## **Technical Report**

| Title:       | XRD Mineralogical Analysis of DGR-1 and DGR-2 Core                                   |
|--------------|--|
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DGR Site Characterization Document Intera Engineering Project 06-219



| Intera Engineering DGR Site Characterization Document |  |                      |  |
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## 1 Introduction

Intera Engineering Ltd. has been contracted by Ontario Power Generation (OPG) to implement the Geoscientific Site Characterization Plan (GSCP) for the Bruce site located on Lake Huron, Ontario. The purpose of this site characterization work is to assess the suitability of the Bruce site to construct a Deep Geologic Repository (DGR) to store low-level and intermediate-level radioactive waste. The GSCP is described by Intera Engineering Ltd. (2006).

As part of the GSCP, Intera Engineering Ltd. retained Activation Laboratories ("ActLabs") of Ancaster, Ontario to undertake mineralogical testing of cores collected from boreholes DGR-1 and DGR-2. This Technical Report summarizes the results of the x-ray diffraction (XRD) analysis of DGR-1 and DGR-2 core samples to quantify the major minerals present in the cores and to identify the clay minerals present in the clay-sized fraction.

Work described in this Technical Report was undertaken in accordance with Test Plan TP-07-01 – Laboratory Testing of DGR-1 and DGR-2 Solid Core for Geochemistry & Mineralogy (Intera Engineering Ltd., 2007a), which was prepared following the general requirements of the DGR Project Quality Plan (Intera Engineering Ltd., 2007b).

#### 2 Background

Core samples of 76 mm diameter were collected during diamond coring of boreholes DGR-1 and DGR-2 at the Bruce site from January until August, 2007. All core samples were vacuum sealed within nitrogen flushed polyethylene and aluminium foil/polyethylene bags following core retrieval and the general preservation and handling requirements of TP-06-10 (Intera Engineering Ltd., 2007c). Thirty-four preserved core samples from boreholes DGR-1 and DGR-2 were shipped to ActLabs under chain of custody procedures (following procedure DGR P4). Table 1 lists the Intera sample identifier based on depth in metres below ground surface (mBGS), geologic formation and description of the 14 DGR-1 samples selected for mineralogical testing.

| Intera Sample ID | Formation      | Sample Description                            |  |
|------------------|----------------|---|--|
| DGR1-049.16      | Amherstburg    | Light brown-grey, fine grained dolostone      |  |
| DGR1-097.08      | Bois Blanc     | Grey-brown cherty dolostone                   |  |
| DGR1-130.03      | Bass Islands   | Dark grey, very fine grained dolostone        |  |
| DGR1-156.63      | Bass Islands   | Grey-brown fine grained dolostone             |  |
| DGR1-231.49      | Salina E Unit  | Grey-tan brecciated dolostone                 |  |
| DGR1-267.78      | Salina B Unit  | Tan argillaceous dolostone                    |  |
| DGR1-322.19      | Salina A2 Unit | Massive light grey-blue anhydritic dolostone  |  |
| DGR1-361.76      | Salina A1 Unit | Grey argillaceous dolostone                   |  |
| DGR1-399.85      | Gasport        | Grey dolomitic limestone                      |  |
| DGR1-419.99      | Cabot Head     | Red-maroon massive shale                      |  |
| DGR1-446.25      | Manitoulin     | Tan-grey, fine-grained, crystalline dolostone |  |
| DGR1-455.45      | Queenston      | Red-maroon massive shale                      |  |
| DGR1-456.01      | Queenston      | Red-maroon massive shale                      |  |
| DGR1-460.77      | Queenston      | Grey-green and red-maroon shale               |  |

 Table 1
 Formations, Description & Depths of DGR-1 Core Samples

Table 2 lists similar information for the 19 DGR-2 samples. One sample – DGR2-687.42 – was sent for petrographic analysis but was not analysed by XRD or lithogeochemistry and SEM/EDS.

