended solids (TSS), described herein, have been developed to provide procedures for conducting these tests at the Jericho Diamond Mine, NU. These test results will be used to augment the offsite laboratory test work and will provide a means of onsite quality control of water derived from the open pit and the Processed Kimberlite Containment Area (PKCA). This technical memorandum is a supplementary document in support of the 2011 Pit Dewatering Addendum to Processed Kimberlite Management Plan (EBA, 2011a).

As described in the Pit Dewatering Addendum, water impounded in the open pit will be pumped into the PKCA.The pit water will mix with the PKCA impounded water, and the PKCA water will be discharged into the receiving environment (Stream C3). Nitrates and TSS were identified as the constituents that had the potential to cause an excedance of the water licence discharge criteria. Daily field parameter measurements and onsite nitrate and TSS analysis from the pit water discharge area and the PKCA will be performed during the pit dewatering period. Weekly offsite laboratory analysis will also be conducted as specified in the Pit Dewatering Addendum and as required by Part G. 6 of the water license.

The daily onsite results will be analyzed to confirm that the water discharged from the PKCA does not exceed the water licence discharge criteria. If exceedances are indicated adaptive management measures for the pit dewatering activities, as discussed in the Pit Dewatering Addendum, will be implemented.

2.0 ONSITE ANALYSIS PROCEDURES

2.1 Nitrate Analysis

The onsite nitrate analysis will be performed using a Hach DR2700 Portable Spectrophotometer (Hach, 2008b) with Chromotropic Acid reagents. When nitrate in the water sample react with chromotropic acid under strongly acidic conditions, it will yield a yellow colour with a maximum absorbance at 410 nm (Hach, 2008a). The Spectrophotometer will analyze the change in colour and convert the signal into a nitrate concentration. A standard operation procedure (SOP) for the onsite nitrate analysis has been developed based on the manufacturer instruction, and included in Appendix A.

2.2 TSS Analysis

The testing procedure and apparatus for the onsite TSS analysis are both in accordance with the *Standard Methods for Standard Methods for the Examination of Water and Wastewater* (Standard Method No: 2540 D). This is consistent with the testing method in the offsite laboratory. The testing involves passing the water sample through a pre-weighed filter, and then drying and weighing the filter retaining suspended solids in the water sample. The TSS concentration will be calculated based on the weight of retained solids and the volume of water passing through the filter. A standard operation procedure (SOP) for the onsite TSS analysis has been developed, and included in Appendix B.

3.0 QUALITY ASSURANCE AND QUALITY CONTROL

3.1 Quality Assurance

Quality Assurance (QA) refers to a set of coordinated actions such as plans, specifications, and policies that assure that a measurement program can be quantifiable and produce data of known quality.

3.1.1 Field Staff Qualification

Staff involved in the water sampling and onsite analysis must be trained, such that they are competent in collecting and handling water samples, and in operating and managing the required onsite laboratory equipment.

3.1.2 Laboratory Equipment Installation and Maintenance

All water quality testing instruments will be located in a separate environment laboratory within the camp complex. The environment laboratory will be maintained at a steady room temperature .The testing instruments will be installed by an experienced environmental technician, and the procedure will be based on the manufacturer instructions or applicable standard methods. The maintenance, servicing, and operation of the equipment will be according to the manufacturer instructions.

3.1.3 Water Sample Storage and Handling

The water samples are collected in the morning by environmental personnel onsite. Collected water samples are stored in a dedicated refrigerator maintaining a temperature between 2°C and 4°C. The samples are analyzed within 12 hours following the sample collection. Prior to the analysis, the samples are rested to allow equilibrium to room temperature.

3.1.4 Records Keeping

The onsite analysis results along with the quality control (QC) records will be recorded in the paper copy and later entered into electronic format for storage on the site server. These records will be stored within the onsite environment laboratory and will be interpreted for pit dewatering management. These records and data interpretation will be available for review by authorized outside parties upon request.

3.1.5 Offsite Laboratory Qualification

The Canadian Association for Laboratory Accreditation (CALA) provides accreditation programs to review laboratories' procedures, methods, and quality controls. Samples that are submitted to the offsite laboratories will be submitted to a CALA accredited laboratory.

