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CUSTOMIZED SPECTRAL LIBRARIES FOR EFFECTIVE MINERAL EXPLORATION: MINING NATIONAL MINERAL COLLECTIONS

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Abstract—Infrared (Visible-Near Infrared-Shortwave Infrared (VNIR-SWIR)) spectroscopy is a cost-effective technique for mineral identification in the field. Modern hand-held spectrometers are equipped with on-board spectral libraries that enable rapid, qualitative analysis of most minerals and facilitate recognition of key alteration minerals for exploration. Spectral libraries can be general or customized for specific mineral deposit environments. To this end, careful collection of spectra in a controlled environment on pure specimens of key minerals was completed using the National Mineral Reference Collection (NMC) of the Geological Survey of Canada. The spectra collected from specimens in the 'Kodama Clay Collection' were processed using spectral plotting software and each new example was validated before being added to a group of spectra considered for incorporation into the on-board library of the handheld ASD-TerraSpec Halo near-infrared (NIR) mineral identification instrument. Spectra from an additional suite of mineral samples of the NMC containing *REE*, U, Th, and/or Nb are being prepared for a new, publicly available spectral library. These minerals commonly occur in carbonatite or alkali intrusive deposits, and as such will assist in the exploration for critical metals.

Key Words—ASD-TerraSpec, Clay Minerals, Infrared Spectroscopy, National Mineral Reference Collection, Near Infrared, Optical Reflectance Spectroscopy, *REE*-bearing Minerals, Shortwave Infrared.

INTRODUCTION

Explorationists require rapid analytical results to make cost-effective decisions during field and drilling campaigns. Technological advancements in field instrumentation over the last few decades have facilitated acquisition of *in situ*, non-destructive, rapid, qualitative, semi-quantitative, and quantitative chemical analyses, but calibration, interferences, and matrix effects still must be considered. Hand-held X-ray Fluorescence Spectroscopy (XRF) has become more precise (*i.e.* lower detection limits) in detecting elements of interest in exploration programs and Laser-Induced Breakdown Spectroscopy (LIBS) can now be used in the field (Lemière and Uvarova, 2017; Somers and Sholtz, 2017). For certain mineral deposit types, however, the recognition and detection of hydrothermal alteration minerals (*e.g.* Thompson and Thompson, 1996) are of paramount importance for exploration as they form a much larger target than mineralization itself. This is especially important in volcanogenic massive sulfide (VMS) (*e.g.* Date *et al.*, 1983; Shanks, 2012), porphyry copper (*e.g.* Cooke *et al.*, 2014; Halley *et al.*, 2015), epithermal gold (high- or low-sulfidation types) (*e.g.* White and Hedenquist, 1995; Simmons *et al.*, 2005), Iron Oxide-Copper-Gold (IOCG) (*e.g.* Tappert *et al.*, 2013; Mauger *et al.*, 2016), sedimentary exhalative (SEDEX) (*e.g.* Goodfellow and Lydon, 2007), Mississippi Valley-type (MVT) (*e.g.* Leach *et al.*, 2005, 2010), and unconformity-associated uranium deposits (*e.g.* Jefferson *et al.*, 2007a,b) (Table 1). For example, changes in clay mineralogy provide information on genesis and/or alteration history, and may reflect different source rocks, fluids, fluid evolution, or depths of formation. In epithermal gold deposits, the high sulfidation process gives rise to alunite, a characteristic alteration mineral (Berger, 1986). In Proterozoic basins such as the Athabasca and Thelon basins of northern Canada, alteration clay minerals can guide the location of more detailed geophysical surveys, surface sampling, and, ultimately, drilling operations during exploration for unconformity-related uranium deposits (Jefferson *et al.*, 2007b; Riegler *et al.*, 2014).

For rapid mineral identification, both portable X-ray diffraction (XRD) (Sarrazin *et al.*, 2005) and infrared (Visible-Near Infrared-Shortwave Infrared (VNIR-SWIR)) spectroscopy are effective techniques (*e.g.* Thompson *et al.*, 1999). X-ray diffraction requires some sample preparation whereas VNIR-SWIR spectroscopy does not, other than ensuring a dry,

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Table 1. Examples of diagnostic minerals associated with different mineral deposit types (Thompson and Thompson, 1996; Thompson *et al.*, 1999).

Deposit type	Key minerals
Volcanogenic massive sulfide	Fe-chlorite, sericite (muscovite), phengite, Fe-carbonate, quartz
Low sulfidation epithermal gold	Calcite, adularia, illite, smectite, baryte
High sulfidation epithermal gold	Pyrophyllite, alunite, dickite, diaspore, baryte, illite, quartz
Porphyry copper	Illite, kaolinite, smectite, sericite (muscovite), biotite, chlorite, plagioclase, actinolite, epidote, calcite, K-feldspar, anhydrite, hematite, prehnite, zeolites, quartz, pyrophyllite, alunite, dickite
Iron-oxide-copper-gold	Biotite, sericite, amphibole, chlorite, carbonate minerals, albite, K-feldspar, magnetite
Sedimentary exhalative	Fe-carbonate, muscovite, phengite, chlorite, smectite, apatite, tourmaline, baryte, albite
Mississippi Valley	Calcite, illite, kaolinite, quartz
Unconformity uranium	Dickite, kaolinite, illite, sudoite, Fe-chlorite, dravite, Mg-foitite, APS* minerals

* APS = alumino-phosphate-sulfate minerals

contaminant-free surface. Some modern hand-held VNIR-SWIR spectrometers are also equipped with on-board spectral libraries which act as standards in enabling rapid, qualitative analysis of most minerals, and this permits recognition of key alteration minerals, provided the on-board spectral library has been customized for the target mineral deposit environments.

Spectral libraries

Reflectance spectroscopy of minerals began in the early 1900s with work on minerals, inorganic surfaces, and coatings by Coblentz (1906) at the U.S. National Bureau of Standards. Much of the ensuing mineralogical work focused on glass, as well as quartz and cristobalite as analogs for glass structures (Lyon and Burns, 1963). In the early 1970s, a systematic reflectance spectroscopic study of reference rocks and minerals was initiated by Hunt and Salisbury (1970) as they measured the visible (VIS: 350–700 nm; ~28,500–14,300 cm^{-1}), near infrared (NIR: 700–1300 nm; ~14,300–7700 cm^{-1}), and shortwave infrared (SWIR: 1300–2500 nm; ~7700–4000 cm^{-1}) behavior to assist in remote sensing studies. These types of studies (Hunt and Salisbury, 1970, 1971; Hunt *et al.*, 1971a,b,c, 1973; Hunt and Ashley, 1979) were among the first to examine some minerals in the SWIR region. In addition, compilations of the mid- to far-infrared spectra of minerals (Farmer, 1974), clay minerals (Marel and Beutelspacher, 1976), and soil minerals (Kodama, 1985) have made valuable contributions to characterizing common minerals. The early history of reflectance spectroscopy was summarized by Thompson *et al.* (1999), who examined the key infrared "active" minerals (in the SWIR region) occurring in various ore-forming environments.

During the 1990s, development of hand-held instruments permitted direct collection of spectra from rocks and minerals in the field, to ground-truth remotely sensed data (*e.g.* Landsat Thematic Mapper). Several VNIR-SWIR-based instruments were developed during this period, including the GER-IRIS (Geophysical Environmental Research, Inc., CSIRO, Australia), ASD-FieldSpec (Analytical Spectral Devices, Inc., now Malvern Panalytical, Longmont, Colorado, USA, hereafter referred to as ASD-Fieldspec), and PIMA (Portable Infrared Mineral Analyzer, Integrated Spectronics Pty. Ltd, Castle Hill, New South Wales, Australia, referred to hereafter as PIMA). Some of these instruments depended upon solar illumination (*e.g.* GER-IRIS) while others used internal light sources (*e.g.* PIMA) (Thompson *et al.*, 1999; Lau *et al.*, 2003). The ASD-Fieldspec included a contact probe with internal light, but could also function using sunlight. With the arrival of these instruments and concurrent progress in software development, mineral reference databases were created and/or expanded to assist interpretation. Some databases were available with the purchase of an instrument. For example, The Spectral Geologist (TSG, CSIRO, www.thespectralgeologist.com.au) was originally associated with the PIMA and then later with the ASD-TerraSpec4 (Analytical Spectral Devices, Inc., now Malvern Panalytical, Longmont, Colorado, USA, referred to hereafter as ASD-TerraSpec). The SpecMin's (Spectral International, Inc., Arvada, Colorado, USA) database was originally associated with the ASD-FieldSpec. Following SpecMin's acquisition by Spectral Evolution (SEI; Lawrence, Massachusetts, USA), the SpecMin database became part of SEI's EZ-ID software that is associated with SEI's OreExpress instrument (Sherry, 2017). These databases contain a library of reference minerals that can be customized for the geological environment of interest. Subsequently, other reference mineral databases evolved (*e.g.* U.S.G.S. Spectral Library – http://speclab.cr.usgs.gov/spectral-lib.html and the NASA-Jet Propulsion Lab Spectral Library – http://speclib.jpl.nasa.gov/documents/jpl_desc; Clark *et al.*, 1990).