| Intera Sample ID | Formation                   | Sample Description                                   |  |
|------------------|-----------------------------|--|--|
| DGR2-451.33      | Queenston                   | Red-maroon massive shale                             |  |
| DGR2-482.45      | Queenston                   | Interbedded green shale and grey limestone           |  |
| DGR2-508.93      | Queenston                   | Red-maroon shale                                     |  |
| DGR2-535.56      | Georgian Bay                | Interbedded grey-green shale and limestone           |  |
| DGR2-550.28      | Georgian Bay                | Interbedded dark grey-green shale and limestone      |  |
| DGR2-570.73      | Georgian Bay                | Interbedded dark grey-green shale and limestone      |  |
| DGR2-590.10      | Georgian Bay                | Dark grey-green shale                                |  |
| DGR2-606.62      | Georgian Bay                | Dark grey-green shale                                |  |
| DGR2-606.96      | Georgian Bay                | Dark grey-green shale                                |  |
| DGR2-626.29      | Blue Mountain               | Dark grey-green massive shale                        |  |
| DGR2-644.49      | Blue Mountain               | Dark grey-black massive shale                        |  |
| DGR2-659.31      | Collingwood Member, Cobourg | Interbedded grey shale and argillaceous limestone    |  |
| DGR2-669.27      | Cobourg                     | Grey argillaceous limestone                          |  |
| DGR2-677.93      | Cobourg                     | Mottled grey argillaceous limestone                  |  |
| DGR2-695.51      | Sherman Fall                | Grey argillaceous limestone                          |  |
| DGR2-704.87      | Sherman Fall                | Grey fossiliferous argillaceous limestone            |  |
| DGR2-745.97      | Kirkfield                   | Interbedded grey shale and argillaceous limestone    |  |
| DGR2-816.85      | Gull River                  | Grey argillaceous limestone/mudstone                 |  |
| DGR2-844.95      | Cambrian                    | Tan-grey argillaceous dolostone with minor sandstone |  |

| Table 2 | Formations, Descriptions & Depths of DGR-2 Core Samples |
|---------|---|
|---------|---|

The core samples were inspected by Actlabs and Dr. Eva Schandl on receipt at Actlabs. If the cores were laminated with shale intervals in the case of carbonate rocks or were principally shale, then the shale was retained by Actlabs and analysed for clay-mineral identification of oriented (basal-plane) samples based upon standard ethylene glycol treatment and sample heating to 400 °C and 550 °C. In this case, the thin sections were prepared from the bulk rock and results recorded by photomicrographs (i.e., TR-07-12, Intera Engineering Ltd., 2009a). That results in Dr. Schandl's report being intentionally biased towards the carbonate rocks rather than the shales. In addition, Actlabs analyzed the whole rock by XRD to determine mineral concentrations.

ActLabs determined the mineralogy of both the DGR-1 and DGR-2 cores by x-ray diffraction – this report – and by petrography through Dr. Schandl, whose results are recorded in TR-07-12 (Intera Engineering Ltd., 2009a). Additionally, DGR-1 and DGR-2 cores underwent a complete 'lithogeochemical' analysis that included measurement of 10 oxides and approximately 50 trace elements. These data and the results of analysing the Ordovician shales and argillaceous limestones by scanning electron microscopy (SEM) coupled with an energy dispersive spectrometer (EDS) are presented in TR-08-02 (Intera Engineering Ltd, 2009b).

Sample descriptions provided in Tables 1 and 2, are based on the general core log descriptions of the individual core runs that provided the cores selected for laboratory testing. For this reason and because the observed variability of lithology within individual core samples, the descriptions provided in Tables 1 and 2 may show slight variations from those that may be determined from mineralogical results given in Section 3.