3.2 Quality Control

Quality Control (QC) is a system of maintaining standards for measurement through field and laboratory testing when following a defined or industry standard. Measures of QC success include precision, reproducibility of test results, and accuracy.

3.2.1 Precision Check and Reproducibility

To check the analysis instruments and testing procedures' precision, split samples will be prepared at the onsite laboratory. A split sample is a discrete water sample separated into two identical tests and used to determine the reproducibility of the analysis. In theory, the individual test results from the split sample should be identical when analyzed with the same instrument. One split will be processed for every ten (10) samples for both nitrate and TSS analysis. It should be noted the onsite laboratory split samples will not replace the required blank, duplicate, and split samples for the weekly offsite laboratory analysis. Detailed descriptions of the QC sampling for offsite laboratory analysis are included in the General Monitoring Plan (EBA, 2011b).

If the relative percent difference (RPD)[1] of the split samples is larger than 5% for nitrate analysis or 10% for TSS analysis the testing procedures will be reviewed and, if applicable the manufacture will be contacted.

3.2.2 Accuracy Check and Equipment Calibration

For the nitrate analysis, the accuracy of the DR 2700 Spectrophotometer will be checked weekly using the nitrate-N standard solutions. If the RPD is larger than 5%, additional calibrations will be performed following the manufacturer instructions.

In addition, onsite analysis will be performed from the split samples collected for the weekly offsite laboratory analysis. If the RPD of the analysis is larger than 5% for nitrate analysis or 10% for TSS analysis additional calibrations will be performed.
For the TSS analysis, the apparatus setup and the testing procedures follow the same standard method as the offsite laboratory, and the analysis. The only component of the apparatus that requires an accuracy check and calibration is the electric weighing scale. The accuracy and calibration of the electric weighing scale is conducted weekly using calibration weights. If the RPD is larger than 5%, additional calibrations will be performed using the calibration weights. In addition, onsite analysis will be performed on the same water samples that were submitted for the weekly offsite laboratory analysis. If the RPD of the analysis is larger than 10%, additional calibrations or adjustments to the apparatus setup will be performed following the offsite laboratory's instructions.

[1] Relative Percent Difference between values a and b:

shear diamonds		**APPENDIX A** **Onsite Nitrate Analysis Using Chromotropic Acid Method**	
Owner: Environment Laboratory	**Approver:** Environment Department	**Issue:** 1.0	**Creation Date:** June 17, 2011 **Revision Date:** N/A

1.0 OBJECTIVE

The objective of the onsite nitrate analysis is to obtain daily nitrate concentrations of PKCA and pit water. These results will be reviewed to ensure that PKCA discharge water does not exceed the water licence discharge criteria for nitrate, and to trigger potential adaptive management measures for pit dewatering.

2.0 SCOPE OF APPLICATION

The onsite nitrate analysis will be conducted daily during pit dewatering, in compliance with the Pit Dewatering Addendum. Samples to be analyzed include samples from monitoring stations JER-SWQ-02, JER-SWQ-04, and JER-SWQ-04A, laboratory split samples, and standard solution checks.

The reported detection limit for the onsite nitrate testing unit is 0.2 mg/L as indicated in the manufacture instruction.

3.0 INTERFERENCES

The following table listed the constituents that may interfere with the nitrate analysis:

Interfering substance	Interference level	Interference Probability [(1)]
Barium	A negative interference at concentrations greater than 1 mg/L.	Not Likely
Chloride	Interfere above 1000 mg/L.	Not Likely
Nitrite	A positive interference at concentrations greater than 12 mg/L.	Not Likely
Copper	Positive interference at all levels	Low

Note:

1. The Interference Probability is determined by comparing the interference level and pit water quality results in February 2011. When the pit water is pumped and mixed with PKCA water, the potential interference is expected to be lower.

