Broadband Measurements of the Complex Permittivity of Carbonaceous Asteroid Regolith Analog Materials

A. L. Boivin1, D. Hickson2, C. Tsai3, A. Cunje1, R. R. Ghent1,4, and M. G. Daly2

1 Solar System Exploration Group, Department of Earth Sciences, University of Toronto, Toronto, Ontario, Canada, 2 Centre for Research in Earth and Space Science, York University, Toronto, Ontario, Canada, 3 Department of Physics, University of Toronto, Toronto, Ontario, Canada, 4 Planetary Science Institute, Tucson, AZ, USA

Abstract We develop a full methodology to measure the complex relative permittivity of planetary regolith analog materials in vacuum and up to 120 °C over a broad range of frequencies (400 MHz to 8.5 GHz or wavelengths 18.8–3.5 cm). We demonstrate our method with measurements of analog regolith materials appropriate for asteroid (101955) Bennu, the target of National Aeronautics and Space Administration’s OSIRIS-REx mission: individual and mixed components of UCF/DSI-CI-2, a new carbonaceous asteroid regolith simulant produced by Deep Space Industries based on CI chondrite meteorite mineralogy. We measure, for the first time, the effect of carbonaceous material on the complex relative permittivity of asteroid regolith analogs by measuring the powdered serpentine component of the simulant mixed with varying amounts of carbonaceous material in vacuum at 25 and 40 °C. We find that at a bulk density of 1.60 g/cm³ and wavelength of 12.6 cm, serpentine with 5 wt% carbonaceous material has $\varepsilon_r = 3.30 \pm 0.01$ and $\tan\delta = 0.016 \pm 0.003$ and that carbonaceous material increases the attenuation of electromagnetic energy in our samples. Ground-based radar (at 12.6- and 3.5-cm wavelengths) has previously been used to investigate carbonaceous asteroid (101955) Bennu. Our measurements provide new constraints on the attenuation of radar energy in granular carbonaceous materials.

Plain Language Summary We measure the electrical parameters that are responsible for absorbing and reflecting radar waves in powders that have been mixed to resemble the composition of the regolith of the asteroid Bennu. Regolith is powdered rock material that covers the surfaces of most rocky planetary bodies. Bennu is the target of National Aeronautics and Space Administration’s OSIRIS-REx mission, which is a spacecraft that will return regolith from the surface of Bennu to Earth. Radar has been used from Earth to look at Bennu, so it is important to understand how the electrical parameters of the regolith on Bennu affect radar signals. Bennu’s regolith is thought to contain organic carbon. We have measured the effect of adding organic carbon to our samples and found that adding carbon increases absorption of radar waves.

1. Introduction

Asteroids make up a large and highly varied population of bodies in our solar system. Asteroids as well as comets contain, encoded within their physical, chemical, and orbital properties, information related to the formation of the planets and the conditions of the early solar system. Understanding the composition and physical properties of these bodies is therefore key to understanding the origin and evolution of the solar system. National Aeronautics and Space Administration’s (NASA) OSIRIS-REx mission, which launched in September 2016, aims to increase our knowledge and understanding of these properties for the asteroid (101955) Bennu, a B-type asteroid (SMASII spectral taxonomic classification). B-type asteroids are thought to be some of the most primitive carbonaceous asteroids with compositions relatively close to the solar nebula (Lauretta et al., 2015). OSIRIS-REx plans to study Bennu in orbit using a suite of instruments including optical cameras, spectrometers, and a laser altimeter before acquiring a sample of regolith from the surface and sending it back to Earth (Lauretta et al., 2017).

In preparation for this mission, ground-based radar data at 12.6- and 3.5-cm wavelengths were used to help characterize Bennu as well as produce a shape model of the asteroid (Chesley et al., 2014; Nolan et al., 2013). Bennu is spheroidal, has a mean diameter of $492 \pm 20$ m, and appears smooth at scales down to 7.5 m
Radar data were also used to derive the near-surface bulk density of the asteroid, a key engineering factor for sample acquisition and return. Nolan et al. (2013) calculated either 1.65 or 0.9 g/cm³ for the near-surface bulk density, while Hickson et al. (2018) used the same data as well as laboratory permittivity measurements to derive a new estimate of 1.27 ± 0.33 g/cm³. The relationship between radar albedo and bulk density of the near-surface depends on the real part of the relative complex permittivity of the material (denoted as $\varepsilon'$), in this case regolith (Gavin et al., 1985; Magri et al., 2001).

Spectroscopic data suggest that Bennu most closely resembles hydrated carbonaceous chondrite CI- and CM-group meteorites and that it closely resembles asteroid (24) Themis. Spectroscopic analysis of Themis indicates the possible presence of frozen water as well as organic material, suggesting that Bennu may also contain similar compounds (Lauretta et al., 2015). Thermal IR data, radar data, and geophysical analysis of its shape, density, and rotation state all suggest that Bennu is very likely to have regolith (Lauretta et al., 2015). Thermal modeling indicates that current surface temperatures peak at around 340 K at the equator and 200 K at the poles. In the past, Bennu may have experienced surface temperatures higher than 500 K, which may be high enough to evolve volatiles or alter organics. Modeling indicates, however, that temperatures drop off rapidly with depth such that regolith 3 to 5 cm deep would have experienced temperatures approximately 100 K less than at the surface (Lauretta et al., 2015).

An extensive body of work exists on measurements of the relative permittivity of both terrestrial and lunar samples. The relative permittivity is a complex quantity such that $\varepsilon' = \varepsilon' - i\varepsilon''$. M. J. Campbell and Ulrichs (1969) and Ulaby et al. (1988) made measurements of terrestrial rocks and rock powders. Olhoeft and Strangway (1975) reviewed 92 measurements of Apollo lunar samples and fit the data using the Lichtenecker geometrical mean formula. They also established a relationship for tan $\delta$ (the loss tangent or $\varepsilon'' / \varepsilon'$) by relating it to the bulk density, $\rho_b$, and the weight percent of combined FeO + TiO$_2$. Carrier et al. (1991) did similar regressions using an updated data set and found $\varepsilon' = 1.919\rho_b$. Fa and Wieczorek (2012) re-fit available permittivity data measured at 450 MHz to functions that depend on bulk density, porosity, abundance of FeO and TiO$_2$, and the ferromagnetic resonance. They concluded that for lunar samples with fixed bulk density or porosity, $\varepsilon'$ is constant across all samples and independent of FeO, TiO$_2$, or the ferromagnetic resonance, whereas tan $\delta$ depends primarily on TiO$_2$. A comparison of permittivity measurements of Apollo samples and lunar regolith simulants can be found in Calla and Rathore (2012).

