

REFLECTANCE EXPERIMENT LABORATORY (RELAB)

DESCRIPTION AND USER'S MANUAL

Spectroscopic data acquired in the laboratory provide the interpretive foundation upon which compositional information about unexplored or unsampled planetary surfaces is derived from remotely obtained reflectance spectra. The RELAB is supported by NASA as a multi-user spectroscopy facility, and laboratory time can be made available at no charge to investigators who are in funded NASA programs. RELAB has two operational spectrometers available to NASA scientists: 1) a near-ultraviolet, visible, and near-infrared bidirectional spectrometer and 2) a near- and mid- infrared FT-IR spectrometer. The overall purpose of the design and operation of the RELAB bidirectional spectrometer is to obtain high precision, high spectral resolution, bidirectional reflectance spectra of earth and planetary materials. One of the key elements of its design is the ability to measure samples using viewing geometries specified by the user. This allows investigators to simulate, under laboratory conditions, reflectance spectra obtained remotely (i.e., with spaceborne, telescopic, and airborne systems) as well as to investigate geometry dependent reflectance properties of geologic materials. The Nicolet 740 FT-IR spectrometer currently operates in reflectance mode from 0.9 to 25 μm . Use and scheduling of the RELAB is monitored by a 4-member advisory committee. NASA investigators should direct inquiries to the Science Manager or RELAB Operator.

Personnel and Address	2
Instrument Specifications: UV-Vis-NIR Bidirectional Spectrometer	3
Instrument Specifications: Nicolet Nexus 870 FT-IR Spectrometer	4
General Operations	5
User Instructions	6
User Registration Form	8
Sample Information Form	9
Compositional Analysis Form	10
Measurement Request Form	11

==> RELAB Web Site: <http://www.planetary.brown.edu/relab/>

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INSTRUMENT SPECIFICATIONS: UV-VIS-NIR BIDIRECTIONAL SPECTROMETER

Operating Spectral Range: Nominal (0.32 to 2.55 μm); Possible (0.30 to 2.60 μm)

<i>Mode</i>	<i>Range (μm)</i>	<i>Resolution (nm)</i>	<i>Grating</i>	<i>Detector</i>
A	0.3-0.85	≤ 1.7	1180	Photomultiplier
B	0.6-1.8	≤ 3.4	590	InSb
C	1.7-2.6	≤ 6.8	295	InSb
D	2.0-3.6	≤ 13	147	InSb

Sampling Interval: 5 or 10 nm standard, 1 nm minimum. Inverse centimeter sampling optional.

Precision: Instrumental error is less than 1/4 % in reflectance.

Geometry: Bidirectional reflectance; Spectral goniometry.
Incident and reflected beam can each be varied from normal to 70°.
($-10 < i < 70$; $-70 < e < 70$).
Phase angle minimum 10°, maximum 140°.

Sample: Particulate sample or soil (reflectance):
Measured horizontal and uncovered (normally rotated slowly).
Typical amount required is 500 mg.
Sample should be no smaller than 20 mg.
More than 10 g is unnecessary.
Chip or rock (reflectance):
Measured horizontal and uncovered on a height-adjustable stage.
Regions to be measured should be no smaller than 1.5 mm.
Maximum sample size is 10 cm in diameter (2.5 cm to be spun).
Thin section (reflectance or transmittance):
Measured horizontal on halon (reflectance) or on an aperture (transmittance).
Regions to be measured should be no smaller than 1.5 mm.
Surface should be polished and clean (no carbon coating).

Reference Standard: Halon (pressed; approximate Lambertian surface; reflectance < 0.97).
Corrections based on the NBS calibration are applied automatically
Corrections for the non-Lambertian properties of halon are available on request.

Light Source: Quartz halogen lamp; Jarrel Ash half-meter monochromator.

Optics: Order sorting filters, front surface mirrors, depolarizer, apertures.

Typical Run Time: Measurements over a spectral range of 0.35 to 2.55 μm at 5 nm sampling interval take ~2 hours per sample.