#### 3 Methods

#### 3.1 Whole Rock

The samples were pulverized, ground in a ceramic mortar and back-loaded in an aluminum sample holder to minimize preferred orientation of the crystallites. Powder XRD was used to identify and quantify minerals present in the samples. The measurements were performed using a Panalytical X'Pert Pro PW3040 Multi Purpose Diffraction system controlled and operated by Panalytical B.V. X'Pert Data Collector software, Version 2.2a. Experimental data were processed using Panalytical B.V. X'Pert HighScore software, Version 2.2.1. The measurement conditions were:

| Start Position [°2Theta]     | 3.0100       |
|------------------------------|--------------|
| End Position [°2Theta]       | 89.9900      |
| Step Size [°2Theta]          | 0.0200       |
| Scan Step Time [s]           | 1.0000       |
| Scan Type                    | Continuous   |
| Divergence Slit Type         | Fixed        |
| Divergence Slit Size [°]     | 1.0000       |
| Receiving Slit Size [mm]     | 0.4000       |
| Measurement Temperature [°C] | 25.00        |
| Anode Material               | Cu           |
| K-Alpha1 [Å]                 | 1.54060      |
| K-Alpha2 [Å]                 | 1.54443      |
| Generator Settings           | 50 mA, 40 kV |
| Spinning                     | 2rps         |

#### 3.2 Clay Fraction

A four-step process was followed before X-ray diffraction was performed using Phillips PW 1050 diffractometer equipped with Cu X-ray source and operated at 40 kV and 30 mA.

Firstly, the clay fractions were separated from the samples by milling the cores and soaking the resulting powders in purified water for several hours, sonicating and decanting the clay fraction after timed sedimentation. In order to isolate more clay from some DGR-2 carbonate rock samples with low clay content (i.e., DGR2-677.93, DGR2-695.51, DGR2-816.85, & DGR2-844.95), these samples were repeatedly soaked in purified water, sonicated and centrifuged ten times.

Secondly, the decanted suspensions containing the clay fraction were filtered through a 0.4  $\mu$ m filter. In most cases only a small amount of fine fraction was obtained. For such cases the material collected on the filter was re-dispersed in a very small amount of purified water and the concentrated suspension was placed on a glass slide and allowed to dry at room temperature. If a substantial amount of clay fraction material was obtained,

several drops of the suspension were dried on the glass slide to obtain the oriented film. The film was dried and used for X-ray diffraction.

Subsequently, the samples were placed in a desiccator with ethylene glycol, evacuated for 15 minutes and left to saturate overnight. The samples were examined by X-ray diffraction immediately after taking them out of the desiccator. Finally, the glycolated samples were heated at 400 °C and at 550 °C for 1½ hours and examined by X-ray diffraction after each treatment.

## 4 Results

## 4.1 Whole Rock

Mineral identification was performed using search and match feature of X'Pert HighScore software. Appendix A shows the results of search and match analysis for selected samples (DGR1-322.19, DGR1-399.85, DGR2-606.62, DGR2-816.85 and DGR2-844.95). The samples were chosen such that every mineral identified in the submitted samples is represented and its characteristic diffraction lines are shown for reference. Accordingly, the appended diffractograms show the lines from all identified minerals first and additionally, the diffraction lines from every mineral encountered in selected samples is shown separately for reference.

Once the minerals were identified, their structures, as referenced in structural crystallographic databases, were used for full profile Rietveld refinement<sup>1</sup> to obtain the relative concentrations of the minerals. The calculated profiles were fitted to the experimental profiles by the full profile Rietveld refinement with the following structural parameters allowed to vary: scale factor, unit cell parameters (only for the initial steps), preferred orientation, zero shift and the peak shape. Appendix B presents results of the Rietveld analysis for all DGR1 and DGR2 core samples. The red curve shows the measured diffraction pattern, and the blue curve shows the calculated pattern. The lower graph shows the difference between the measured and the calculated patterns. Tables 3 and 4 present the results of the quantitation by the Rietveld approach for DGR-1 and DGR-2, respectively.

Calcite, dolomite and quartz are common minerals throughout the DGR-1 cores, i.e., throughout the Devonian and Silurian sedimentary sequence of rocks. With depth, the clay minerals illite and chlorite [clinochlore] become more common as shale units are encountered. Soluble minerals such as gypsum, anhydrite and halite become more common beneath the Salina F Unit, with halite present in the Cabot Head and Manitoulin Formations.