Stillman and Olhoeft (2008) conducted permittivity and magnetic permeability measurements of 12 analog Mars regolith samples between 1 MHz and 1 GHz at Mars temperatures (180 – 300 K). They concluded that, for Mars analog materials, electromagnetic properties are temperature dependent and that the magnetic permeability must be taken into account. They also noted that, in contrast to the Moon, water, especially in clays, may cause significant attenuation on Mars. Further review of Mars relevant measurements can be found in, for example, Stillman and Olhoeft (2008), Stillman et al. (2010), Stillman and Grimm (2011), Mattei et al. (2014), and references therein.

The permittivity of analog cometary material has also been investigated. For example, Brouet et al. (2014) made complex relative permittivity measurements of JSC Mars-1 Martian soil simulant and volcanic ash in three frequency bands (50 – 500 MHz, 2.45 – 12 GHz, and 190 GHz), and Brouet et al. (2015) made broadband $\varepsilon'$ measurements of JSC-1A lunar regolith simulant and volcanic ash from 0.05 to 6 GHz as well as complex relative permittivity measurements at 190 GHz. Heggy et al. (2012) measured the complex permittivity of four powdered meteorites from 5 to 100 MHz: three ordinary chondrites and one mesosiderite. Brouet et al. (2016) made 12 $\varepsilon'$ measurements of porous ice samples and 26 measurements of dust-ice mixtures at a range of porosities where the dust fraction was made up of JSC-1A lunar regolith simulant. See also Heggy et al. (2001), Herique et al. (2002), and references therein.

Measurements made in support of asteroid radar investigations are sparse; however, some meteorites have been measured. For example, as noted above, Heggy et al. (2012) made measurements of four powdered meteorites. Fensler et al. (1962) reported measurements of two solid ordinary chondrites (one L6 and one H5) as well as several tektites and rocks between 420 to 1,800 MHz, and M. J. Campbell and Ulrichs (1969) measured six solid meteorite samples (five ordinary chondrites and one enstatite chondite) at 450 MHz. Garvin et al. (1988) also measured six solid meteorite samples at 500 MHz, one of which was a carbonaceous chondrite (Murchison, CM2). They found $\varepsilon' = 4.7$ and tan $\delta = 0.11$ for Murchison at 500 MHz. A summary of permittivity measurements made in support of asteroid and cometary missions as well as the importance of radar in the study of asteroids can be found in Herique et al. (2018).
Although the real part of the relative complex permittivity ($\varepsilon'_r$) is known to depend primarily on bulk density and moisture and only weakly on mineralogy and other factors, the imaginary part ($\varepsilon''_r$), which is responsible for the attenuation of the signal, depends more strongly on mineralogy as well as other factors including moisture, temperature, and frequency. To further our ability to characterize the physical properties of the regoliths of asteroids like Bennu and other planetary bodies and to improve the quantitative utility of planetary radar data, it is necessary to further understand the effects of mineralogy, temperature, and frequency on the complex relative permittivity for granular materials. Despite extensive work, the literature lacks a systematic exploration of the influence of these parameters on the complex permittivity.

We develop a full methodology to measure the relative complex permittivity of planetary regolith analog materials over a broad range of frequencies (400 MHz to 8.5 GHz or wavelengths 18.8 – 3.5 cm) while systematically varying composition and temperature. Our method includes full tracking of measurement uncertainties. Measurements can be made either hot (up to 120 °C) or at room temperature. In order to remove moisture, we bake our samples prior to measurement, and we make hot as well as room temperature measurements in vacuum (≤126.7 Pa) to prevent readsorption of water.

We demonstrate our method by making measurements in vacuum of samples analogous to the regolith of Bennu. We present measurements of the serpentine phyllosilicate and kerogen substitute components of a new carbonaceous asteroid regolith simulant based on CI meteorite composition available from Deep Space Industries (UCF/DSI-CI-2, www.deepspaceindustries.com/simulants/). Kerogen-like organic compounds have been found in carbonaceous meteorites and are expected to be present in the regolith of asteroid (101955) Bennu (e.g., Delbo & Michel, 2011). Our simulant is provided in separate unmixed components, allowing us to make our own mixtures to systematically investigate the effects of increasing the weight fraction of the kerogen substitute component on the complex permittivity of the powdered serpentine component and, therefore, its effect on the attenuation in decibels per meter in Bennu-like regolith. To our knowledge, the measurements presented in this paper target for the first time the effect of carbonaceous material on the complex relative permittivity of asteroid regolith using suitable analogs based on the composition of CI chondrite meteorites.

2. Theory and Background

The total electromagnetic loss in a material is controlled by the electromagnetic properties of that material: permittivity ($\varepsilon$), permeability ($\mu$), and conductivity ($\sigma$). The permittivity and permeability are unitless complex numbers and are usually reported relative to the permittivity and permeability of free space such that

$$\varepsilon^* = \frac{\varepsilon}{\varepsilon_0} = \varepsilon'_i - i\varepsilon''_i,$$

$$\mu^* = \frac{\mu}{\mu_0} = \mu'_i - i\mu''_i,$$

where $\varepsilon'_i$ and $\varepsilon''_i$ are the relative and absolute complex permittivity, respectively; $\varepsilon_0$ is the permittivity of free space, $\varepsilon'_i$ and $\varepsilon''_i$ are the real and imaginary parts of the relative complex permittivity, respectively, and similarly for $\mu$. The loss tangent (tan $\delta$) controls the dissipation of electromagnetic energy in a material. The dielectric and magnetic loss tangents are defined as

$$\tan \delta_d = \frac{\varepsilon''_i}{\varepsilon'_i},$$

$$\tan \delta_m = \frac{\mu''_i}{\mu'_i},$$

where $\tan \delta_d$ is the dielectric loss tangent and $\tan \delta_m$ is the magnetic loss tangent. The total electrical loss tangent (tan $\delta_e$) includes the effect of conductivity and can be written as

$$\tan \delta_e = \frac{\varepsilon''_i + \frac{\sigma}{\omega\varepsilon_0}}{\varepsilon'_i},$$

where $\omega$ is the angular frequency. The combined electromagnetic loss tangent then becomes (Stillman & Olhoeft, 2008)

$$\tan \delta_{em} = \tan \left( \frac{\delta_d + \delta_m}{2} \right) = \frac{\sigma}{\mu_i}.$$
where $\alpha$ is the attenuation parameter and $\beta$ is the phase parameter of the material (e.g., Ulaby & Long, 2014). For a detailed explanation of each loss tangent including phase angle relationships, we point the reader to Stillman (2006). The maximum penetration depth of a material is related to $\alpha$ and is defined as (Ulaby & Long, 2014)

$$\delta_p = \frac{1}{2\alpha}. \tag{7}$$

Equation (7) is the maximum (theoretical) penetration depth assuming no scattering. The true (measurable) penetration depth will depend on the parameters of the radar equation including transmitted power, receiving and transmitting antenna gain, distance to target, and radar cross section. Measurements presented in this paper are limited to nonmagnetic materials such that $\mu_r = 1$.