INSTRUMENT SPECIFICATIONS: Thermo Nexus 870 FT-IR SPECTROMETER

Operating Spectral Range: Nominal (1.0 to 50 μm) Possible (0.8 to 200 μm)

<i>Mode</i>	<i>Range (μm)</i>	<i>Light Source</i>	<i>Beam Splitter</i>	<i>Detector</i>
A	1.0– 4.7	Quartz	Si-on-CaF ₂	TE cooled DTGS
A*	0.9– 4.5	Quartz	Si-on-CaF ₂	LN ₂ cooled InSb
B	1.3 - 28	Glowbar	Ge-on-KBr	TE cooled DTGS
C	14.3-200	Glowbar	Solid substrate	DTGS

Sampling Interval: 16, 8, 4, 2, 1 or 0.5 cm^{-1}

Default: 4 cm^{-1}

Geometry: Biconical - off axis for multiple sample holder (Pike attachment)
Transmission (normal incidence)

Sample: Particulate sample or soil (reflectance):

Measured horizontal and uncovered in purged (H₂O, CO₂ free) environment.

Typical amount required is 100 - 500 mg.

Sample should be no smaller than 20 mg.

More than 1 g is unnecessary.

Multiple samples measured in sequence (same environment)

Chip (reflectance):

Measured horizontal and uncovered.

Regions to be measured should be no smaller than 1 mm.

Maximum size is 1 cm in height and 3 cm in diameter.

Thin section and single crystal (transmission):

Measured vertical, attached to an appropriate aperture board.

Regions to be measured should be no smaller than 1.5 mm.

Surface should be polished and clean (no carbon coating).

Reference Standard: Brushed gold (reflectance)

Blank (transmittance)

Additional Attachments: Pike AutoDiff Diffuse Reflectance Autosampler [new]

Continuum Microscope system [new]

Transmission mode currently operational

Reflectance mode (with 2-D capabilities) being tested.

Software: OMNIC, Atlas [new]

GENERAL OPERATIONS

Use of the RELAB should be initially arranged through the Science Manager. New Investigators must fill out the attached *User Registration Form*. All investigators should provide information on each sample to be measured using the attached *Sample Information Form* and *Compositional Analysis Form* if chemical or mineral composition is available. A completed *Measurement Request Form* must be provided for each set of samples to be measured under a common measurement condition. This information is very important in order to maintain a useful and up-to-date data base of spectroscopic data.

Bidirectional Spectrometer:

Powders, chips, or slabs can be run using the RELAB bidirectional spectrometer. Although sample dishes come in various sizes, the smallest sample that can be accurately handled is about 20 mg, while the optimum sample size is about 500 mg.

The standard sample dish for the bidirectional spectrometer is aluminum coated with black teflon. Its reflectance is less than 3% throughout the entire spectral range of the bidirectional spectrometer.

For analysis of heterogeneous samples, viewing optics include precision apertures for measurements from broad sample areas up to 1 cm in diameter down to areas as small as 1 mm in diameter.

To initiate a data run the USER must supply and specify the following run parameters: (a) sample name and properties; (b) angle of incidence (light source) and angle of emission (reflectance); (c) wavelength range (start and stop); and (d) sampling interval. Nominal settings are provided.

Based on these run parameters specified by the user, the RELAB Operator configures the system and performs all the data acquisition. A complete spectrum from 350 to 2550 nm at 5 nm sampling interval requires about 2 hours of laboratory run time per sample, depending on characteristics.

At the end of a run, values for the sample signal, the standard signal, the offset, the ratio of sample/halon (after offset subtraction), and the standard deviation of signal variance during integration on the sample are recorded on disk in a file with all run parameters and text. These data files, each identified by a unique run ID, become part of the data base collection. NBS calibration of halon is a (multiplicative) correction to the ratio data producing reflectance values for the sample.

FT-IR Spectrometer:

A Nicolet Nexus 870 FTIR spectrometer is operational for samples of particulate samples using a Pike AutoDiff multi-sample attachment. Infrared [off-axis] biconical reflectance spectra are normally produced relative to a gold standard. Infrared spectra may be scaled to and merged with bidirectional reflectance spectra to produce continuous spectra from 0.3 to 26 μm . The Pike multi-sample biconical reflectance attachment is normally requested by outside users as the standard mode for midIR data. Use of the Nicolet Continuum microscope is available, but requires training or prior experience. Contact the RELAB science manager or operator for information.