4.0 EQUIPMENT

Required Reagents:

- Test 'N Tube™ NitraVer® X Reagent Set
- Required Apparatus (powder pillows): TenSette® Pipet, 0.01 to 1.0 ml,;
- Pipet tips, for TenSette Pipet;
- Test Tube Rack (1-3); and
- DR 2700 Portable Spectrophotometer.
- timer

Calibration Standards:

- Deionized Water;
- Nitrate-N Standard Solution, 10 mg/L NO_3-N; and
- Nitrate-N Standard Solution, Voluette® Ampule, 500 mg/L NO_3-N.

5.0 SAMPLE HANDLING AND STORAGE

The sample should be stored at between 2°C and 4°C, and needs to be analyzed within 24 hours after sampling.

6.0 PROCEDURE

6.1 Analysis Preparation

- Install the light shield in Cell Compartment #2 before performing the test;
- For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust;
- Allow the sample to equilibrate to room temperature; and
- Filter each water sample through the filter provided by the offsite laboratory.

6.2 Sample Analysis

- Turn on DR2700 Spectrophotometer, select "**344 N, Nitrate HR, TNT**" from Stored Programs, and press Start;
- Insert an adapter if required (refer to user manual for orientation);
- Remove the cap from a NitraVer X Reagent A Test 'N Tube vial and add **1.00 ml** of sample;

- Cap the tube and invert **ten (10)**[1] times to mix;
- Wipe the vial and insert into the 16-mm cell holder;
- **ZERO** the instrument (display will show: 0.0 mg/L NO^3-N);
- Remove the vial from the instrument. Using a funnel, add the contents of **one** NitraVer X Reagent B Powder Pillow to the vial;
- Cap and invert **ten (10)** times to mix; Note: some solid matter will not dissolve.
- Start the instrument timer. A **five-minute** reaction time will begin. Do not invert the vial again. A yellow color will develop if nitrate is present; and
- Within five minutes after the timer expires, wipe the prepared sample and insert it into the cell holder.

6.3 Calculation and Recording

- Read the results in mg/L NO_3-N; and
- Record the results in the paper copy of the analysis record form, and enter the results in the electronic record form.

7.0 PRECISION AND ACCURACY CHECKS

7.1 Precision Check

- For **every ten (10) samples**, prepare and analyze a split sample;
- Calculate the Relative Percent Difference (RPD)[2] of the split samples; and
- Record the split sample result in the analysis record form:
  - If the RPD is less than 5%, the instrument precision is acceptable; and
  - If the RPD is larger than 5%, review the instrument user manual and/or consult with the manufacture for further instructions.

7.2 Standard Accuracy Check and Calibrations

There are two standard accuracy check and calibration methods as recommended by the manufacturer: Standard Solution Method (Section 7.2.1.1) and Standard Addition Method (Sample Spark) (Section 7.2.1.2).

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1 One inversion includes: (1) Holding the vial in a vertical position with the cap pointing up; (2) Turning the vial upside-down, (3) Wait for all of the solution to flow down to the cap; (4) Pause; (5) Return the vial to an upright position; and (5) Wait for all the solution to flow to the bottom of the vial.

2 Relative Percent Difference between values a and b:

- Perform **weekly** Standard Solution Method (Section 7.2.1.1);
- Calculate the RPD of between the analyzed standard solutions and the actual solution concentration;
- Record the Standard Solution Check result in the analysis form:
  - If the RPD is less than 5%, the instrument accuracy is acceptable; and
  - If the RPD is larger than 5%, perform Standard Addition Method (Section 7.2.1.2) and repeat Standard Solution Method (Section 7.2.1.1.) to recheck accuracy.

7.2.1 Standard Solution Method

The required material includes 10.0 mg/L Nitrate-N standard solution.

The operation process includes:

- Use the 10.0-mg/L nitrate-N standard solution in place of the sample;
- Perform regular concentration measurement as described in Section 6.0,
- To adjust the calibration curve using the reading obtained with the standard solution, navigate to Standard Adjust (OPTIONS>MORE>STANDARD ADDITIONS); and
- Turn on the Standard Adjust feature, and accept the displayed concentration.

7.2.2 Standard Additions Method (Sample Spike)

The required materials include:

- High range Nitrate Nitrogen Voluette® Ampule Standard, 500 mg/L NO_3-N;
- Ampule breaker;
- TenSette Pipet and Pipet Tips; and
- Mixing cylinders (3).