2.1. Complex Permittivity Measurements Using Transmission/Reflection Methods

The complex permittivity of a sample can be determined using a coaxial transmission line and a two-port vector network analyzer (VNA). Each end of the transmission line is connected to one port on the VNA, and the scattering parameters (S-parameters) are measured. This yields four complex S-parameters, which are denoted as $S_{xy}$ such that $x$ is the port at which the signal is measured and $y$ is the port at which the signal originates. The resulting S-parameters are related to the reflection and transmission coefficients of the material in the transmission line such that

$$S_{11} = S_{22} = \frac{\Gamma(1 - T^2)}{1 - T^2}, \tag{8}$$

$$S_{21} = S_{12} = \frac{T(1 - \Gamma^2)}{1 - T^2}. \tag{9}$$

where $\Gamma$ and $T$ are the reflection and transmission coefficients, respectively, and

$$\Gamma = \frac{\gamma_0 \mu_r^* - \gamma}{\gamma_0 \mu_r^* + \gamma}, \tag{10}$$

$$T = \exp(-\gamma d), \tag{11}$$

where $\gamma_0$ and $\gamma$ are the propagation constants in the empty and filled line, respectively, $\mu_r^*$ is the complex magnetic permeability ($\mu_r^* = 1$ for nonmagnetic materials), and $d$ is the length of the sample (= to the length of the transmission line) and

$$\gamma_0 = \frac{2\pi}{\lambda_0} \sqrt{1 - \left(\frac{\lambda_0}{\lambda_c}\right)^2}, \tag{12}$$

$$\gamma = \frac{2\pi}{\lambda_0} \sqrt{\varepsilon_r \mu_r^* - \left(\frac{\lambda_0}{\lambda_c}\right)^2}, \tag{13}$$

where $\lambda_0$ is the free-space wavelength, $\lambda_c$ is the cutoff wavelength of the waveguide ($\lambda_c \to \infty$ for a coaxial waveguide), and $\varepsilon_r^*$ is the complex permittivity of the sample (e.g., Baker-Jarvis et al., 1993; Boughriet et al., 1997; Nicolson & Ross, 1970; Weir, 1974).

In practice, when samples placed in the transmission line are not homogeneous as may be the case with powdered samples, $S_{11} \neq S_{22}$ and $S_{21} \neq S_{12}$. This yields a difference between $\varepsilon_r^*[S_{11}, S_{21}]$ and $\varepsilon_r^*[S_{22}, S_{12}]$.

3. Methods

To make measurements of the permittivity of powdered materials, we use a modified and expanded version of the workflow established by Sotodeh (2014). We measure the S-parameters of the sample using a two-port network analyzer, and these parameters are then used to compute the permittivity.

We make S-parameter measurements using a 14-mm-diameter coaxial transmission line connected to an Agilent (now Keysight) two-port E5071C ENA VNA (300 KHz to 12 GHz) using Maury Microwave SC-35 cables with GR900-QMMJ 3.5- to 14-mm adapters. These cables have high stability with flexure and a relatively long flex life (deterioration over time due to flexure), making them ideal for these measurements. Nevertheless, deterioration over time can be monitored by making periodic measurements of a reflection calibration standard (short or open). We use the GR900-LZ15 15-cm-long 50-Ω reference transmission line manufactured by General Radio. We chose this line due to the very high repeatability of the connections made with the
Figure 1. Experimental setup used for our measurements. (a) Two-port network analyzer connected to sample holder (coaxial transmission line) within vacuum chamber. (b) Sample holder with heating tape connected to temperature controller inside vacuum chamber. (c) Custom packing funnel mounted on sieve shaker with sample holder attached. Funnel system uses a centering rod to hold the center conductor of the coaxial transmission line in place during packing. (d) Packed sample holder with custom Teflon® washer inserted to contain powdered sample.

General Radio 14-mm connectors, the fact that it is easier to pack powdered samples into a wider diameter line (compared to the available 7-mm-diameter line) and to minimize edge effects at the boundary between sample and transmission line wall (Hickson et al., 2017; Sotodeh, 2014). The length of the line is a compromise between measurement accuracy (especially for $\epsilon''_r$) and sample size (Sotodeh, 2014). The dimensions of this transmission line make it suitable for measurements between DC (direct current, 0 Hz) and 8.5 GHz, given a capable measurement system. It should be noted that scratches may form on the inner surface of the transmission line due to repeated use. This may cause deterioration in data quality at high frequency. Since the inner surface of the GR900-LZ15 is coated with a thin layer of silver, scratches may also reduce the conductivity of the surface and therefore increase the skin depth into the walls of the transmission line, affecting low-frequency measurements. The state of deterioration can be monitored over time by making periodic measurements of the empty line.

We pack our samples into the transmission line using a custom-made funnel, which holds the center conductor in place. To ensure even packing, we mount the funnel and attached transmission line on a sieve shaker (Gilson Performer III SS-3), which we keep active during the packing process. In order to prevent the sample from spilling out of the transmission line, we cap both ends of the line with 1.7-mm-thick Teflon® washers.
Figure 2. Example S-parameter data of powdered serpentine where the complex S-parameters have been split into their magnitude and phase components. The settings used in this paper to make S-parameter measurements on the Agilent E5071C ENA are intermediate frequency bandwidth (IFBW) = 10 Hz, averaging factor = 5, power = $-10$ dB/m, and number of points = 601. Note that a low IFBW and a high averaging factor can both increase the dynamic range (lower the noise floor) of the instrument at the cost of measurement time. We use a low IFBW with a small averaging factor as a compromise between dynamic range and measurement time. The phase accuracy could be further improved introducing an electrical delay in the vector network analyzer to compensate for the electrical length of the transmission line. S-parameter = scattering parameters.