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According to Schneider *et al*. (2009), spectral libraries for hyperspectral purposes could be custom built to map mine faces by including simulated environmental conditions (different light sources, shade, moisture). For details regarding the application of portable infrared spectrometers in geological exploration, see Agar and Coulter (2007) and Sherry (2017).

The National Mineral Collection (NMC) held at the Geological Survey of Canada (GSC) contains >100,000 minerals, of which 68,000 have been catalogued. The 'Catalogue' is an internal GSC database containing mineral accession numbers and accompanying object records. The NMC was formally recognized in 1961 and is jointly managed by the GSC (reference minerals) and the Canadian Museum of Nature (display specimens) (Steacy and Williams, 1976; Stirling *et al.*, 2011). This vast collection provides both an opportunity and a challenge for reflectance spectroscopy studies. Different physical and chemical properties of minerals result in their unique spectral reflectance characteristics and many are "infrared active" due to the presence of molecules that vibrate when they absorb light (Clark, 2004; Hirschmugl, 2004; Hauff, 2005). Spectral reflectance (absorption) features may also be caused by electronic charge transfers (VIS), crystal field effects (VIS/NIR), and conduction bands (UV-SWIR) (Clark, 2004). The molecular vibration (SWIR) energy can be correlated with bond length; hence, the wavelength position of the mineral bonds can be used to identify mineral species (Table 2). Thus, any minerals that are spectrally responsive can be analyzed by infrared spectroscopy to populate a searchable spectral database with which to compare and match unknown minerals analyzed in the field or laboratory. The pure reference-type minerals contained in the NMC (or other national collections) are valuable in that reflection spectra collected from many samples of the same mineral species from different localities will show the possible range in spectral characteristics. In addition, collection using the same instrument by the same team provides consistency in terms of the data.

Analyzing the large number of available samples required to build a comprehensive, all-inclusive database is an enormous challenge. To contribute to this collective endeavor, the NMC was used as a test to determine if high quality spectra suitable for use in a spectral reference library could be acquired from the minerals in the collection. The initial focus was on phyllosilicate minerals collected by Dr H. Kodama (Department of Agriculture and Agri-Food, Canada), as this mineral group is spectrally responsive in a variety of ways (Clark, 2004). The "Kodama Clay Collection" spectral library (Percival *et al.*, 2016a) was modeled after the U.S.G.S. Digital Spectral Library and the NASA-Jet Propulsion Laboratory Library. About 50 spectral reflection datasets (processed as absolute reflectance) are incorporated into the TerraSpec Halo (Analytical Spectral Devices, Inc., now Malvern Panalytical, Longmont, Colorado, USA, referred to hereafter as ASD-Halo) Library, which contains >500 spectra of >150 minerals that are spectrally active in the VIS, NIR, and SWIR regions. The ASD-Halo is one of the most recent field-portable instruments that provides rapid, qualitative analysis of up to seven possible minerals present immediately after spectral collection. Following the success of the Kodama Clay Collection Spectral Library, construction of another spectral library for all (spectrally responsive) minerals associated with critical metal deposits, namely *REE*-, U-, Th-, and Nb-bearing minerals found in carbonatite and alkaline intrusive rocks, is underway.

MATERIALS AND METHODS

Samples

The Kodama Clay Collection includes pure clay minerals from the kaolinite-serpentine, talc-pyrophyllite,

Table 2. Characteristic wavelength bands of major absorption features in the SWIR region for common mineral groups (after Hauff, 2005).

Position (nm)	Molecule	Mineral group
1400–1500	OH and Water	Clays, sulfates, hydroxides, zeolites, prehnite
1560	NH_4	NH_4 species, epidote
1760–1800	OH	Sulfates
1900	Water	Clays
2020, 2120	NH_4	NH_4 species
2160–2220	Al–OH	Minerals containing Al–OH bonds such as clays, sulfates, micas, *etc*.
2230–2295	Fe–OH	Minerals containing Fe–OH bonds such as clays, amphiboles, sulfates, micas, *etc*.
2300–2360	Mg–OH	Minerals containing Mg–OH bonds such as clays, amphiboles, chlorites, *etc*.
2300–2350	CO_3^{2-}	Carbonates
2370–2400	Mg–OH	Amphiboles, talc, *etc*.

Note that although OH is structural, water in these minerals can be in the form of surface-adsorbed water, with varying degrees of polarization, which affects wavelength band position. This not only depends upon mineral type, but also on particle size, water content, and other structural factors.

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mica, chlorite, vermiculite, smectite, and sepiolite-palygorskite groups and from a variety of mixtures and mixed-layer clay minerals. Most of the samples in the library were collected by Dr H. Kodama, but some of the samples were obtained commercially from the American Petroleum Institute (A.P.I. Clay Mineral Standards: IR spectra viewed at http://minerals.gps.caltech.edu/files/atr/myweb5/clay.htm), The Clay Minerals Society Source Clays Repository (http://www.clays.org/source-clays_history.html), Japan Clay Science Society (http://www.cssj2.org/english/reference_clay_e/), and Ward's Natural Science Establishment (now Ward's Science: www.wardsci.com/store/content/externalContentPage.jsp?path=/www.wardsci.com/en_US/products_geology_earth_science.jsp). In all, 166 samples were analysed and spectra were vetted for their quality by comparison with spectra in publicly available spectral libraries or in the scientific literature. In contrast, the NMC contains >1300 specimens representing 152 *REE*-U-Th-Nb-bearing minerals. From this collection, >500 specimens were deemed suitable for spectral analysis and the quality of each spectrum is currently being evaluated.

Spectral analysis

Spectra of each sample were collected using a TerraSpec4 Hi-Res VIS-NIR-SWIR portable spectrometer, which has a variable spectral resolution that ranges from 3 nm in the visible and near-infrared (VNIR) range to 6 nm in the shortwave infrared (SWIR) range, with a spectral sampling of 1 nm across the entire range. To ensure consistently high-quality data collection, a systematic step-by-step approach was adopted, based on ASD-Malvern Panalytical (Longmont, Colorado, USA) protocols. All measurements were made in a 'dark box' (Figure 1a) to reduce interference from extraneous natural and artificial (*e.g.* fluorescent) light. Each day, prior to use, the spectrometer system was calibrated using a wavelength standard (composed of rare earth oxides + Mylar) from ASD-containing spectral features across the entire 350–2500 nm wavelength range to ensure wavelength consistency across all reference mineral samples that were scanned. Automated software (*Wavelength Verification*, ASD–Malvern Panalytical) was used to ensure consistent interpretation and identification of