The Upper Ordovician shales tend to be dominated by illite and quartz, while the middle Ordovician carbonates are principally calcite with minor dolomite and illite. However, the deepest samples – from the Gull River (DGR2-816.85) and the Cambrian (DGR2-844.95) formations were principally dolomite. Pyrite was present in the shales typically at a few percent. Soluble minerals were relatively rare with halite detected within the Queenston (447-518 mBGS) and Georgian Bay (DGR2-606.62) Formations and anhydrite in the Sherman Fall Formation (DGR2-704.87). The samples DGR2-669.27 and DGR2-677.93 were checked for presence of crystalline forms of anhydrous and hydrated magnesium sulphate, MgSO<sub>4</sub>. The experimental patterns were compared with the simulated patterns of anhydrous and hydrated magnesium sulphate from the PDF4/Minerals database. No evidence of these crystal forms were found in DGR2-669.27 and DGR2-677.93.

<sup>&</sup>lt;sup>1</sup> The Reitveld method is "a *standardless* full profile approach to quantitative phase analysis", i.e., of mineral phases in an X-ray powder-diffraction pattern involving a least-squares fit to the diffractogram. See Raudsepp, M. and E. Pani, 2003, chapter 8: *Application of Rietveld Analysis to Environmental Mineralogy*, in Environmental Aspects of Mine Wastes, editors, J.L. Jambor, D.W. Blowes and A.I.M. Ritchie, Mineralogical Association of Canada, Short Course Series Volume 31, pp. 165-180.

| Intera sample ID | Minerals Present | Concentration (%) | Notes                       |
|------------------|------------------|-------------------|-----------------------------|
| DGR1-049.16      | Calcite          | 41.8              |                             |
|                  | Dolomite         | 55.7              |                             |
|                  | Quartz           | 2.5               |                             |
| DGR1-097.08      | Calcite          | 43.3              |                             |
|                  | Dolomite         | 21.7              | The quartz content          |
|                  | Quartz           | 34.9              | is chert                    |
|                  | Illite           | 0.1               |                             |
|                  | Dolomite         | 99.7              |                             |
| DGR1-130.03      | Quartz           | 0.3               |                             |
|                  | Dolomite         | 98.8              |                             |
| DGR1-156.63      | Quartz           | 1.2               |                             |
|                  | Dolomite         | 76.1              |                             |
| DGR1-231.49      | Quartz           | 5.1               | Small unidentified          |
|                  | Gypsum           | 13.5              | pook of 27 5° 20            |
|                  | Pyrite           | 0.1               | peak at 27.5 20.            |
|                  | Illite           | 4.3               |                             |
|                  | Clinochlore      | 0.9               |                             |
|                  | Dolomite         | 29.6              |                             |
| DGR1-267.78      | Quartz           | 22.7              |                             |
|                  | Gypsum           | 15.0              |                             |
|                  | Halite           | 0.7               |                             |
|                  | Illite           | 28.7              |                             |
|                  | Clinochlore      | 3.2               |                             |
|                  | Anhydrite        | 84.6              |                             |
| DGR1-322.19      | Gypsum           | 2.2               | Small unidentified          |
|                  | Calcite          | 7.8               | peak at 32.8° 20            |
|                  | Dolomite         | 3.4               |                             |
|                  | Quartz           | 1.6               |                             |
|                  | Feldspar (Sr)    | 0.3               |                             |
|                  | Calcite          | 81.1              |                             |
| DGR1-361.76      | Dolomite         | 7.7               | Small unidentified          |
|                  | Quartz           | 1.8               | peaks at $27.5^{\circ}$ and |
|                  | Annyarite        | 1.5               | 28.4 20                     |
|                  |                  | 7.9               |                             |
| DOD4 200 05      |                  | 75.9              |                             |
| DGR 1-399.85     | Doionite         | 10.7              | diffraction peaks           |
|                  | Quartz           | 1.0               | bolder motorial             |
|                  | Illito           | 0.3               | noidei materiai.            |
|                  | Dolomite         | 58.8              | Not full fit of some        |
| DGR1-419 99      | Quartz           | 17.3              | neaks due to                |
| BOR(1-418.55     | Halite           | 1 1               | strong preferred            |
|                  | Illite           | 17.9              | orientation of the          |
|                  | Clinochlore      | 4.8               | clay minerals               |
|                  | Dolomite         | 13.4              |                             |
| DGR1-446.25      | Quartz           | 31.6              |                             |
|                  | Halite           | 0.4               |                             |
|                  | Hematite         | 3.1               |                             |
|                  | Illite           | 47.2              |                             |
|                  | Clinochlore      | 4.3               |                             |
|                  | Calcite          | 35.3              |                             |
| DGR1-455.45      | Dolomite         | 8.6               |                             |
|                  | Quartz           | 15.2              |                             |