Figure 1 shows our measurement and sample packing setup, and Figure 2 shows an example S-parameter measurement made with this setup. To calculate the bulk density of the sample, we weigh the transmission line before and after packing using a Sartorius MSA524P-100-DI Cubis Precision Balance, which provides an accuracy of ±0.0005 g. Since static electricity can often be an issue when dealing with powdered samples, the balance is also equipped with an ionizer, which we use before and during packing to neutralize static electricity.

3.1. Instrument Calibration and Uncertainties

We calibrate the VNA and make S-parameter measurements using the METAS VNA Tools II software (Wollensack et al., 2012). We use the METAS software to control the VNA instead of the default software. It has several advantages including advanced calibration techniques, full tracking of Type B uncertainties (VNA noise floor, VNA linearity, VNA calibration drift, cable stability, and connector repeatability), and a full journal of the calibration and measurement, thus ensuring a repeatable and traceable measurement process (Wollensack et al., 2012). We follow the ISO Guide to the Expression of Uncertainty in Measurement and define Type A uncertainty as uncertainty evaluated by the statistical analysis of series of observations and Type B as uncertainty evaluated by means other than the statistical analysis of series of observations (ISO/IEC GUIDE 98-3, 2008). By measuring the VNA noise floor, drift, and linearity and by keeping track of all connections and every time a cable is moved, VNA Tools II can propagate the Type B uncertainty in the S-parameters throughout the calibration and measurement process. This is useful both to provide insight into sources of uncertainty during measurement and to propagate uncertainty throughout the permittivity calculation.
Figure 3. Real part of the relative permittivity (a) and loss tangent (b) measurements of a Rexolite® cylinder machined to fit into the transmission line showing the different contributions of all sources of uncertainty in $\varepsilon_r'$ and $\tan \delta$. Measurements have been corrected for the air gap between the sample and the transmission line. Sources include the estimated Type A uncertainty in our measurements and the sources of Type B uncertainty (in both magnitude and phase): connector repeatability, VNA noise, VNA linearity, VNA drift, and cable stability. A significant proportion of the Type B uncertainty at low frequencies is due to VNA phase uncertainty. These uncertainties and their magnitudes are typical for all our measurements. The negative loss tangent shown at low frequency is not physical but is instead an artifact of the algorithm used to calculate the complex permittivity when the material is very low loss (such as Rexolite®) and the high uncertainties at these frequencies. Note the log scale on the frequency axis. VNA = vector network analyzer.

In order to estimate the Type A uncertainty of our measurements, we measured the same sample of Rexolite®, machined into a cylinder to fit into the transmission line, 10 times on different occasions, and calculated the standard deviation of the results over frequency. We found the maximum and the mean standard deviation in $\varepsilon_r'$ to be 0.008 and 0.003, respectively. For $\tan \delta$, we identified two uncertainty regions: the low-frequency region (<100 MHz) and the high-frequency region (≥100 MHz). In the low-frequency region, we found the maximum and mean standard deviation to be 0.009 and 0.003, respectively, while in the high-frequency region, they are 0.002 and 0.0002, respectively. We use the maximum standard deviation ($\sigma_{\text{max}}$) in $\varepsilon_r'$ and $\tan \delta$ in our Type A uncertainty calculations.

Due to the possibility of inhomogeneous packing of powdered samples, we also compute, for every measurement, the difference between $\varepsilon_r'[S_{11}, S_{21}]$ and $\varepsilon_r'[S_{22}, S_{12}]$ as well as $\tan \delta[S_{11}, S_{21}]$ and $\tan \delta[S_{22}, S_{12}]$ at each frequency point, similarly to Brouet et al. (2016). We estimate the Type A uncertainty at each frequency.
point as

\[
\text{Type A uncertainty for } \varepsilon_r' = \text{Max}(\sigma_{\varepsilon_r'}^{\text{max}}, |\varepsilon_r'[S_{11}, S_{21}] - \varepsilon_r'[S_{22}, S_{12}]|).
\]

Type A uncertainty for \( \tan \delta = \text{Max}(\sigma_{\tan \delta}^{\text{max}}, |\tan \delta[S_{11}, S_{21}] - \tan \delta[S_{22}, S_{12}]|).
\]

(14)

(15)

The total uncertainty at each frequency point is determined by adding the variances of the Type A and Type B uncertainties:

\[
\text{Total uncertainty} = \sqrt{\text{(Type A uncertainty)}^2 + (\text{Type B uncertainty})^2}.
\]

(16)

In addition to determining the uncertainties at each frequency point, equation (16) defines the lowest possible values measurable by our instrument at each frequency point for each measurement. This is important for very low loss materials.

In order to assess the accuracy of our measurement method, we measure a cylinder of cross-linked polystyrene (Rexolite® 1422, www.rexolite.com/specifications/) machined to fit into our transmission line. Rexolite® has a very stable permittivity across a wide rage of frequencies with manufacturer-provided values of \( \varepsilon_r' = 2.53 \) from 1 MHz to 500 GHz and \( \tan \delta = 0.0001 \) at 10 MHz and 0.0003 at 10 GHz. Since our sample (outer diameter = 14.22 mm and inner diameter = 6.24 mm) is slightly smaller than the exact volume of the transmission line (outer conductor diameter = 14.288 mm and inner conductor diameter = 6.204 mm), an air gap correction must be performed to get the sample parameters. We perform an air gap correction postmeasurement according to the method of Baker-Jarvis et al. (1993). Figure 3 shows a Rexolite® measurement showing the contribution of all sources of uncertainty where the Type B uncertainties include both magnitude and phase uncertainties. The uncertainties shown in Figure 3 are typical of all our measurements. In this paper, we present data from 400 MHz and above because it is appropriate for Earth-based radar. At 400 MHz, we measure \( \varepsilon_r' = 2.53 \pm 0.06 \) and \( \tan \delta = 0.001 \pm 0.03 \), and at 8.5 GHz, we measure \( \varepsilon_r' = 2.53 \pm 0.01 \) and \( \tan \delta = 0.0007 \pm 0.002 \). Our measurements are generally in very good agreement with the manufacturer-provided values for the real part of the permittivity of Rexolite® 1422. Although the loss tangent of Rexolite® is too low to be precisely measured using this setup, the manufacturer-provided values for \( \tan \delta \) do fall within the uncertainty of our measurements.