USER INSTRUCTIONS

1) After the initial contact with the RELAB Science Manager to arrange use of the facility, users should contact the RELAB Operator directly to schedule samples to be measured. Investigators should carefully select the most appropriate sample preparation and RELAB configuration for their specific science requirements. If uncertain of measurement strategy, contact the RELAB Operator or Science Manager for advice or suggestions. [During periods of heavy use of the facility, requests for extensive numbers of spectra may be given lower priority in the measurement queue, therefore it is important to prioritize measurements in any large request.]

2) Investigators (and/or their students) should fill out the attached **User Registration Form** with up-to-date information. Sample ID's assigned at the RELAB use the investigators' three initials. If you have published work using RELAB spectra, please also include a **publication list**. These will be included in a public spectroscopy bibliography on the RELAB web site. (It is unnecessary to send references already included.)

3) For *each set of samples* to be measured under common conditions (same geometry, wavelength range, sample interval, etc), the investigator is requested to fill out the attached **Measurement Request Form**. It includes information to specify the type of measurement to be made in the RELAB.

- a) Applicable Samples: Identify all the submitted sample names which are to be measured under a common measuring condition described in the form, or write "All".
- b) Research Type: For accounting purposes, circle at least one category which accounts for support of this research. The research type should reflect the NASA involvement of the PI/CoI or Sponsor.
- c) Wavelength Range and Sampling Interval or Resolution: Choose one of standard values or specify.
- d) Geometry: Incidence and emergence angles are measured from the vertical direction in positive and negative values of degrees (negative values are on the opposite site). For example, a geometry with source light at 30° inclination and detector at 20° inclination from the vertical direction would be (30, -20) which makes 50° phase angle. Phase angle must be 10° or larger for the Bidirectional Spectrometer and all measurements are currently in the plane of scattering. Geometry for the FT-IR Spectrometer is fixed with broad conical incidence and reflectance at (30, -30, but out of the plane of scattering).
- e) Date Desired: Indicate the date spectral data need to be completed.
- f) Public Release Date: Specify when spectroscopic data on this sample can be included in a public data base available to other investigators in digital form. Typical period is three years. Default is immediate release.
- g) Data Transfer Preference: Choose all desired (most common is electronic transfer). Floppy disks are available in IBM or Macintosh format (3.5").

4) For *each sample* the investigator must fill out the attached **Sample Information Form**. The information requested includes:

- a) Sample Name: Formal name or the investigator's name for the sample (can be used in a data base search).
- b) Origin: The geographical site from which the sample was obtained or where it was prepared. If it is extraterrestrial, provide appropriate information - Meteorite fall/find location, Apollo lunar site, etc.
- c) Location: Where the sample will be (semi)permanently stored.
- d) Project Topic: Purpose for measuring the samples; general application.
- e) Comment: General comments about the sample and/or its history.
- f) Texture: Powder, slab, coarse-grained sand, fine-grained soil, thin section, etc.
- g) Minimum and Maximum Particle Size: For particulate samples, the maximum and minimum (if known) particle sizes in micrometers.
- h) General Type, Type, Subtype: Three levels of sample description in addition to Origin. See example below (multiple keywords are allowable in both the Type and Subtype fields).

Descriptive examples:

<i>General Type</i>	<i>Type</i>	<i>Subtype</i>
Mineral	Silicate	Pyroxene, Enstatite
Mineral	Carbonate	Calcite
Rock	Igneous, Plutonic	Granite
Rock	Metamorphic	Gneiss
Soil	Sand	Quartz
Soil	Mare	Bulk, Immature
Meteorite	Achondrite	Basaltic, Eucrite
Experimental	Heated	Basalt
Man-made	Mixture	Olivine, Enstatite
Biological	Plant	Maple Leaf

5) Whenever available, *modal mineralogy* for the sample and/or *chemical analysis* for either the bulk sample or for individual mineral constituents should be provided on the **Compositional Analysis Form**. This important information will be stored in a separate file linked to the sample.