| Table 3 | Summary of Mineral Identification and Quantification for DGR-1 Cores |
|---------|--|
|---------|--|

|             | Hematite    | 1.1  |                      |
|-------------|-------------|------|----------------------|
|             | Clinochlore | 9.7  |                      |
|             | Illite      | 30.1 |                      |
|             | Calcite     | 33.1 |                      |
| DGR1-456.01 | Dolomite    | 22.2 |                      |
|             | Quartz      | 16.0 |                      |
|             | Hematite    | 1.4  |                      |
|             | Halite      | 3.1  |                      |
|             | Clinochlore | 7.3  |                      |
|             | Illite      | 16.8 |                      |
|             | Calcite     | 41.1 | Not full fit of some |
| DGR1-460.77 | Dolomite    | 10.8 | peaks due to         |
|             | Quartz      | 11.9 | strong preferred     |
|             | Gypsum      | 0.5  | orientation of the   |
|             | Clinochlore | 8.4  | clay minerals        |
|             | Illite      | 27.3 | -                    |

#### Table 4 Summary of Mineral Identification and Quantification for DGR-2 Cores

| Intera Sample ID | Minerals    | Concentration | Nedez  |
|------------------|-------------|---------------|--|
|                  | Present     | (%)           | Notes  |
|                  | Calcite     | 34.7          |  |
| DGR2-451.33      | Dolomite    | 31.4          |  |
|                  | Quartz      | 11.1          |  |
|                  | Clinochlore | 5.2           |  |
|                  | Illite      | 17.5          |  |
|                  | Calcite     | 17.1          |  |
| DGR2-482.45      | Dolomite    | 17.5          | Small unidentified peak at 27.5° 20            |
|                  | Quartz      | 21.3          |  |
|                  | Clinochlore | 8.7           |  |
|                  | Illite      | 35.5          |  |
|                  | Calcite     | 24.2          |  |
| DGR2-508.93      | Dolomite    | 16.1          | Small unidentified peak at 27.5° 20            |
|                  | Quartz      | 20.2          |  |
|                  | Clinochlore | 7.2           |  |
|                  | Illite      | 32.3          |  |
|                  | Calcite     | 11.2          |  |
| DGR2-535.56      | Dolomite    | 9.5           | Small unidentified peaks at 19.1° and 27.5° 20 |
|                  | Quartz      | 20.0          |  |
|                  | Clinochlore | 8.4           |  |
|                  | Illite      | 51.0          |  |
|                  | Calcite     | 6.4           |  |
| DGR2-550.28      | Dolomite    | 12.1          | Small unidentified peaks at 19.1° and 27.5° 20 |
|                  | Quartz      | 30.2          |  |
|                  | Clinochlore | 14.0          |  |
|                  | Illite      | 37.3          |  |
|                  | Calcite     | 3.2           |  |
| DGR2-570.73      | Dolomite    | 8.9           | Small unidentified peaks at 18.8° and 24.5° 20 |
|                  | Quartz      | 28.5          |  |
|                  | Clinochlore | 11.2          |  |
|                  | Illite      | 48.2          |  |
|                  | Calcite     | 5.2           |  |
| DGR2-590.10      | Dolomite    | 1.7           |  |
|                  | Quartz      | 46.3          |  |
|                  | Pyrite      | 2.9           |  |