In order to measure the S-parameters of powdered samples capped by Teflon® washers, the measurement plane of the VNA must be moved from the VNA port to the sample-washer interface. We achieve this using both a VNA calibration and a postmeasurement connector deembedding step. We calibrate using the Thru-Reflect-Match method, a highly accurate broadband VNA calibration regarded as more accurate than the popular Short-Open-Load-Through calibration method (Pulido-Gaytán et al., 2015). This moves the measurement plane to the connector-washer interface. To move the interface to the sample, and since the Teflon® washers used to cap the transmission line have a slight impedance mismatch with the line, a deembedding step must be done to remove their influence and get the true sample S-parameters (e.g., Keysight Technologies, 2014). We achieve this by modeling the S-parameters of a 1.7-mm-thick Teflon® washer using equations (8)–(13).

As shown in Hickson et al. (2017), boundary effects in the sample holder when measuring powdered samples can significantly affect the measured permittivity, especially for high permittivity materials and when the grain size is large. Hickson et al. (2017) propose a model to correct for this boundary effect and to solve for the solid permittivity of the material being measured, given the exact dimensions of the transmission line, the measured permittivity of the powdered sample, the average particle diameter, and the bulk sample porosity. This same model can be applied to correct the measured permittivity of the powdered sample for the boundary effect, if the solid permittivity, the average particle diameter, and the bulk sample porosity are known. This effect should be taken into consideration if very highly accurate measurements are required; however, this correction has not yet been tested on the imaginary part of the permittivity. For this reason, and since samples measured in this paper have small grain sizes and do not have high permittivity values, we ignore these boundary effects.

3.2. Dry Measurements

When measuring samples analogous to dry environments such as the Moon or an asteroid, samples should be completely devoid of adsorbed water as even small amounts of surface water will affect the permittivity (B. A. Campbell, 2002; Ulaby & Long, 2014). For measurements of planetary analog materials, care should
Measurements of the real part of the complex relative permittivity of a sample of powdered serpentine. The sample was baked and then allowed to sit under ambient conditions for approximately 45 min, while the vector network analyzer was calibrated before being put in vacuum. Measurements were taken before the vacuum was turned on (Baked Serpentine in the figure legend) and then once every hour for 4 hr.

To quantify the loss of volatiles, we weigh samples before and after being baked. We then weigh the transmission line, fill it with the sample, weigh it again, and place it in a vacuum chamber. We then evacuate the chamber and leave the sample in vacuum for 2 hr prior to measuring to remove any moisture that would have been readsorbed onto the sample from the atmosphere during calibration. To ensure vacuuming successfully removes this extra moisture, we have weighed the transmission line postpacking, left it on the balance for approximately 30 min such that the weight of the sample could be seen to gradually increase, left the sample in the evacuated chamber for 2 hr, and observed upon reweighing the transmission line that the sample weight had returned to its original postpacking weight. This has also been confirmed by measuring over time in the vacuum chamber and finding that measurements leveled off after 2 hr in the chamber (Figure 4).

3.3. Hot Measurements

The upper regolith on planetary surfaces can experience a wide range of temperatures over both orbital and rotation periods. The peak surface temperature for asteroid (101955) Bennu at perihelion in its current orbit, for example, has been estimated as 117 °C, and temperatures could have been as high as 227 °C in the recent past (Lauretta et al., 2015). Since dielectric properties can vary with temperature (e.g., Stillman & Grimm, 2011; Stillman & Olhoeft, 2008), it is necessary to conduct laboratory measurements of regolith analog materials at elevated temperatures. It should be noted that temperature cycling and other space weathering effects can affect the properties of regolith grains on the surface of an airless body such as Bennu. Additional laboratory experiments would be needed to fully understand these effects on the permittivity of asteroid regolith.

To increase the sample temperature when packed in the GR900-LZ15 coaxial transmission line (made of brass), we have attached Kapton flexible heating tape to the outer surface of the transmission line, capable of heating to 120 °C. Opposite to this, an Omega SA1-RTD-4W surface mount resistance temperature detector (RTD) is attached. Both the heating tape and RTD are connected to an Omega CNI8 series temperature controller, which used proportional-integral-derivative control to maintain the transmission line at an operator-specified temperature. Once the RTD measures a constant temperature equal to the target temperature with no fluctuations, we leave the sample for 2 hr at the target temperature to ensure that the transmission line and sample have reached equilibrium at that temperature before making an S-parameter measurement. The Maury Microwave SC-35 cables used to connect to the transmission line have an operating temperature range of −55 to 125 °C and are therefore suitable for these measurements. Based on the thermal properties of brass, the volume expansion of the transmission line due to heating is estimated to be below 1%. To verify that thermal expansion does not cause significant error in our measurements, we measured the empty transmission line in vacuum at eight temperatures, ranging from ambient temperature (23 °C) to 90 °C (Figure 5), and be taken as to the nature of the target environment. Water contained in soils can be considered either bound water or free water. Bound water molecules are defined as having a force acting upon them, which inhibits their response to a suddenly applied electric field, while free-water molecules are free to respond (Ulaby & Long, 2014).

Baking samples in an oven at 250 °C for 48 hr prior to packing the transmission line can remove moisture. In cases where bound water may be desired, or for materials where high temperatures may damage or alter the sample, a lower temperature of 115 °C can be used. The amount of time a sample should be left in the oven can be determined by progressively weighing the sample as it is baked and observing when the weight ceases to change. We found that the weight of most of our samples leveled off after about 36 hr in the oven. Highly temperature-sensitive samples, which may degrade or be altered at moderate temperatures, or samples meant to be representative of primitive materials, which may have never been exposed to high temperatures in their lifetime, can be baked in vacuum. Using the same method of progressive weighing, we have found that baking in vacuum at ≤126.7 Pa (≤950 mTorr) and 40 °C for 48 hr successfully removes moisture from our samples. At this pressure, water above 0.01 °C only exists in the vapor phase (Lemmon et al., 2017). All of the powdered sample measurement results presented below have been baked in vacuum.
calculated the maximum standard deviation in \( \varepsilon'_r \) across frequency to be 0.005, which is less than our Type A uncertainty.