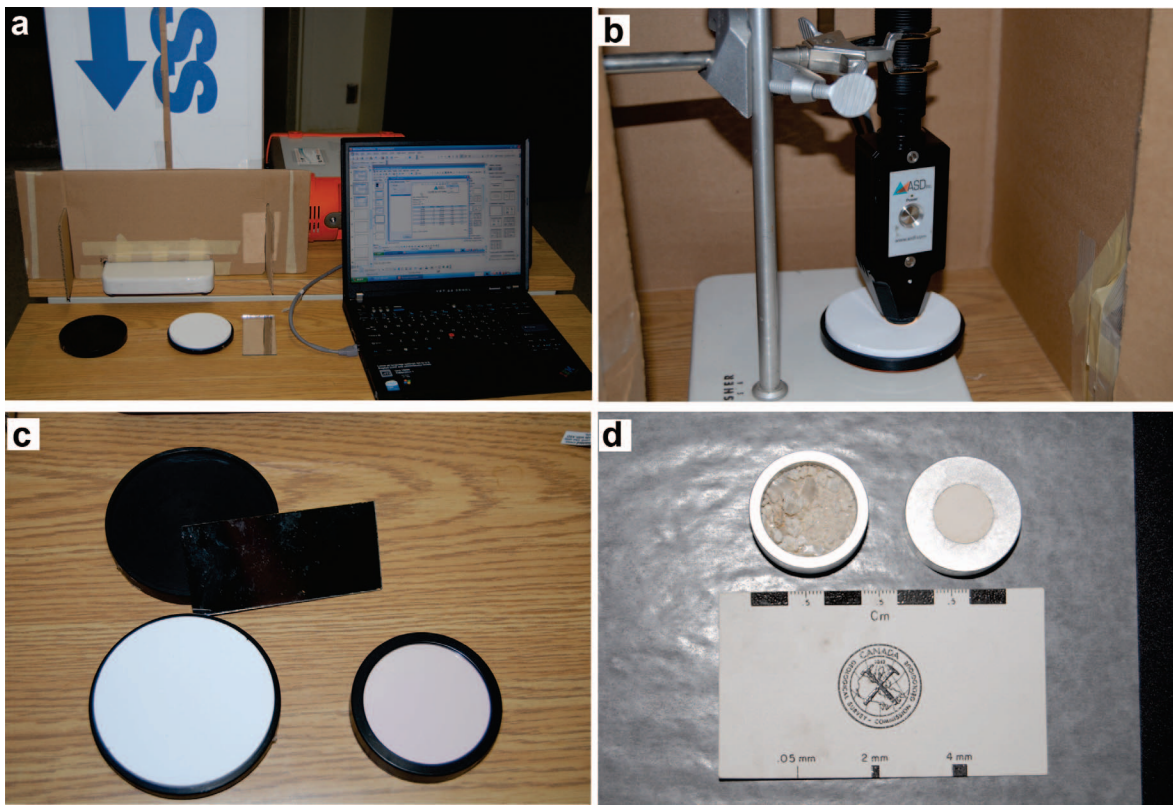

Figure 1. Dark box set-up for collecting spectra: (a) dark box and computer set-up; (b) retort stand in box holding probe in contact with the Spectralon puck; (c) Spectralon puck, calibration puck, and back of holder for dark current measurements; (d) aluminum holders used for packing powder samples.

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wavelength performance (*i.e.* Terraspec scans the rare earth oxides + Mylar standard puck and wavelengths must be within ±0.2 nm, otherwise the calibration fails). Subsequent steps included wavelength verification of the instrument at the beginning of each collection day, followed by optimization procedures, collection of white reference (using Spectralon puck; Figure 1b,c) and dark current (Figure 1c). For all measurements, the instrument and contact probe were turned on for at least 40 min before spectral measurements, to ensure that the probe and spectrometers were sufficiently warmed up and stable.

For every sample, three spectra were collected on the same area (spot size = 10 mm) using *IndicoPro* instrument controller software (ASD-Malvern Panalytical). Powdered samples were pressed into small aluminum holders (Figure 1d) and filled to the top to ensure a good seal with the contact probe. After collection, the spectra were brought into *ViewSpec Pro* software (ASD-Malvern Panalytical) for further processing. First, each spectrum was corrected using 'splice correction' to smooth the spectrometer transition (offset) steps produced between the VNIR and SWIR1 detectors (at 1000 nm), and the SWIR1 and SWIR2 detectors (at 1830 nm). For the Kodama Clay Collection, each spectrum was exported as an ASCII-type file. Each set of spectra was then brought into *MS-Excel*® and averaged. The average spectrum was then used to create the plots and exported as a .pdf file. The averaged data with wavelength information was then processed to create a .txt file for easy accessibility (Percival *et al.*, 2016a). For the *REE*-U-Th-Nb-bearing minerals, ASD's *IndicoPro* software averaged the three spectra during collection. The averaged spectrum was then exported as an ASCII-type file and processed as described above.

REFLECTANCE SPECTROSCOPY IN MINERAL EXPLORATION

Near infrared reflectance spectroscopy has gained wide acceptance in exploration programs through remote sensing applications (Duke, 1994; Perry, 2004; Bedini, 2017) and field and laboratory measurements (Thompson *et al.*, 1999; Zhang *et al.*, 2001; Kerr *et al.*, 2011; Peter *et al.*, 2015). Early field and laboratory work focused on the application of the SWIR region in identifying minerals of interest in alteration zones (Hunt and Ashley, 1979; Thompson *et al.*, 1999). Current work makes use of the full VIS-NIR-SWIR regions (350−2500 nm) to characterize common and uncommon minerals in a variety of environments of formation. With improved hyperspectral mapping capabilities combined with ground truthing (Neave *et al.*, 2016), explorationists will continue to utilize these technologies. Some examples are described briefly below.

Volcanogenic massive sulfide deposits are characterized by hydrothermal transformations of host rocks (*e.g.* rhyolite) resulting in a variety of minerals, especially fine-grained chlorite and sericite. Early studies in Tasmania using a PIMA-II spectrometer demonstrated that white micas grade from phengite (peripheral zones) to sodic muscovite (proximal zones) in the Rosebery stratiform Zn+Pb deposit (Herrmann *et al.*, 2001). These types of findings were supported by Yang *et al.* (2011) who studied changes in white mica at the Hellyer polymetallic VMS deposit (Tasmania). More recently, VNIR-SWIR spectroscopy was applied by Laakso *et al.* (2015, 2016) to differentiate phyllosilicates based on their Al-OH and Fe-OH absorption features in a study of the Izok Lake VMS deposit, Nunavut, Canada. Those authors determined that proximal phyllosilicates had an average Al-OH (white mica) absorption feature at 2203 nm *vs.* a distal Al-OH absorption feature at 2201 nm, and Fe-OH (biotite/chlorite) absorption features at 2254 nm (proximal) *vs.* 2251 nm distal phyllosilicates. These slight spectral shifts can be used to detect, remotely, changes in mineralogy, not only at the hand-sample scale using a field-portable instrument, but also at the outcrop scale (*e.g.* 0.5−1.0 m spatial resolution) using a hyperspectral sensor deployed from a fixed-wing aircraft (*e.g.* Ruitenbeek *et al*., 2012).

While investigating the possibility of using spectral analysis in exploration, a series of pilot studies was carried out by Kerr *et al.* (2011) in Newfoundland, Canada. In the case of epithermal gold deposits, Kerr *et al.* (2011) applied this technology to Bobbys Pond and Western Avalon districts. For Bobbys Pond, a barren zone was examined and pyrophyllite, alunite, and topaz were identified readily. Detection of the presence of topaz, difficult to recognize in hand specimens or petrographically, was determined to be geologically feasible (Kerr *et al.*, 2011), as also reported by others (Taylor, 2007). In the Western Avalon studies, in addition to identifying pyrophyllite and alunite, dickite was detected. This mineral is an indicator of Au mineralization in high-sulfidation epithermal systems (Kerr *et al.*, 2011). Other studies at the Mankayan Cu-Au district in Luzon, Philippines (Chang *et al.*, 2011), and the Cerro La Mina epithermal prospect in Patagonia, Argentina (Ducart *et al.*, 2006), have also demonstrated the application of SWIR in identifying advanced argillic alteration in these systems using hand-held instruments and hyperspectral remote sensing.

Exploring for IOCG deposits using reflectance spectroscopy is more problematic. The IOCG deposits are characterized by an abundance of Ti-poor iron oxides and sulfide-deficient ore, and the host rocks tend to be intensely altered by telescoping metasomatic reactions. Vectoring to mineralization is possible, however, through recognition of the systematic, prograding alteration assemblages or metasomatic facies in the field (Corriveau *et al.*, 2010, 2016; Potter *et al.*, 2013; Percival *et al.*, 2016b). Early, regional-scale metasomatic minerals include albite, K-feldspar, magnetite,

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and amphibole-group minerals, of which only amphibole-group minerals are infrared active. As the systems evolve to biotite- and sulfide-bearing facies (high-temperature magnetite K-Fe to lower-temperature hematite K-Fe assemblages and hydrothermal brecciation), however, the metasomatic reactions produce mica, chlorite, and carbonate minerals (Corriveau *et al.*, 2010, 2016), which tend to be very infrared active. The composition of white mica minerals was shown by Tappert *et al.* (2013) to be a useful indicator for IOCG-type deposits. Those authors examined the composition of phengite associated with the Olympic Dam deposit in South Australia with near-infrared and mid-infrared analyses and reported that phengite associated with mineralization in intensely sericite-altered zones has less Si and more Al relative to phengite from weakly sericite-altered zones. This high-Al phengite is characterized by an Al-OH absorption feature at 2206 nm compared to 2213 nm for the lower-Al phengite. Measurements in the mid-infrared region produced several stronger reflectance peaks that also distinguish these two varieties of phengite (Tappert *et al.*, 2013). At the Olympic Dam deposit, wavelength shifts for sericite and chlorite can be used to vector to mineralization in an IOCG ore-forming system (Mauger *et al.*, 2016)).