|             | Clinochlore   | 10.1 |  |
|-------------|---------------|------|--|
|             | Illite        | 33.7 |  |
|             | Calcite       | 15.9 |  |
| DGR2-606.62 | Dolomite      | 18.0 |  |
|             | Quartz        | 33.5 |  |
|             | Halite        | 1.4  |  |
|             | Clinochlore   | 8.0  |  |
|             | Illite        | 23.3 |  |
|             | Calcite       | 3.5  |  |
| DGR2-606.96 | Dolomite      | 7.9  |  |
|             | Quartz        | 43.1 | Small unidentified peak at 65.2° 20            |
|             | Pyrite        | 1.8  |  |
|             | Clinochlore   | 12.2 |  |
|             | Illite        | 31.6 |  |
|             | Calcite       | 4.1  |  |
| DGR2-626.29 | Dolomite      | 7.8  |  |
|             | Quartz        | 45.2 | Small unidentified peak at 65.2° 20            |
|             | Pyrite        | 1.4  |  |
|             | Clinochlore   | 9.9  |  |
|             | Illite        | 31.6 |  |
|             | Calcite       | 11.5 |  |
| DGR2-644.49 | Dolomite      | 2.2  |  |
|             | Quartz        | 47.2 |  |
|             | Pvrite        | 2.8  |  |
|             | Clinochlore   | 8.5  |  |
|             | Illite        | 27.8 |  |
|             | Calcite       | 74.2 |  |
| DGR2-659 31 | Dolomite      | 21.3 |  |
|             | Quartz        | 3.8  |  |
|             | Illite        | 0.8  |  |
|             | Calcite       | 80.5 |  |
| DGR2-669 27 | Dolomite      | 8 1  |  |
| DONE 000.27 | Quartz        | 6.2  |  |
|             | Illite        | 5.2  |  |
|             | Calcite       | 89.5 | A small amount of gypsum was possibly          |
| DGR2-677 03 | Dolomite      | 60   | present in the clay fraction specimen, which   |
| 0012-011.93 | Quartz        | 0.5  | was not detected in the bulk sample. It is not |
|             | Illito        | 3.1  | possible to conclude if the original sample    |
|             | mite          | 5.1  | contained gypsum or aphydrite, since the       |
|             |               |      | contained gypsum of annyunte, since the        |
|             |               |      | the sample in water                            |
|             | Calaita       | 56 7 | Small unidentified peak at 27.5° 20            |
|             | Delemite      | 30.7 | Small unidentilled peak at 27.5 20             |
| DGR2-095.51 | Duortz        | 42.4 |  |
|             |               | 0.0  |  |
|             |               | 0.1  |  |
|             |               | 0.0  |  |
|             |               | 82.9 |  |
| DGR2-704.87 | Dolomite      | 4.2  |  |
|             | Quartz        | 5.9  |  |
|             | Annyarite     | 1.8  |  |
|             |               | 5.2  |  |
|             |               | 88.6 | Small unidentified peak at 32.3° 20            |
| DGR2-745.97 | Dolomite      | 3.1  |  |
|             | Quartz        | 5.0  |  |
|             | Feidspar (Sr) | 0.3  |  |
|             | Illite        | 3.0  |  |
|             |               | 1    |  |

| DGR2-816.85 | Calcite<br>Dolomite<br>Quartz    | 19.9<br>78.8<br>1.2 | Small unidentified peak at 27.5° 20 |
|-------------|----------------------------------|---------------------|-------------------------------------|
| DGR2-844.95 | Dolomite<br>Quartz<br>Microcline | 97.0<br>2.9<br>0.1  | Small unidentified peak at 29.5° 20 |

## 4.2 Clay Fraction

Typical XRD patterns of the four diagnostic preparations used for clay identification are shown in Figure 1. The low angle diffraction peaks can be assigned to chlorite and illite as indicated by the arrows. The changes (or their absence) in the positions or/and intensities of the peaks for the four preparations are used for the clay identification as shown in the summary Table 5 for illite and chlorite (basal peak orders in square parentheses).