The operation process includes:

- After reading test results, leave the sample cell (Unspiked Sample) in the instrument;
- Select standard additions from the instrument menu (OPTIONS>MORE>STANDARD ADDITIONS);
- Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the Unspiked Sample reading will appear in the top row;
- Open the standard solution ampule;
- Use the TenSette Pipet to prepare spiked samples: add 0.1 mL, 0.2 mL and 0.3 mL of standard to three mixing cylinders with 25-mL portions of fresh sample;

- Add 1 mL of the 0.1 mL spiked sample to a TNT vial and follow the *Chromotropic Acid method for TNT* test procedure. Repeat for each spiked sample. Measure each of the spiked samples in the instrument; and
- Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

7.3 Laboratory Accuracy Check

- Calculate the RPD of between the offsite laboratory analysis and onsite analysis results;
- Record the calculation results in the analysis form:
  - If the RPD is less than 5%, the instrument accuracy is acceptable;
  - If the RPD is larger than 5%, perform Standard Solution Method (Section 7.2.1.1.) or Standard Addition Method (Section 7.2.1.2) to calibrate the instrument.

8.0 REFERENCES

Hach Company. (2008a), Chromotropic Acid Method for Testing Nitrate, Method No.: 10020, File No.: DOC316.53.01068, 5th Edition, Updated in February 2008. [Online] *www.hach.com/fmmimghach? /CODE%3ADOC316.53.0106815609%7C1*

Hach Company. (2008b), DR2700 Portable Spectrophotometer User Manual, File No.: DOC026.53.00809, 3th Edition, Updated in September 2008. [Online] http://www.hach.com/fmmimghach? /CODE%3ADOC026.53.00809-200816935%7C1

shear diamonds		Onsite Total Suspended Solids (TSS) Analysis Using Vacuum Filtration and Oven Drying	
Owner: Environment Laboratory	**Approver:** Environment Department	**Issue:** 1.0	**Creation Date:** June 17, 2011 **Revision Date:** N/A

1.0 OBJECTIVE

The objective of the onsite total suspended solids (TSS) analysis is to obtain daily TSS concentrations of PKCA and pit water samples. The analysis result will be reviewed to ensure that the PKCA discharge water does not exceed the water licence discharge criteria for TSS, and to trigger potential adaptive management measures for pit dewatering.

2.0 SCOPE OF APPLICATION

The onsite TSS analysis will be conducted daily during pit dewatering in compliance with the Pit Dewatering Addendum. Samples to be analyzed include samples from monitoring stations JER-SWQ-02, JER-SWQ-04, and JER-SWQ-04A, laboratory split samples, and standard solution checks.

The testing procedure and apparatus for the onsite TSS analysis are in accordance with the *Standard Methods for Standard Methods for the Examination of Water and Wastewater* (Standard Method No: 2540 D). The offsite laboratory detection limit for TSS analysis is 3 mg/L, and is adapted in the onsite laboratory.

3.0 INTERFERENCES

- Water containing a high total dissolved solids (TDS), such as calcium, magnesium, chloride and/or sulphate requires longer drying period, proper desiccation and rapid weighting;
- Large particles and agglomerates need to be excluded prior to analysis; and
- Disperse visible oil and grease prior to analysis.

4.0 EQUIPMENT

- Laboratory Oilless Diaphragm Vacuum Pump/Compressor;
- Tubing (D.I ¼'');
- Vacuum Volumetric Flask;
- PVC Vacuum Manifold, 3 places;
- ProWeight® glass fibre filters for TSS (on aluminum plates) and clamps;
- Filter Forceps, stainless;

- Deionized water;
- Graduated cylinders (50mL, 100ml, 250ml, 500ml);
- Drying oven (103-105°C) and Thermometer;
- Secador™ 4.0 Desiccator Cabinet
- Silica Gel Desiccant (grade 48); and
- Sautorious ME Series Electronic Analytical Balance (to .0001 g) .

5.0 SAMPLE STORAGE

The sample should be stored at 4 °C, not be allowed to freeze, and needs to be analyzed within 24 hours after sampling.