The application of near-infrared spectroscopy to sedimentary-hosted (sedimentary exhalative or SEDEX) lead-zinc deposits is relatively new. This technology was tested by Peter *et al.* (2015) in a pilot study in the Selwyn Basin, Yukon, Canada, on deposits in the Howard's Pass and MacMillan Pass districts. These deposits are stratabound and lack discrete, well developed hydrothermal alteration zones adjacent to mineralization. The "ore" minerals are pyrite, galena, and sphalerite, with siderite, white mica (muscovite, phengite), montmorillonite, apatite, ±baryte, and K-Ba-feldspar comprising gangue minerals. The organic carbon and pyrite contents of the black shale host rocks make them spectrally challenging due to low reflectance and weak absorption features. Furthermore, many of these minerals have no (VNIR-SWIR) spectral signatures, and those that do (*e.g.* white mica, carbonate, apatite, baryte) are generally present at insufficient levels to be detected reliably by spectroscopy. The mineralization (and immediately adjacent host shale) at Howard's Pass is characterized by phengite ($\geqslant$2214 nm), whereas muscovite ($\leqslant$2209 nm) prevails everywhere else. At MacMillan Pass, siderite, muscovite, phengite, and montmorillonite are all identified spectrally immediately below and within the mineralized zone. Further work on the application of reflectance spectroscopy to this deposit type is warranted to evaluate its utility in the exploration for SEDEX deposits.

In a study of the Pebble porphyry Cu-Au-Mo deposit in Iliamna, Alaska, SWIR spectroscopy was demonstrated by Harraden *et al.* (2013) as suitable for use to distinguish illite, sericite, kaolinite, and pyrophyllite in drill core. Those authors assigned alteration types to different ranges of Al-OH absorption features between 2160 and 2220 nm and produced a detailed map. These authors noted that the Au and Cu mineralization is closely associated with the lower wavelengths of the Al-OH absorption features associated with pyrophyllite and sericite and concluded that SWIR spectroscopy is a useful tool for exploration as the clay minerals were linked genetically to mineralization. In another study on a porphyry Cu deposit from Batu Hijau, Indonesia, chlorite absorption features near 2250 and 2340 nm were determined by Neal *et al.* (2018) to correlate with the Mg# (Mg/[Mg+Fe]) with respect to wavelength position and depth of features. As Mg# increases, the absorption minimum wavelengths at 2249 and 2332 nm were noted to increase to 2254 and 2343 nm, respectively. Reflectance spectroscopy is useful in studying chlorite minerals in propylitic alteration zones according to Neal *et al.* (2018).

In the search for diamondiferous kimberlites, Hauff and Percival (2006) showed that differentiating G-10 garnets from non-chrome-bearing garnets is possible. This was accomplished by placing the fiber optic cable from the contact probe of an ASD Field SpecPro directly onto small (~1 mm) garnet grains extracted from heavy mineral concentrates (Figure 2). The spectral range for the chrome absorption feature is between 478 and

Figure 2. Fiber optic cable used to capture directly a VNIR-SWIR spectrum from a small discrete garnet grain.

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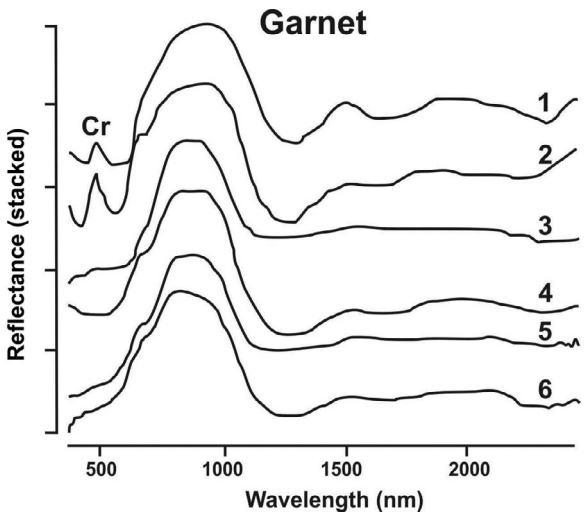

Figure 3. Stacked VNIR-SWIR reflectance spectra of garnet minerals in kimberlite exploration. (1) G-10 garnet from Roberts Victor Mine, South Africa; (2) G-10 garnet from Tanzania; (3) G-9 garnet from Tanzania; (4) G-9 garnet from Kirkland Lake, Canada; (5) G-5 garnet from Tanzania; and (6) G-1/G-2 garnet from Jagersfontein, South Africa. Samples obtained from Dan Schulze (University of Toronto, numbers 1 and 4) and Bruce Kjarsgaard (GSC, numbers 2, 3, 5, and 6) (after Hauff and Percival, 2006).

481 nm while the purple-red color is captured in the VIS part of the spectrum (Figure 3). The application of reflectance spectroscopy to diamond exploration, however, is restricted, at present, to field and laboratory analyses rather than through airborne or satellite-based remote sensing due to the scarcity, rarity, and small grain size of the indicator minerals.

Field-portable instruments have been used since the mid-1990s in the exploration for unconformity-associated uranium deposits in the Athabasca Basin, Saskatchewan, Canada (Earle, 1994, 1995, 1996; Earle *et al.*, 1996). The initial work involved the use of a PIMA-II instrument to estimate the proportion of clay minerals using an empirical approach (Earle, 1994). This proposal was tested rigorously using synthetic admixtures of the clay (dickite, kaolinite, illite, clinochlore, sudoite) (Figure 4) and non-clay (quartz, dravite, or magnesiofoitite of the tourmaline group) minerals that reflect diagenetic and hydrothermal alteration of the red-bed sandstones, and possibly vector to ore (Zhang *et al.*, 2001). In creating the binary and ternary mixtures of the various minerals, the empirical method for quantitative analysis was improved by Zhang *et al.* (2001) who also reported that clay minerals present in abundances as low as 1% were detectable, whereas in XRD analyses, at least 3–5 wt.% of a mineral is required for detection.

Mapping the distribution of clay minerals in the overlying sandstone, clay-rich intraclasts, or discrete layers during down-hole logging can assist in vectoring to ore (Percival *et al.*, 2002, 2013) and highlights subtle changes in clay-mineral distribution between the east and west sides of the Athabasca Basin (Figure 5; Bosman and Percival, 2014). Clay mineralogical changes from diagenetic dickite to hydrothermal illite are commonly observed to be associated with uranium deposits in the eastern part of the basin (Hoeve and Quirt, 1984, 1987; Percival *et al.*, 1993; Kyser *et al.*, 2000; Zhang *et al.*, 2001; Quirt, 2010; ASD, 2013). In contrast, mineralogical changes appear to be less extensive and more subtle in the western part of the basin and deposits hosted in crystalline basement rocks (Figure 5).

The increasing demand for high-technology devices has sparked increased interest in exploring for critical metals including *REE*, platinum-group elements, nio-

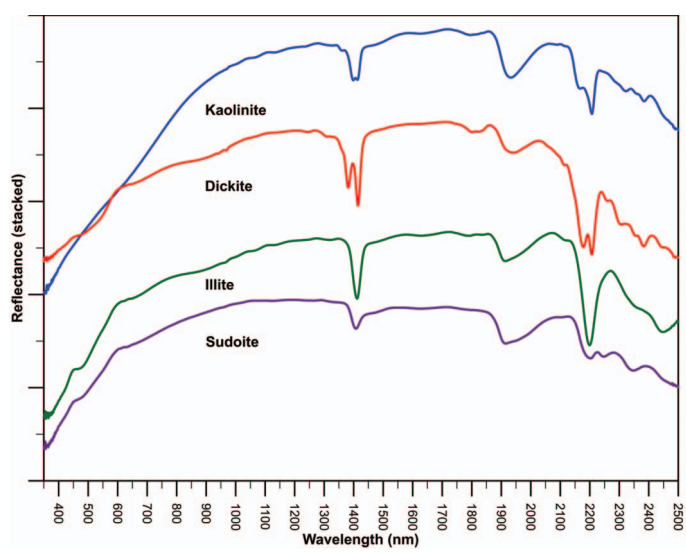

Figure 4. Stacked VNIR-SWIR reflectance spectra of key minerals of interest for unconformity-associated uranium exploration in the Athabasca Basin, Saskatchewan, Canada.