Figure 1 Typical X-Ray Diffraction Patterns of the Four Diagnostics Preparations Used for Clay Identification. Illite and Chlorite Peaks are Indicated by the Arrows

No substantial amount of clay fraction could be separated from six of the fourteen samples of DGR-1 core. The remaining eight samples contained clay fraction in varying amounts and in all of these samples Illite or Illite and Chlorite were identified. Table 6 summarizes the clay identification in the DGR-1 core; where clay was identified, it was typically illite and chlorite. Most of the samples of DGR-2 core also contained illite and chlorite, as shown in Table 7. The clay mineral diffractograms are shown in Appendix C.

# Table 5 Typical Diffraction Peaks Positions and Their Induced Changes Used in the Identification of<br/>Clay Minerals Present in DGR-1 and DGR-2 Cores

| Treatment                    | Peak position  | Peak position | Peak position  |
|------------------------------|----------------|---------------|----------------|
| Air dried oriented           | ~7 Å           | ~10 Å         | ~14 Å          |
| Ethylene glycol              | No change      | No change     | No change      |
| Heated ≈ 400° C              | No change      | No change     | No change      |
| Heated ≈ 550° C              | Destroyed      | No change     | No change      |
| Identification $\rightarrow$ | Chlorite [002] | Illite [001]  | Chlorite [001] |

#### Table 6 Identification of Clay and Other Minerals in the Fine Fraction of DGR-1 Cores

| Intera Sample ID | Clay Minerals<br>Present | Other Minerals Present in the Fine Fraction      |
|------------------|--------------------------|--|
| DGR1-049.16      | No clay                  | Calcite, Dolomite, possibly Hillebrandite        |
| DGR1-097.08      | No clay                  | Quartz, Magnesium Calcite                        |
| DGR1-130.03      | No clay                  | Dolomite   |
| DGR1-156.63      | No clay                  | Dolomite, Quartz                                 |
| DGR1-231.49      | Illite, Chlorite         | Dolomite, Quartz, Gypsum                         |
| DGR1-267.78      | Illite, Chlorite         | Dolomite, Quartz, Gypsum                         |
| DGR1-322.19      | No clay                  | Dolomite, Quartz, Gypsum                         |
| DGR1-361.76      | Illite                   | Dolomite, Quartz, Gypsum, possibly Hillebrandite |
| DGR1-399.85      | No clay                  | Magnesium Calcite, Halite                        |
| DGR1-419.99      | Illite, Chlorite         | Quartz, Dolomite                                 |
| DGR1-446.25      | Illite, Chlorite         | Quartz, Dolomite                                 |
| DGR1-455.45      | Illite, Chlorite         | Quartz, Calcite, Dolomite                        |
| DGR1-456.01      | Illite, Chlorite         | Quartz, Calcite, Dolomite                        |
| DGR1-460.77      | Illite, Chlorite         | Quartz, Calcite, Dolomite                        |

| Intera Sample ID | Clay Minerals<br>Present | Other Minerals Present in the Fine Fraction        |
|------------------|--------------------------|--|
| DGR2-451.33      | Illite, Chlorite         | Quartz, Calcite                                    |
| DGR2-482.45      | Illite, Chlorite         | Quartz, Calcite, Dolomite                          |
| DGR2-508.93      | Illite, Chlorite         | Quartz, Calcite, Dolomite                          |
| DGR2-535.56      | Illite, Chlorite         | Quartz, Calcite, Dolomite                          |
| DGR2-550.28      | Illite, Chlorite         | Quartz, Calcite, Dolomite                          |
| DGR2-570.73      | Illite, Chlorite         | Quartz, Calcite, Dolomite                          |
| DGR2-590.10      | Illite, Chlorite         | Quartz, Calcite, Dolomite                          |
| DGR2-606.62      | Illite, Chlorite         | Quartz, Calcite, Dolomite                          |
| DGR2-606.96      | Illite, Chlorite         | Quartz, Calcite, Dolomite                          |
| DGR2-626.29      | Illite, Chlorite         | Quartz, Calcite,                                   |
| DGR2-644.49      | Illite, Chlorite         | Quartz, Calcite,                                   |
| DGR2-659.31      | Illite, Chlorite         | Magnesium Calcite, Quartz                          |
| DGR2-669.27      | Illite, Chlorite         | Magnesium Calcite, Quartz                          |
| DGR2-677.93      | No clay                  | Magnesium Calcite, Quartz + Gypsum or<br>Anhydrite |
| DGR2-695.51      | Illite                   | Magnesium Calcite                                  |
| DGR2-704.87      | Illite, Chlorite         | Quartz, Magnesium Calcite                          |
| DGR2-745.97      | Illite, Chlorite         | Magnesium Calcite, Quartz                          |
| DGR2-816.85      | No clay                  | Magnesium Calcite, Quartz, Dolomite                |
| DGR2-844.95      | No clay                  | Dolomite, Quartz, Magnesium Calcite                |