6.0 PROCEDURE

6.1 Analysis Preparation

- Allow the sample to equilibrate to room temperature.

6.2 Sample Analysis

- Dry the filters for a minimum of 1 at 100°C +/- 5°C
- Let the filters cool 15min into the desiccator
- Weight each filter, and record the weight in mg as **Initial Weight (IW), place in labelled** aluminum weighing dish**;**
- Using forceps, install filter (hatched side down) on filter apparatus and wet with DI water.
- Assemble filtering apparatus and start suction;
- Shake the sample bottle to obtain homogeneous particle size;
- Choose a sample volume that will yield a residue between 2.5 and 200mg;
  - Typically 250 mL water for mild turbidity sample and 50-100 mL for highly turbid sample;
  - If volume filtered fails to meet minimum yield, increase sample volume up to 1 L; and
  - If filtration is more than 10 min, decrease sample volume.
- Pour the sample of determined volume into a graduated cylinder. Record the sample volume in mL as **Volume Filtered**;
- Pour the sample into the filter holder and open the manifold valve. Rinse the cylinder with deionized water into the filter holder at least five (5) times;
- Rinse the filter with three (3) successive 10 mL volumes of deionized water, and allow complete drainage between washings;

- Continue to vacuum for additional three (3) min after filtration is complete;
- Carefully remove filter from filtration apparatus, fold it in two, and transfer to the same labelled aluminum weighing dish as support;
- Dry the filters in the oven for a minimum of one (1) hour at 100°C +/- 5°C , and cool the filter in the desiccator for 15 min;
- Weigh the sample
- Repeat the drying, cooling, desiccating and weighing until the weight change is less than 0.5 mg; and
- Record the weight in mg as **Final Weight (FW).**

6.3 Calculation and Recording

- The TSS is then calculated as below:

$$\text{TSS}\left(\frac{\text{mg}}{\text{L}}\right) = \frac{\text{Final Weight (mg)} - \text{Initial Weight(mg)}}{\text{Volume Filtered(ml)}} \times 1000$$

- Record the results in the paper copy of the analysis record form, and enter the results in the electronic record form.

7.0 PRECISION AND ACCURACY CHECKS

7.1 Precision Check

- For **every ten (10) samples**, prepare and analyze a split sample;
- Calculate the Relative Percent Difference (RPD)[1] of the split samples; and
- Record the split sample result in the analysis record form:
  - If the RPD is less than 10%, the instrument precision is acceptable; or
  - If the RPD is larger than 10%, review the applicable standard methods and/or consult with the offsite laboratory for further instructions.

7.2 Onsite Accuracy Check and Calibration

7.2.1 Standard Blank Check

- Perform weekly Standard Blank Check using prepared standard of 50mg/L Infusorial Earth

Dilute standard to 25 mg/L in de-ionized water

If the measure TSS RPD < 10% the result is acceptable the measured TSS RPD >10% the result is un-acceptable and procedures and equipment must be evaluated to find the source of inaccuracy.

1 Relative Percent Difference between values a and b:

7.2.2 Method Blank Check

- Perform **weekly** Method Blank Check for the apparatus using 1000 L deionized water.
  - If the measured TSS is <3 mg/L (detection limit of the offsite laboratory), the method blank check is passed; and
  - If the measured TSS is >3 mg/L, review the applicable standard methods and/or consult with the offsite laboratory for further instructions.

7.2.3 Check on Weighing Scale

- Perform **weekly** accuracy check of the weighing scale using calibration weights;
- Calculate the RPD of between the measured weights and the actual weights; and
- Record the Standard Solution Check result in the analysis form:
  - If the RPD is less than 5%, the instrument accuracy is acceptable; or
  - If the RPD is larger than 5%, review the scale user manual and/or contact the scale manufacturer for further instructions.

7.3 Laboratory Accuracy Check

- Calculate the RPD of between the offsite laboratory analysis and onsite analysis results;
- Record the Standard Solution Check result in the analysis form:
  - If the RPD is less than 10%, the instrument accuracy is acceptable; or
  - If the RPD is larger than 10%, review the applicable standard methods and/or consult with the offsite laboratory for further instructions.

8.0 REFERENCES

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