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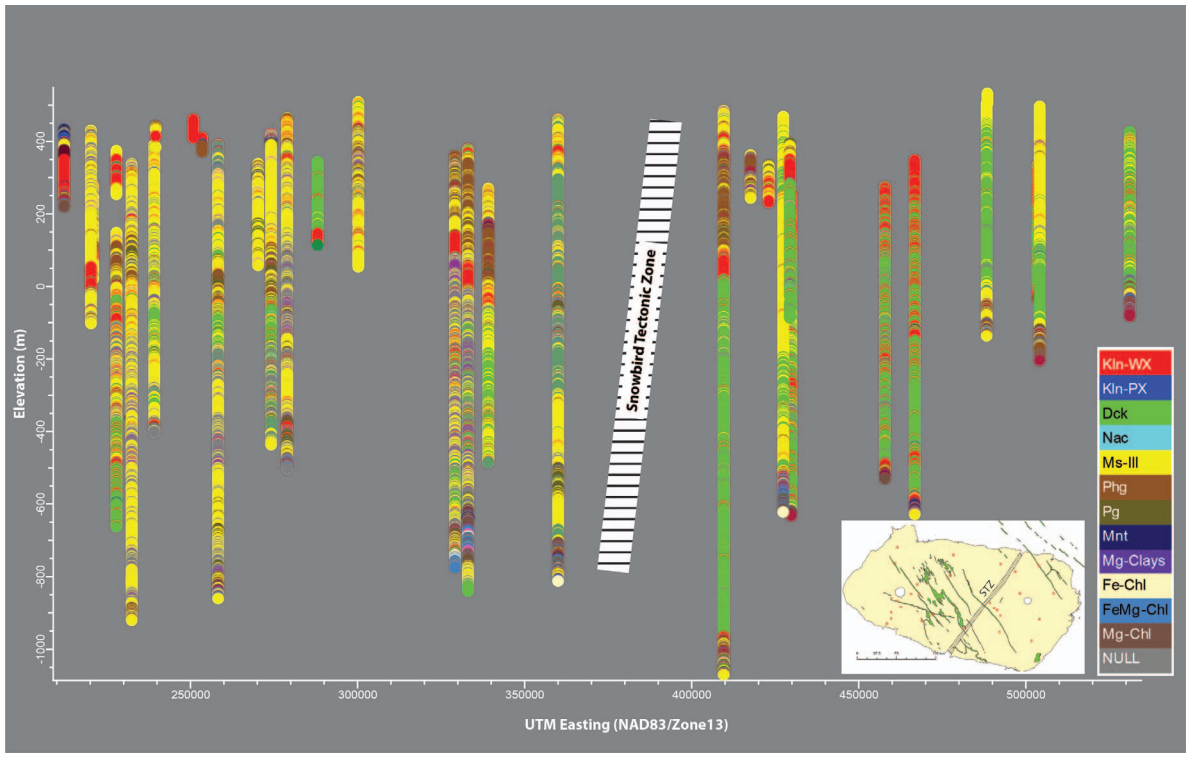

Figure 5. Comparison of clay distribution between the east and west sides of the Athabasca Basin, Saskatchewan, based on plotting in *The Spectral Geologist* (TSG, CSIRO) software (version 7.1.0.069). Note the change from the east side (dominant green = dickite) to the west side (dominant yellow = muscovitic/illite) of the Basin. Inset is a generalized map of Athabasca Basin showing the spatial distribution of drillholes (red circles), surface projection of mafic intrusive rocks interpreted from airborne magnetic data (green polygons), and the location of the Snowbird Tectonic Zone (STZ) which separates the Rae Province (west) from the Hearne Province (east). Kln-Wx: Well crystalline kaolinite; Kln-Px: poorly crystalline kaolinite; Dck: Dickite; Nac: nacrite; Ms-Ill: muscovitic Illite; Phg: phengite; Pg: paragonite; Mnt: montmorillonite; Mg-clays: magnesium clays; Fe-Chl: iron chlorite; Fe/Mg-Chl: iron-magnesium chlorite; Mg-Chl: magnesium chlorite. Less common minerals were removed from the legend.

bium, tantalum, indium, and tungsten (Percival *et al.*, 2017). The principal host of *REE*-bearing minerals is carbonatite or peralkaline granite rocks. The use of reflectance spectroscopy was evaluated by Kerr *et al.* (2011) to identify *REE*-bearing minerals in Labrador. Those authors noted that the minerals produce absorption features in the NIR region rather than the typical SWIR region and, hence, these properties could be used to detect these unusual minerals. In a more recent study (Neave *et al.*, 2016)), neodymium was shown to produce a strong absorption feature in the NIR and could be used as a pathfinder element for *REE* mineralization associated with carbonatites. Certain samples from various deposits (worldwide) were selected by Neave *et al.* (2016), who suggested that the results could be applied to hyperspectral studies at meter-scale resolution. Their work built upon previous work by Rowan *et al.* (1986) who compared reflectance spectra for rock samples (*i.e.* okaite, rauhaugite, sövite) from four North American localities (Mountain Pass, California; Iron Hill, Colorado; Gem Park, Colorado; and Oka, Quebec). Neodymium, the most abundant *REE*, was demonstrated by Rowan *et al.* (1986) to produce absorption features that were detectable even in rocks with low (~300 ppm) Nd abundances. Similar evidence, based on research on Nd in apatite and carbonate minerals hosted by the *REE*-bearing carbonatite deposit at Cummins Range, Western Australia, was provided by Huntington and Laukamp (2015). Hyperspectral mapping, with a tripod-mounted Hypex imager, was used (Boesche *et al.*, 2015) for the detection of Nd in carbonatites of the Fen Complex, Norway. Those authors concluded that spectroscopy-enabled mapping of outcrops for *REE*-bearing minerals was possible, provided acquisition conditions were good (no rain, no clouds) and the outcrops had little to no weathering. A study of the *REE*-bearing fluorocarbonate minerals bastnäsite, parisite, and synchysite as well as the *REE*-bearing phosphates monazite and xenotime showed deep absorption features in the NIR and SWIR regions corresponding to various lanthanide elements present in the crystal structure (Turner, 2015; Turner *et al.*, 2014, 2015). Those authors also determined that carbonate-absorption features at 2243 nm and a doublet at 2312 and 2324 nm were typical for bastnäsite, whereas synchysite and parisite have carbonate-related absorption features at 2337 and 2324 nm, respectively.

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According to Turner *et al.* (2014), the spectral signatures of these carbonate-bearing minerals were also distinct from the *REE*-bearing phosphates mentioned above.

BUILDING SPECTRAL LIBRARIES

Mineral libraries available with the purchase of a spectrometer tend to be proprietary. They include the common VNIR-SWIR active minerals, especially those with strong SWIR patterns. The libraries may be available on-board the instrument, or through third-party software such as TSG (CSIRO) or Specmin (Spectral International). These libraries, however, are typically not comprehensive, and the result is that analyzed minerals that are not in the library will be difficult to identify. Although several public domain libraries are available as noted above, accessible, searchable reference libraries, in standardized formats, would be valuable for interpretation (Kerr *et al.*, 2011).

Kodama Clay Collection

The National Reference Mineral Collection of the GSC contains many pure specimens that have been characterized by X-ray diffraction (XRD) and/or electron probe microanalyses (EPMA). The Kodama Clay Collection was the initial test (Figure 6; Percival *et al.*, 2016a). This Spectral Library contains information about the specimen analyzed, including collection date, locality, and brief sample description; a text file of the data (relative reflection) so that it can be manipulated by other programs for plotting; and an image of the plot generated in *MS-Excel*®. The minerals in this sub-collection of the NMC were organized according to Dana's classification system (see Gaines *et al.*, 1997). The Kodama Clay Collection Spectral Library is a free, downloadable Open File consisting of a series of zip files (https://doi.org/10.4095/297564).

Many of the minerals in the Kodama Clay Collection have been described in previous publications; hence, the mineral specimens were deemed to be monomineralic. Sufficient material from the mineral samples exists to compare polytypes (Figure 7) and changes in grain size (Figure 8). For example, stacked reflection spectra of the kaolin group (Figure 7) show the changes in absorption features between less crystalline minerals (halloysite and kaolinite) relative to the well crystallized minerals (dickite and nacrite; sharp and well developed doublet). The different grain-size fractions for biotite from the Bancroft, Ontario, area, however, show very little difference among the spectra. The spectra collected from the various specimens were assessed by ASD, Inc., and select spectra, based on their quality, representativeness, or uniqueness were incorporated into the on-board library for the ASD-Halo instrument.

REE-, U-, Th-, and Nb-bearing minerals

A similar spectral library of *REE*-U-Th-Nb-bearing minerals is being built. From the specimens available, just over 500 samples were deemed appropriate for analysis, resulting in spectra for 152 mineral species (Table 3). Rejection was based on the quality of the spectrum, grain size, and/or the presence of impurities/intergrowths in the sample. Reflection spectra collected from some typical *REE*-bearing minerals that occur in carbonatite or alkali intrusive environments are shown in Figure 9. Note that for bastnäsite ($(Ce,La,Y,Nd)(CO_3)F$), the main absorption features occur in the VIS to NIR regions of the electromagnetic spectrum, between 550 and 950 nm, whereas for monazite ($(Ce,La,Nd)(PO_4)$), absorption features in this region appear flatter when plotted in this stacked format. The synchysite ($Ca(REE)(CO_3)_2F$) sample may be contaminated with another mineral based on the deep absorption features near 1450 and 1900 nm, or may represent a lanthanide element and water absorption

Spectrum Title	Date Spectra Collected	ID #	Description	Data	Plot(s)
71.01 Phyllosilicates, Phyllosilicate Sheets of Six-Membered Rings with 1:1 layers					
71.01.01 Kaolinite group, $Al_2Si_2O_5(OH)_4$					
71.01.01.01 Dickite					
NMC068170 Dickite (Massive)	June 14 2013	1001	Description	Data	Plot
NMC068170 Dickite (Rusty)	June 14 2013	1002	Description	Data	Plot
NMC068170 Dickite (Creamy White)	June 14 2013	1003	Description	Data	Plot
NMC068171 Dickite (Powder)	July 24, 2013	1004	Description	Data	Plot
71.01.01.02 Kaolinite					
NMC068120 Kaolinite (KGa-2)	August 15, 2013	1005	Description	Data	Plot
NMC068121 Kaolinite (H-5; Massive)	July 24, 2013	1006	Description	Data	Plot
NMC068122 Kaolinite (H-1)	July 14, 2013	1007	Description	Data	Plot
NMC068123 Kaolinite (H-9; Massive)	July 16, 2013	1008	Description	Data	Plot
NMC068124 Kaolinite (Powder)	August 15, 2013	1009	Description	Data	Plot

Figure 6. Set-up of the Spectral Library of the Kodama Clay Collection. Minerals listed according to Dana's Classification. Description, data as text file, and plot are hyperlinked for viewing and downloading.