| Table 7 | Identification of Clay and Other Minerals in the Fine Fraction of DGR-2 Cores |
|---------|---|
|---------|---|

## 5 Data Quality and Use

As indicated in Tables 3 and 4, for some samples residual low intensity diffraction peaks could not be identified and hence a corresponding mineral(s) was not included in the Rietveld refinement. This introduces an error into the quantification since the calculation procedure constrains the total concentration to 100%. An unidentified peak at 27.5 deg 20 was present in several diffraction patterns, but peaks at other angles were also present although less frequently. The performed Rietveld refinement did not incorporate possible ion substitutions in the crystal structures as variables for the refinement. Taking such substitutions into account might improve the refinement fit at the expense of the number of variables to consider and to justify. The preparation of clay-sized fraction samples would have resulted in the dissolution of any soluble minerals such as halite, anhydrite and gypsum. Therefore, the absence of soluble minerals in these XRD patterns is to be expected. By the same mechanism, the possible detection of gypsum in the Cobourg Formation sample (Table 7, DGR2-677.93) may have been caused by the hydration of anhydrite to gypsum during sample preparation.

#### 6 Conclusions

Cores from DGR-1 and DGR-2 were examined by X-ray powder diffraction both in whole-rock and clay-sized fractions. Calcite, dolomite and quartz were the most common minerals in the Silurian and Devonian formations. Gypsum and anhydrite were common in some of the deeper Salina units and halite was detected in the Salina B Unit and the Gasport Formation of the Middle Silurian and in the Cabot Head and Manitoulin Formations of the Lower Silurian. Clay minerals were sometimes absent in samples collected above the Cabot Head Formation (411-431 mBGS), however when present, illite was the principal clay mineral observed in DGR-1 cores.

The Upper Ordovician shales (447-652 mBGS) sampled by DGR-2 were relatively uniform in mineralogical properties. Calcite, dolomite and quartz comprised 40-80% of the total minerals with illite, chlorite (clinochlore) and pyrite making up the balance. Halite was identified in several Queenston samples and one Georgian Bay sample. The Middle Ordovician limestones (652-778 mBGS) contained minor amounts of dolomite, quartz, chlorite and illite. The deepest sedimentary rocks examined by XRD – the Gull River Formation and the Cambrian – were both principally dolomite and lacked a clay fraction.

#### 7 References

Intera Engineering Ltd., 2009a. Technical Report: Petrographic Analysis of DGR-1 and DGR-2 Cores, TR-07-12, Revision 0, April 16, Ottawa.

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#### APPENDIX A

Search and Match Analysis for Selected Core Samples

(DGR1-322.19, DGR1-399.85, DGR2-606.62, DGR2-816.85 and DGR2-844.95)













User: Aniceta














































## Page: 1 of 1



APPENDIX B

Rietveld Analysis of X-Ray Powder Diffractograms for all DGR-1 and DGR-2 Core Samples

File: A07-0626-1



File: A07-0652-16



File: A07-0765-1



File: A07-0765-2











File: A07-1274-1





File: A07-1247-3



File: A07-1311-1



File: A07-1311-2



File: A07-1311-3



File: A07-2009-1























File: A07-2338-2



File: A07-2338-5






User: Aniceta





File: A07-2536-1



User: Aniceta

File: A07-2536-2



User: Aniceta

APPENDIX C

X-Ray Powder Diffractograms of the Clay-Sized Fraction





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