6) Samples should be sent to the RELAB Operator with completed forms and any other necessary instructions. Although not necessary, any NASA investigator is welcome to visit the RELAB at Brown University to prepare their samples for measurement in person and to use available data analysis tools. This should be prearranged to prevent schedule conflicts. A small office is available for visitors.

7) Data products are sent to the investigator (digital and/or hard copy). Special requirements should be discussed with the RELAB Operator.

8) RELAB forms are downloadable in PDF form from our web site along with Microsoft Word files. Contact the RELAB Operator if there are any problems downloading the forms.

User Registration Form

NASA-funded projects have access to RELAB at no charge to User.
Each group of measurements must be accompanied by Measurement Request Forms.

Principal Contact:

Date _____

Full Name: _____
 First Middle Initial Last

Affiliation: _____

Mailing Address: _____

E-Mail Address: _____

Phone: _____ Fax: _____

Secondary (or Student) Contact:

Full Name: _____
 First Middle Initial Last

Affiliation: _____

Mailing Address: _____

E-Mail Address: _____

Phone: _____ Fax: _____

* For RELAB use:

PI Code: _____

SI Code: _____

Sample Information Form (One for each sample)

Investigator: _____

Sample Name: _____

Origin: _____

Location: _____

Project Topic: _____

Comment: _____

Texture: _____

Particle Size: _____ μm
(Minimum) (Maximum)

General Type: _____

Type: _____

Subtype: _____

Is compositional information available? () Yes () No
(If yes, please also submit **Compositional Analysis Form.**)

Priority Level: _____ (used in **Measurement Request Form**)

* For RELAB use:

MM Analysis #: _____ Bulk Chem. #: _____

ID: ____ - ____ - ____ - ____

PI: ____

SI: ____

PO: _____

Compositional Analysis Form (One for each sample if available)

Investigator: _____

Sample Name: _____

Text (identify facility and/or investigator performing analysis): _____

Chemical Analysis (wt%):

SiO ₂ : _____%	TiO ₂ : _____%	Al ₂ O ₃ : _____%	Cr ₂ O ₃ : _____%
V ₂ O ₃ : _____%	Fe ₂ O ₃ : _____%	FeO: _____%	CoO: _____%
NiO: _____%	MnO: _____%	MgO: _____%	ZnO: _____%
CaO: _____%	Na ₂ O: _____%	K ₂ O: _____%	P ₂ O ₅ : _____%
: _____%	: _____%	: _____%	: _____%

Include minor or trace elements information on a separate sheet.

Modal Mineralogy:

Mineral / Component	(wt/vol) %	Mineral / Component	(wt/vol) %
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

If available, provide chemical analysis for individual minerals on separate sheets.

* For RELAB use:

Sample ID: _____

M Mineral #: _____

C Analysis #: _____

Measurement Request Form (One for each group or subgroup of samples)

Investigator Name: _____

Applicable Samples: _____

Research Type (circle at least one): PGG Cosmochemistry Astronomy Astrobiology

Student LDAP MDAP MFRP DDAP NASA-ESA-FSU-Japan-Canada

Active missions Mission planning Proposal preparation Other: -

NASA PI/CoI or Sponsor (if different from Investigator) : _____

Bidirectional Spectrometer (* Standard parameters)

Wavelength Range: * 0.32-2.55 μm 0.3-2.6 μm Other _____

Sampling Interval: * 10 nm 5 nm Other _____

Geometry (incidence, emergence angles): * (30, 0) Other _____

FT-IR Spectrometer (* Standard parameters)

Wavelength Range: 1-4 μm * 2-25 μm 17-50 μm Other _____

Wavenumber Resolution: 1 cm^{-1} 2 cm^{-1} * 4 cm^{-1} Other _____

Schedule & Data Transfer

Date desired for the priority levels specified in the Sample Information Forms

Priority Level: _____

Date Desired: _____

Public Release Date: Immediate 1 Year 2 Years 3 Years

Data Transfer Preference: E-mail 3.5" floppy disk [IBM / Macintosh]

FTP [Host: _____ Password: _____ Directory: _____]

CD-R Plot on paper Other _____