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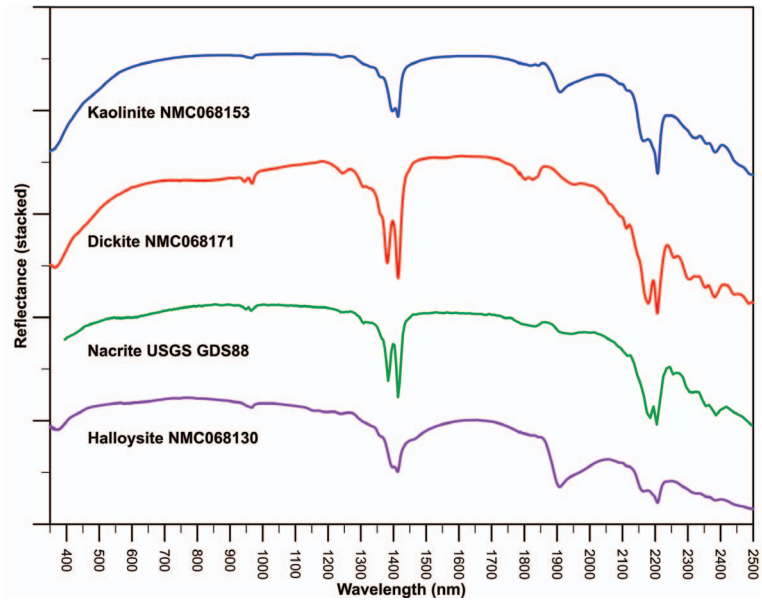

Figure 7. Stacked VNIR-SWIR reflection spectra of kaolin mineral-group polytypes. Nacrite obtained from the U.S.G.S. Spectral Library website (http://speclab.cr.usgs.gov/spectral-lib.html).

feature as described by Turner *et al.* (2014). The apatite sample appears to have a typical reflection pattern for this group of minerals.

To illustrate the utility of a spectral library, several spectra of bastnäsite, synchysite, and monazite samples were plotted (Figures 10–12, respectively). The bastnäsite samples (Figure 10) consistently show deep absorption features in the NIR. Strong minima were found by Turner *et al.* (2014, 2015) at 580, 741, doublet at 792 and 797, and 864 nm, as well as other absorption minima in this region, and strong reflections at 1093, 1251, 1547, 1968, 2243, and 2342 nm in the SWIR region. The absorption minima in the present samples (Figure 10) at 1968 nm are broad and open in comparison to other absorption minima, but the overall patterns correspond well with the spectra of Turner *et al.* (2014, 2015). The samples analyzed from the NMC were collected from different localities (California, Colorado, and Madagascar) and two of the samples have been verified by XRD.

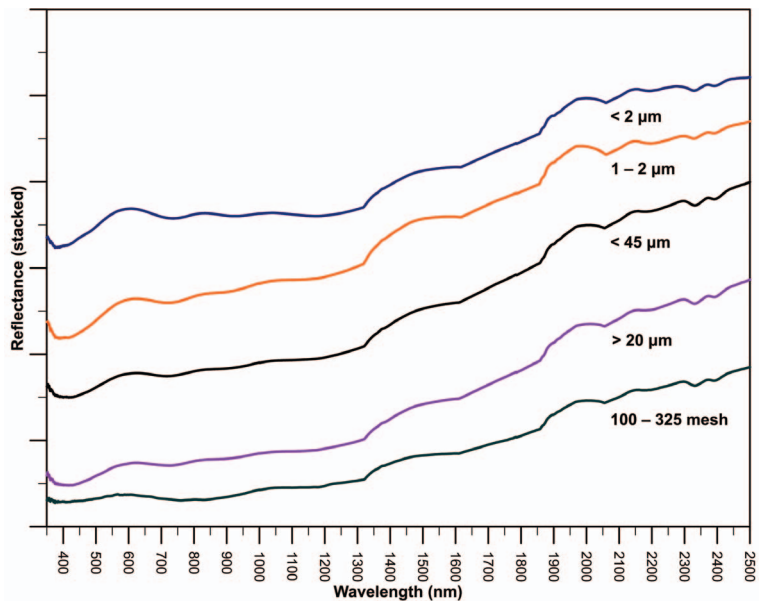

Figure 8. Stacked VNIR-SWIR reflection spectra of biotite samples (not catalogued) from the Bancroft, Ontario, region.

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Table 3. List of minerals and number of specimens in the NMC containing *REE*, U, Th, and/or Nb.

Mineral	No.	Mineral	No.	Mineral	No.	Mineral	No.
Aeschynite-(Ce)	16	Eudialyte	43	Labuntsovite	4	Sazhinite-(Ce)	1
Aeschynite-(Y)	6	Euxenite-(Y)	31	Lanthanite-(Ce)	1	Sazykinaite-(Y)	1
Agardite-(La)	1	Ewaldite	3	Lanthanite-(La)	12	Schuilingite-(Nd)	4
Agardite-(Y)	3	Fergusonite-(Y)	25	Lanthanite-(Nd)	6	Semenovite-(Ce)	2
Allanite-(Ce)	66	Fersmanite	2	Laplandite-(Ce)	2	Senaite	1
Allanite-(La)	5	Fersmite	3	Latrappite	6	Shcherbakovite	1
Allanite-(Y)	1	Florencite-(Ce)	3	Lokkaite-(Y)	4	Simpsonite	4
Alluaivite	1	Fluocerite-(Ce)	4	Loparite-(Ce)	3	Steenstrupine-(Ce)	8
Ancylite-(Ce)	10	Fluorapatite	242	Loranskite-(Y)	1	Stibiotantalite	1
Ashcroftine-(Y)	1	Franconite	7	Lueshite	2	Stillwellite-(Ce)	6
Baotite	2	Gadolinite-(Ce)	2	Mckelveyite-(Y)	1	Synchysite-(Ce)	5
Bastnäsite-(Ce)	15	Gadolinite-(Y)	20	Melanocerite-(Ce)	7	Synchysite-(Y)	4
Belovite-(Ce)	1	Gagarinite-(Y)	4	Mineevite-(Y)	1	Tantalaeschynite-(Y)	1
Bornemanite	1	Gasparite-(Ce)	1	Miserite	8	Tanteuxenite-(Y)	6
Braitschite-(Ce)	1	Gerasimovskite	1	Monazite-(Ce)	47	Tapiolite-(Fe)	2
Brannerite	24	Gerenite-(Y)	20	Monazite-(La)	1	Tapiolite-(Mn)	2
Britholite-(Ce)	9	Gittinsite	13	Monteregianite-(Y)	2	Tengerite-(Y)	7
Britholite-(Y)	1	Goudeyite	3	Murmanite	4	Thalénite-(Y)	8
Brockite	2	Gysinite-(Nd)	1	Nenadkevichite	5	Thorbastnäsite	1
Burbankite	4	Hainite	1	Niobophyllite	2	Thoreaulite	1
Calcioancylite-(Ce)	2	Hellandite-(Y)	5	Niocalite	11	Thorite	38
Calciosamarskite	2	Hibonite	2	Nordite-(La)	1	Thortveitite	4
Carbocernaite	1	Hochelagaite	3	Normandite	1	Tombarthite-(Y)	2
Caysichite-(Y)	6	Huanghoite-(Ce)	2	Okanoganite-(Y)	1	Tritomite-(Y)	10
Cebaite-(Ce)	2	Hydroxylbastnäsite-(Ce)	2	Olmsteadite	1	Tundrite-(Ce)	2
Cerianite-(Ce)	1	Iimoriite-(Y)	1	Parisite-(Ce)	6	Vicanite-(Ce)	2
Cerite-(Ce)	4	Ilmajokite	2	Perovskite	18	Vigezzite	1
Cheralite	1	Ishikawaite	2	Perrierite-(Ce)	1	Vitusite-(Ce)	2
Chernovite-(Y)	1	Ixiolite	3	Phosinaite-(Ce)	1	Vuonnemite	5
Chevkinite-(Ce)	4	Janhaugite	2	Polycrase-(Y)	7	Wakefieldite-(Y)	3
Churchite-(Y)	3	Joaquinite-(Ce)	7	Pyrochlore	54	Wöhlerite	4
Columbite Series	54	Kainosite-(Y)	11	Rankamaite	1	Xenotime-(Y)	10
Cordylite-(Ce)	3	Kamphaugite-(Y)	3	Retzian-(Nd)	1	Yttrialite-(Y)	1
Crichtonite	1	Karnasurtite-(Ce)	2	Rhabdophane-(Ce)	2	Yttrotantalite	1
Davidite-(La)	11	Keiviite-(Y)	4	Rinkite	16	Yttrotungstite-(Y)	5
Dollaseite-(Ce)	2	Kimuraite-(Y)	1	Rowlandite-(Y)	1	Yuksporite	2
Donnayite-(Y)	7	Kobeite-(Y)	3	Sahamalite	2	Zircon	111
Epistolite	10	Kupletskite-(Cs)	2	Samarskite-(Y)	18	Zorite	1