### 3.4. Relative Permittivity Calculation

The permittivity and magnetic permeability of a sample can be analytically calculated using the Nicolson-Ross-Weir algorithm by relating the two-port VNA S-parameters to the reflection and transmission coefficients of the material (Nicolson & Ross, 1970; Weir, 1974). This algorithm, however, suffers from instabilities at half-wavelength intervals in low-loss materials. In order to address these instabilities, Baker-Jarvis et al. (1990) developed an iterative technique that assumes \( \mu_r = 1 \). Boughriet et al. (1997) then developed a noniterative method also assuming \( \mu_r = 1 \). We use this last technique to calculate the permittivity of nonferromagnetic and nonferriimagnetic samples. Magnetic materials, such as magnetite and titanohematites, significantly affect the total electromagnetic loss and cannot be measured with this method. We compute \( \varepsilon'_r, \varepsilon''_r, \) and \( \tan \delta \) from both sets of S-parameters ([\( S_{11}, S_{21} \)] and [\( S_{22}, S_{12} \)]). In order to assess the homogeneity of the sample packing in the transmission line, we then compute the percentage difference at each frequency point for each parameter from both sets of S-parameters as well the maximum and median differences. We calculate the final results by averaging the results from both set of S-parameters (e.g., \( \varepsilon'_r = (\varepsilon'_r[S_{11}, S_{21}]+\varepsilon'_r[S_{22}, S_{12}])/2 \)). We propagate the uncertainties through the permittivity calculation and the S-parameter deembedding step described above is performed before we calculate the permittivities. We have made the Python code used to calculate the complex permittivity of our samples publicly available and open source (Boivin & Hickson, 2018). The code includes propagation of uncertainties as long as they are present in the S-parameter data.

#### 3.4.1. Comparing Multiple Measurements

Due to the fact that bulk density is the single most important factor in the real part of the permittivity, the only way to compare measurements of different samples to one another is to normalize them to a single bulk density (e.g., Stillman & Olhoeft, 2008). This requires the assumption of a dielectric mixing equation. In the past, the Lichtenecker mixing formula (\( \varepsilon'_r = \rho_b^a \) where \( \rho_b \) is the bulk density and \( a \) is a constant) with \( a = 1.93 \pm 0.17 \) has been used, where the value of \( a \) was found via regression analysis of Apollo lunar samples (Olhoeft & Strangway, 1975). Carrier et al. (1991) applied the Lichtenecker mixing formula to an updated list of lunar sample permittivity measurements and found \( a = 1.919 \pm 1.92 \) to be the best fit to all the data. This formula can be extended to the complex permittivity and used to correct measurements to a constant bulk density (e.g., Olhoeft, 1976; Stillman, 2006) such that

\[
\varepsilon_{r,m} = \varepsilon_{r,m}(1.92)^{\rho_b-n-\rho_b,m},
\]  

#### Table 1

Maximum (Max) and Median (Mdn) Differences in the Real Part of the Relative Permittivity

| Sample              | Max| Mdn| Max| Mdn |
|---------------------|----|----|----|----|
| Kerogen 25 °C       | 3.4| 0.21| 1.1| 0.14|
| Kerogen 40 °C       | 3.4| 0.22| 1.1| 0.14|
| Serpentine 25 °C    | 4.0| 0.30| 0.74| 0.10|
| Serpentine 40 °C    | 3.8| 0.36| 0.78| 0.10|
| 2.5 wt% kerogen 25 °C | 2.1| 0.31| 0.74| 0.10|
| 2.5 wt% kerogen 40 °C | 1.9| 0.34| 0.78| 0.10|
| 5.0 wt% kerogen 25 °C | 3.1| 0.43| 1.2| 0.09|
| 5.0 wt% kerogen 40 °C | 3.1| 0.41| 0.74| 0.08|
| 7.5 wt% kerogen 25 °C | 3.0| 0.25| 0.74| 0.09|
| 7.5 wt% kerogen 40 °C | 2.8| 0.25| 2.2| 0.07|

#### Figure 5

Measurements of the empty transmission line in vacuum at different temperatures. A very slight increase in \( \varepsilon'_r \) can be seen with increasing temperature attributed to error due to thermal expansion of the brass transmission line. Note the log scale on the frequency axis.
Figure 6. (a) Real part of the relative permittivity versus frequency of the UCF/DSI-CI-2 serpentine component mixed with 5.0 wt% of the UCF/DSI-CI-2 kerogen substitute measured at 25 °C from both sets of scattering parameters \([S_{11}, S_{21}], [S_{22}, S_{12}]\) as well as their average. (b) Loss tangent versus frequency of the same measurements. All data are normalized to a bulk density of 1.60 g/cm³. Error bars are plotted every 25 data points. Note the log scale on the frequency axis of both plots.

where \(\varepsilon_{r,n}\) is the normalized complex permittivity, \(\varepsilon_{r,m}\) is the measured complex permittivity, \(\rho_{b,n}\) is the bulk density we are normalizing to, and \(\rho_{b,m}\) is the bulk density of the measured sample. We normalize our data to 1.60 g/cm³, which is the same bulk density used in Stillman and Olhoeft (2008).

4. Application of the Dry Method to Analogs of Carbonaceous Asteroids Such as (101955) Bennu

4.1. Analog Sample Composition

The closest spectral analogs to Bennu are CI and CM chondrite meteorites, particularly the CIs (Lauretta et al., 2015). Spectral investigations of CI chondrites reveal that CIs are primarily composed of intergrown saponite (a clay mineral) and serpentine matrix. Depending on the sample, around 10 wt% magnetite and 5 wt% total carbon are present in CI chondrites (Cloutis et al., 2011). The majority of the organic carbon content in carbonaceous meteorites is known to be macromolecular material with a structure similar to kerogen, and similar material is expected to be present on Bennu (Delbo & Michel, 2011). Although the actual composition of the surface of Bennu will remain unknown until the conclusion of the OSIRIS-REx mission, we begin our systematic investigation of the effect of mineralogy on the permittivity of B-type asteroid regolith by focusing on the effect of kerogen in a serpentine matrix without the presence of magnetite. In addition to influencing the relative permittivity, magnetite is known to exhibit magnetic relaxations, which would violate the assumptions made in section 3.4 (Stillman & Olhoeft, 2008). The effect of carbonaceous content on the permittivity of asteroid analog materials has, to our knowledge, never been investigated. In order to be able to safely handle our powdered samples, we have chosen to use components of the UCF/DSI-CI-2 asteroid regolith simulant from Deep Space Industries (www.deepspaceindustries.com/simulants/): We use the phyllosilicate serpentine...
Figure 7. (a) Real part of the relative permittivity versus frequency of the UCF/DSI-CI-2 kerogen substitute, the UCF/DSI-CI-2 serpentine component, and kerogen substitute-serpentine mixtures at 25 °C. (b) Loss tangent versus frequency of the same samples. All data are normalized to a bulk density of 1.60 g/cm³. Error bars are plotted every 25 data points. Note the log scale on the frequency axis of both plots.

component of UCF/DSI-CI-2, which is not asbestos forming, and the kerogen substitute component, which is not carcinogenic. To isolate the effect of the kerogen substitute, we measure the kerogen substitute component mixed into a serpentine matrix at 2.5, 5.0, and 7.5 wt% kerogen substitute as well as pure kerogen substitute and pure serpentine and compare the results.

4.2. Sample Preparation
Since Bennu is not thought to have been exposed to very high surface temperatures during its orbital evolution (Lauretta et al., 2015), the effects of temperature on the kerogen substitute component and its moisture content should be taken into account during sample preparation. To do this, we consider the work of Karsner and Perlmutter (1982) who measured the evolution of gases during drying of seven types of coal at temperatures ranging from 25 to 300 °C. They found that most of the water released was through evaporation occurring at 100 °C and that CO₂ begins to be released above 150 °C, while CO is released above 250 °C.

Pickles et al. (2014) measured the complex relative permittivity of subbituminous coal at two different grain sizes as a function of temperature and frequency with a temperature range of 25 to 650 °C from 912 MHz to 2.466 GHz. They divided their permittivity results into four temperature regions. Region 1, from room temperature to 100 °C, sees a slight increase in both real and imaginary relative permittivity as water in the coal sample is raised to a higher-energy rotational state. In Region 2, from 100 to 250 °C, both the real and imaginary relative permittivity decrease with the evaporation of free water and loosely bound water as well as a decrease in bulk density. Region 3 (250 to 450 °C) sees a further slight decrease in relative permittivity attributed to further evolution of volatile content and further slight decrease in bulk density, and Region 4 (450 to 650 °C) sees a significant increase in relative permittivity as the coal undergoes significant structural and chemical changes. Since some parts of the surface of Bennu may only have reached temperatures as high as approximately 230 °C in the recent past, since other areas may not have been subjected to such high temperatures,
and since temperatures on Bennu are expected to decrease rapidly with depth (Lauretta et al., 2015), we wish to remain within Region 2 or lower while still eliminating free water from our kerogen substitute sample. To achieve this, we bake our samples in vacuum at 40 °C as described in section 3.2.

4.3. Measurement Procedure

First, we weigh the empty transmission line and then pack the sample into the line. We then weigh the filled line and measure under ambient conditions. We then place the transmission line under vacuum and heat the line to 40 °C for 48 hr. After the sample has been baked in vacuum, we remove the transmission line containing the sample, we weigh it, and we recalibrate the instrument. During calibration (~45 min), we maintain the transmission line at a temperature of 40 °C to minimize moisture readabsorption from the atmosphere. Once calibration is complete, we place the packed transmission line under vacuum, this time for 2 hr, prior to making a measurement at 40 °C. Once the measurement is made, we set the heating tape to 25 °C and allow the sample to cool in vacuum for at least 10 hr. Once this is complete, we once again weigh the sample and recalibrate the instrument. We then place the sample in vacuum for another 2 hr before performing the final measurement. This method allows us to get two dry measurements of the sample, one at 40 °C and one at 25 °C, which in turn allows us to investigate temperature-dependent permittivity effects. All other aspects of the measurement procedure are as presented in section 3.

5. Results and Discussion

We present results of our measurements of $\varepsilon'_\text{r}$ and $\tan \delta$ at 25 and 40 °C of the powdered UCF/DSI-CI-2 kerogen substitute, the UCF/DSI-CI-2 serpentine component, and mixtures of the two with 2.5, 5.0, and 7.5 wt% kerogen substitute in serpentine. In order to compare these measurements to one another, we normalize them to a bulk density of 1.60 g/cm³ as described in section 3.4.1. Table 1 shows the maximum and median differences.
Table 2

| Sample               | Bulk density (g/cm³) | \(e'_r\) 25 °C | \(\tan \delta\) 25 °C | \(e'_r\) 40 °C | \(\tan \delta\) 40 °C |
|----------------------|----------------------|------------------|-----------------------|------------------|-----------------------|
| Kerogen              | 0.962 ± 0.002        | 2.95 ± 0.01      | 0.039 ± 0.003         | 2.96 ± 0.01      | 0.040 ± 0.003         |
| Serpentine           | 1.60 ± 0.003         | 3.21 ± 0.01      | 0.014 ± 0.003         | 3.22 ± 0.01      | 0.014 ± 0.003         |
| 2.5 wt% kerogen      | 1.58 ± 0.003         | 3.19 ± 0.01      | 0.015 ± 0.002         | 3.19 ± 0.01      | 0.015 ± 0.002         |
| 5.0 wt% kerogen      | 1.56 ± 0.003         | 3.21 ± 0.01      | 0.016 ± 0.003         | 3.22 ± 0.01      | 0.016 ± 0.003         |
| 7.5 wt% kerogen      | 1.50 ± 0.003         | 3.12 ± 0.01      | 0.016 ± 0.002         | 3.11 ± 0.01      | 0.016 ± 0.002         |

\(a\) Normalized measurement.

between \(e'_r[S_{11}, S_{22}]\) and \(e'_r[S_{22}, S_{12}]\), as well as \(\tan \delta[S_{11}, S_{21}]\) and \(\tan \delta[S_{22}, S_{12}]\) for all our samples. The maximum percentage difference for all samples in \(e'_r\) and \(\tan \delta\) are 4.0% and 2.2%, respectively, and the median percentage difference does not go above 0.43% for \(e'_r\) and 0.14% for \(\tan \delta\). These low maximum and median differences testify to the homogeneity of our samples in the transmission line, and the maximum differences in \(e'_r\) are within those obtained by Brouet et al. (2016). As an example, Figure 6 shows \(e'_r\) and \(\tan \delta\) from both sets of S-parameters as well as their averages for the 5.0 wt% kerogen sample measured at 25 °C, which has the highest median difference in \(e'_r\) of all samples measured (0.43%).