To demonstrate consistency in the spectral patterns, synchysite minerals are plotted in Figure 11. For synchysite, strong absorption minima were found (Turner *et al.*, 2014, 2015) at 580, 740, doublet at 792 and 797, and 864 nm in the NIR region, as well as 1093, 1251, 1547, and 1961 nm in the SWIR region. The synchysite samples examined in the present study (Figure 11) do not show the doublet at 792 and 797 nm. The spectral pattern for these minerals matches poorly the Turner *et al.* (2014, 2015) spectral patterns, and the deep, sharp reflection at ~1450 nm indicates the possible presence of a second mineral. Two of the NMC samples were confirmed by XRD to be synchysite. A variety of monazite samples from the collection (18 of 48 samples available) are shown in Figure 12. The absorption minima in the NIR and SWIR regions are more visible and complex than those shown in Figure 9. More detailed analyses and comparison with published spectra are required.

Infrared reflectance spectra from red to red-brown eudialyte ($Na_4(Ca,Ce)_2(Fe^{2+},Mn^{2+},Y)\ ZrSi_8O_{22}(OH,Cl)_2$) samples are shown in Figure 13. The color of the specimens is evident in the VIS part of the spectrum and two types of spectral patterns are apparent: one set with distinct NIR absorption features, the second set with a more subdued, flatter pattern. The flatter spectra were collected from a group of crystal fragments in the holder and, thus, the probe may not have been in full contact with the fragments. The nature of the mineral specimen may affect the reflectance spectra.

The xenotime (YPO_4) (Figure 14) samples from five different regions show more consistency in their spectral pattern with numerous absorption minima in the NIR and SWIR regions. Similar to the eudialyte samples, spectra 4 and 5 show flatter SWIR patterns than the others. Both samples are described as crystal fragments; thus, contact may have been poor between the probe and the fragments as noted for eudialyte.

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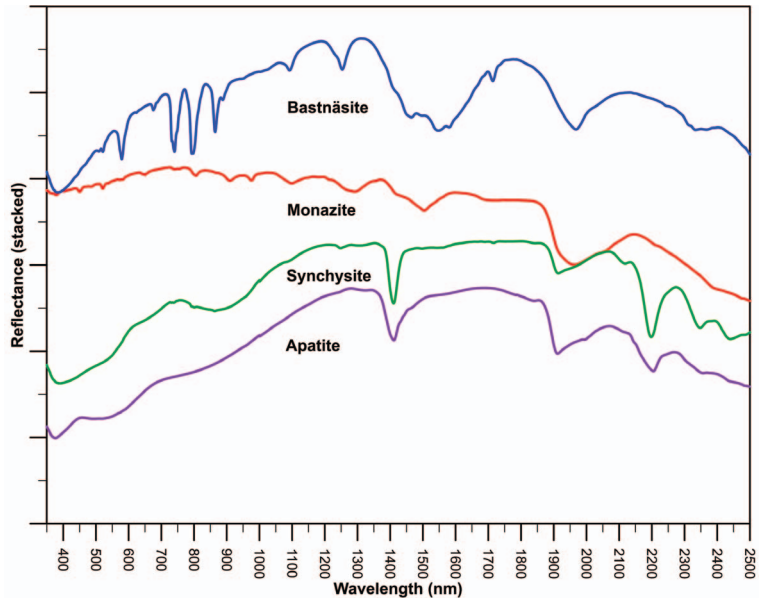

Figure 9. VNIR-SWIR reflectance spectra of *REE*-bearing minerals from the NMC: bastnäsite (NMC017457 Ambositra, Madagascar), monazite (NMC065734 Kerala State, India), synchysite (NMC017436 White Cloud pegmatite, Platte Canyon, Colorado, USA), and apatite (NMC061333 Tregonnec, Finistère, Bretagne, France).

To determine whether the spectral patterns of these uncommon minerals are valid, comparing the spectra studied with published reflection spectra or to know the composition of the mineral by independent methods such as XRD, EPMA, or geochemistry is necessary. Minerals in national collections generally have been analyzed by XRD and/or EPMA but the data tend to be buried in local databases or laboratories.

SUMMARY

Progress over the last few decades in field instrumentation has facilitated acquisition of *in situ*, non-destructive, rapid, mineralogical and mineral-chemical analyses using near-infrared spectroscopy. In some environments, these key infrared-active alteration minerals can provide robust vectors to ore (Table 1). For some

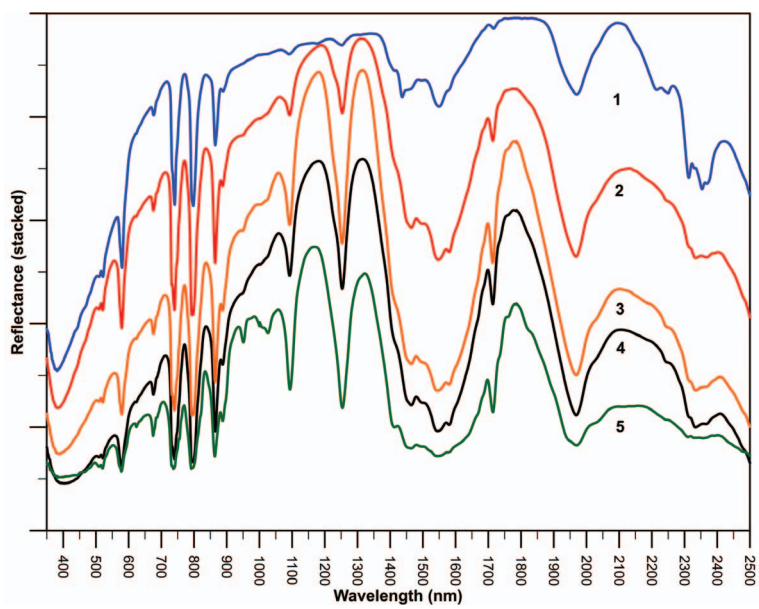

Figure 10. Stacked VNIR-SWIR reflectance spectra of bastnäsite samples from the NMC: (1) NMC65959 Mountain Pass, San Bernardino County, California, USA; (2) NMC17457 Ambositra, Madagascar; (3) NMC17458 Diandakatany, Madagascar; (4) NMC17455 St. Peter's Dome, El Paso County, Colorado, USA; (5) NMC17454 St. Peter's Dome, El Paso County, Colorado, USA. Composition of (4) and (5) verified by XRD.