Figure 7 shows \(e'_r\) and \(\tan \delta\) for all of our kerogen substitute-serpentine measurements made at room temperature (25 °C). Spikes in the data (mostly visible in the loss tangent) are due to a well-documented resonance effect in low-loss materials that occurs when the length of the transmission line is equivalent to a multiple of a half wavelength in the sample (e.g., Baker-Jarvis et al., 1993). The kerogen substitute shows a significant decline in both \(e'_r\) and \(\tan \delta\) with increasing frequency, while the serpentine component only shows very minor frequency-dependent dispersion in \(\tan \delta\). Both \(e'_r\) and \(\tan \delta\) are significantly greater for

Figure 9. Log-log plot of attenuation (\(\eta\)) versus frequency of the UCF/DSI-CI-2 serpentine component, the UCF/DSI-CI-2 kerogen substitute, and 5.0 wt% kerogen substitute-serpentine mix at 25 °C and normalized to a bulk density of 1.60 g/cm³. Error bars are plotted every 25 data points.
the kerogen substitute than for the serpentine, and tan δ for the serpentine is very low, implying that the serpentine would be much more transparent to radar. As the kerogen substitute proportion increases from 2.5 to 7.5 wt%, we see a clear trend of increasing permittivity, although this effect is very small in tan δ at high frequencies.

Figure 8 shows measurements of the UCF/DSI-CI-2 kerogen substitute, the UCF/DSI-CI-2 serpentine component, and 5.0 wt% kerogen substitute-serpentine mix at 25 and 40 °C. Although the kerogen substitute displays a slight temperature dependence in this range, this dependence is not seen in the kerogen substitute-serpentine mixtures at these weight percentages. At 40 °C, the kerogen substitute exhibits slightly greater relative permittivity than at room temperature. This effect appears to be stronger at lower frequency, though uncertainty in that frequency region is greater. Table 2 shows measurement results at 2.38 GHz, which corresponds to a wavelength of 12.6 cm, which has been used to image Bennu using radar from the Arecibo Observatory on Earth (Nolan et al., 2013).

For nonmagnetic materials such as the ones considered here, the attenuation parameter (in nepers per meter), α, of a material is (Ulaby & Long, 2014):

\[
\alpha = 2 \pi \frac{\varepsilon_r}{\lambda_r} \left( \sqrt{1 + \tan^2 \delta} - 1 \right) \left( \frac{\varepsilon_r - 1}{2 \varepsilon_r + 1} \right)^{1/2} \text{(Np/m).} \tag{18}
\]

α can be reported in decibels per meter (e.g., Stillman & Olhoeft, 2008):

\[
\eta = 20 \log_{10}(\varepsilon^\alpha) \text{(db/m).} \tag{19}
\]

Using equation (19), we calculate the attenuation, η, for our samples. Figure 9 shows the calculated attenuations for the serpentine sample, the 5.0 wt% kerogen substitute sample, and the kerogen substitute sample.

At 2.38 GHz, \( n_{\text{serpentine}}^0 = 6.9 \) dB/m, \( n_{5.0 \text{wt\% \text{kerogen}}}^0 = 7.2 \) dB/m, \( n_{5.0 \text{wt\% \text{kerogen}}}^0 = 7.5 \) dB/m, and \( n_{5.0 \text{wt\% \text{kerogen}}}^0 = 7.9 \) dB/m. This corresponds to an increase in the attenuation of 4.2%, 8.0%, and 12.7% from pure serpentine for the 2.5, 5.0, and 7.5 wt% kerogen substitute mixtures, respectively, for an average increase of 4.2% per addition of 2.5 wt% kerogen substitute. Nolan et al. (2013) calculated a near-surface bulk density for Bennu using Earth-based radar of either 1.65 or 0.9 g/cm³, depending on the method used. Adjusting our results to the lower density of 0.9 g/cm³, the attenuations become \( n_{\text{serpentine}}^{0.9} = 5.5 \) dB/m, \( n_{2.5 \text{wt\% \text{kerogen}}}^{0.9} = 5.7 \) dB/m, \( n_{5.0 \text{wt\% \text{kerogen}}}^{0.9} = 6.0 \) dB/m, and \( n_{7.5 \text{wt\% \text{kerogen}}}^{0.9} = 6.3 \) dB/m. As the regolith of Bennu is likely to contain materials such as magnetite, which are likely to further increase η, these values represent a lower bound for the attenuation in the regolith of Bennu.

## 6. Conclusions

We have developed a comprehensive methodology to measure the complex permittivity of planetary regolith materials in hot and dry environments and over a wide range of frequencies. Our method includes error analysis and propagation, and we have made the Python code used to compute the complex permittivity with uncertainties from S-parameters publicly available and open source (Boivin & Hickson, 2018). Measurements can be made in vacuum (≤126.7 Pa), hot (up to 120 °C), and hot vacuum. Our methods allow for accurate measurements over a broad range of frequencies (400 MHz to 8.5 GHz) of both the dielectric constant and the loss tangent of materials. Measurements can be made in conditions that approximate conditions on planetary surfaces, allowing for accurate measurements of planetary analog materials. Since both measurement conditions and sample composition can be varied, these methods also allow for the exploration of the parameter space of factors which influence the complex permittivity of geological materials. A greater understanding of this parameter space is necessary to further improve quantitative analysis and modeling of planetary surface radar data.

We demonstrate our methods with measurements of the kerogen substitute and the serpentine components of the UCF/DSI-CI-2 asteroid regolith simulant from Deep Space Industries. This simulant is based on CI chondrite meteorite composition and is considered analogous to the surface of asteroid (101955) Bennu. We use our results to calculate the attenuation of our samples at 12.6 cm. We also find that increasing the kerogen substitute content of our analog material significantly increases the attenuation in our material. In addition, we determine that the complex relative permittivity of our analog samples are temperature independent between 25 and 40 °C at kerogen substitute fractions relevant to CI chondrites (and possibly, therefore, Bennu). In the future, measurements of Bennu regolith analog materials should be made containing different weight fractions of the mineral magnetite, which is likely to further increase the attenuation.
Acknowledgments

This material is based upon work supported by the Canadian Space Agency and the Natural Sciences and Engineering Research Council of Canada under contracts and grants to R. R. Ghent and M. G. Daly. Data used in this paper are available on Zenodo under the Creative Commons Attribution 4.0 license (DOI: 10.5281/zenodo.1209359). Software used to process the data is available on Zenodo under the MIT license (10.5281/zenodo.1469776).

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