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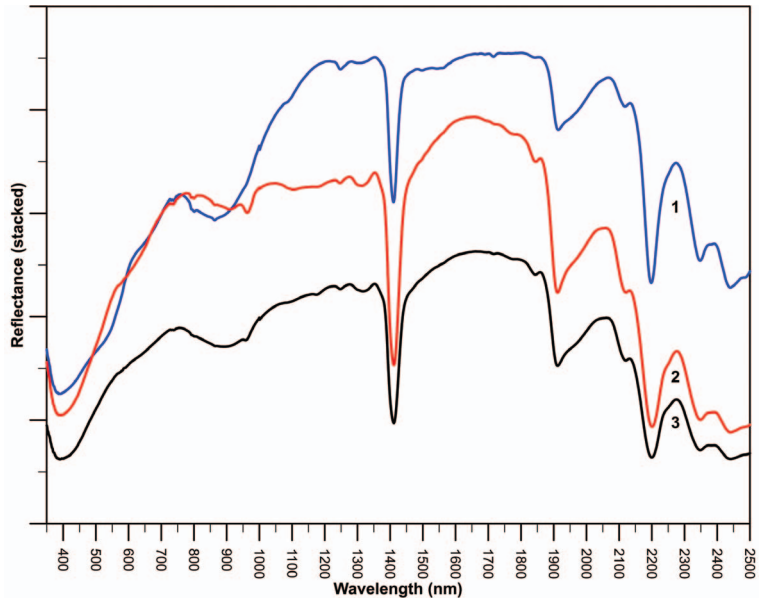

Figure 11. VNIR-SWIR reflectance spectra of synchysite from: (1) NMC17436 White Cloud pegmatite, Platte Canyon, Colorado, USA; (2) NMC17433, variety doverite, Organ Pipe Group, Jefferson County, Colorado, USA; (3) NMC12887 South Platte, Colorado, USA. Composition of (2) and (3) verified by XRD.

users, custom libraries are needed and this should be achieved through the collection of VNIR-SWIR spectra of pure reference minerals in a controlled manner. To encourage the expansion of these techniques, collection of VNIR-SWIR spectra on clay minerals of the Kodama Clay Collection, part of the vast national mineral collection at the Geological Survey of Canada, has been completed (Percival *et al.*, 2016a). As shown in that 2016a study, infrared spectroscopy can also identify *REE*-U-Th-Nb-bearing minerals relevant for exploration for critical metals and work is underway to expand the GSC infrared spectral database to include these minerals. These large mineral collections represent a valuable asset for geoscientists. As national collections become

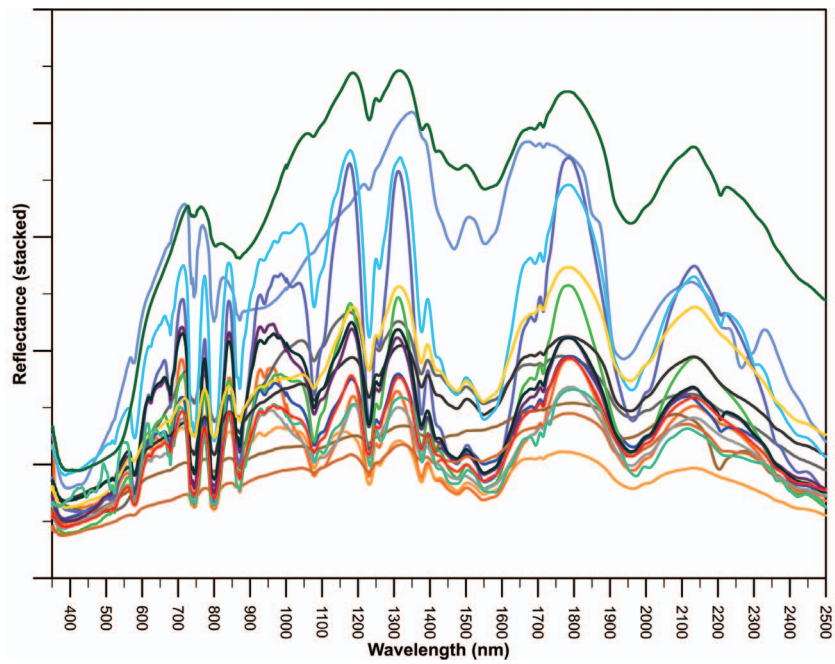

Figure 12. VNIR-SWIR reflectance spectra of 18 of the 48 monazite samples from the NMC.

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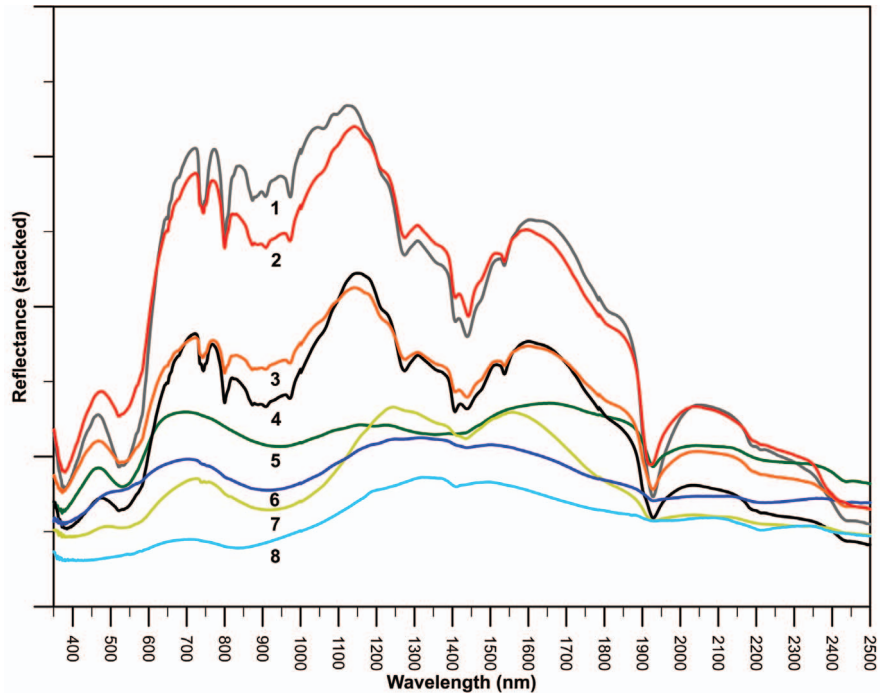

Figure 13. Stacked VNIR-SWIR reflectance spectra of eudialyte from: (1) NMC063870 Kippawa Complex, Temiskamingue County, Quebec; (2) NMC012085 Sheffield Lake, Kipawa River, Temiscamingue County, Quebec; (3) NMC012854 Sheffield Lake, Kipawa River, Temiscamingue County, Quebec: (4) NMC012088, Sheffield Lake, Kipawa River, Temiscamingue County, Quebec. (5) NMC061854 Lovozero Massif, Kola Peninsula, USSR; (6) NMC064961 Mont St. Hilaire, Quebec; (7) NMC067665 Kangerdluarssuk, Ilimaussaq, Greenland; (8) NMC01209 Langesdundsfiord, Norway. Composition of number 8 verified by XRD.

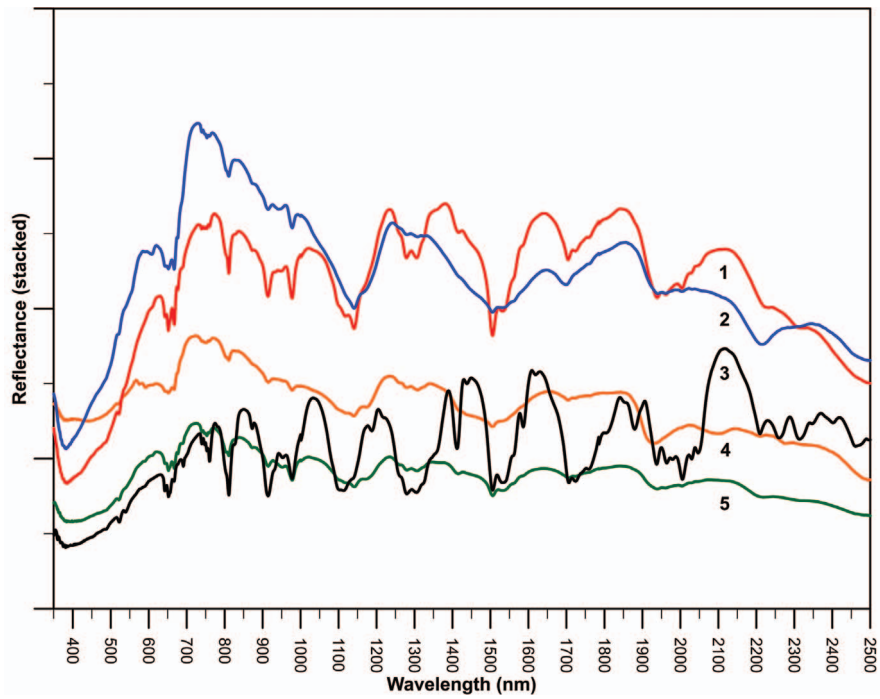

Figure 14. Stacked VNIR-SWIR reflectance spectra of xenotime from: (1) NMC018949 (X-rayed) Tvedestrand, Norway; (2) NMC067303 Gunter Mine, Maynooth area, Ontario; (3) NMC067514 Novo Horizonte, Bahia, Brazil; (4) NMC018946 Southern Pacific Quarry, Nuevo, Riverside County, California, USA (xenotime crystal has garnet inclusion); (5) NMC018947 Iveland, Norway.

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more recognized for their value in Earth sciences research, these data must be available publicly. Building spectral libraries of unique and common minerals ensures the legacy of collections and adds to their scientific and cultural value.

ACKNOWLEDGMENTS

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