APPARATUS TO MEASURE THE MID-INFRARED SPECTRAL EMITTANCE

OF COLD POWDERS IN A VACUUM

by

Alexander F. H. Goetz and Curtis A. Bauman

Division of Geological Sciences*
California Institute of Technology
Pasadena, California

Abstract

A compact apparatus enclosed in a stainless steel vacuum chamber was used to measure the 8-13 μ emittance of silicate powders under simulated lunar conditions. Samples with surface temperatures as low as 180° K were measured. The sample was contained in the removable lid of a constant temperature bath and its upper surface allowed to radiate into a cavity maintained at liquid nitrogen temperature (77° K). The spectral emittance was computed by matching the observed spectrum with a Planck function at specified wavelength intervals. System response was determined with an internal calibrating blackbody maintained at 273° K.

Introduction

The spectral emittance of powdered materials, mainly silicates, has been the object of considerable investigation in the last few years because of its potential importance in remote compositional mapping of the moon and planets (1, 2, 3, 4).

Rock-forming silicates exhibit departures from graybody emission at frequencies (and hence wavelength) corresponding to their basic lattice

---

* Contribution No. 1427

FACILITY FORM 602

N67-26506
(ACCESSION NUMBER)

12
(PAGES)

CR 8458
(NASA CR OR TMX OR AD NUMBER)

_____
(THRU)

/
(CODE)

13
(CATEGORY)

vibrations; such resonances are dependent mainly on the type of Si-O
bonding (5). The fundamental stretching vibrational wavelengths lie in
the 8-13 μ region which corresponds to a major transmission window in
the earth's atmosphere.

All previously published measurements of spectral emittance of silicate
powders at atmospheric pressure and at temperatures near or greater than
lunar sub-solar point temperatures (370° K) to reduce interference from
room temperature sources. The apparatus described in this paper was
designed to more closely simulate lunar surface conditions of temperature,
pressure and thermal gradient. The sample is placed inside a vacuum chamber,
is heated from below and its surface radiates into a cavity maintained at
liquid nitrogen temperature (77° K).

Spectral emittance of a sample, $\epsilon(\lambda)$, is defined as the ratio
between the flux emitted by the sample and that emitted by a blackbody of
the same temperature at a given wavelength.

$$\epsilon(\lambda) = \frac{W_s(\lambda)}{W_{bb}(\lambda)} \tag{1}$$

The energy flux from a blackbody is given by the expression

$$W_{bb}(\lambda) = \frac{C_1 \lambda^{-5}}{e^{C_2/\lambda T} - 1}$$

where C_1 and C_2 are constants. To determine the emittance the sample
surface temperature must be determined.

The thermal conductivity of silicate powders in a vacuum is very
low (10^{-5} - 10^{-6} cgs). It is therefore not possible to measure accurately
the surface temperature by means of thermocouples or similar devices
because of the high point contact thermal resistance.

In silicate powders the departures from graybody emission are relatively sharp (1/2-1 microns wide) with respect to shifts in the Planck function curve due to changes in temperature. In the 8-13 micron region these powders exhibit emittances approaching unity at several wavelength intervals. Using a blackbody reference at a known temperature to determine system response, it is possible to fit the sample spectrum at high emittance wavelength intervals to a Planck function curve in a computer. The ratio of these two then gives the relative spectral emittance over the wavelength region of interest. The term relative is used since the actual kinetic surface temperature is not known and the assumption of unity emittance at given wavelengths is not always valid. However, the relative departures from graybody emission are present, and it is these that are important in identifying composition.

Vacuum System

The vacuum system consisted of a pumping station, a base plate through which the bath water lines were led and a 9 inch high, 7 inch diameter, 1/8 inch wall cylindrical stainless steel chamber. The system was evacuated by a 4 inch oil diffusion pump (750 liter/sec. at 10^{-3} Torr) with a Freon cooled baffle backed by a mechanical pump. The pressure ranged from 10^{-5} to 10^{-6} Torr during experiment runs. The system was usually evacuated in less than an hour. A throttle system was used to avoid evacuating the chamber so rapidly that the powders were disturbed.

Apparatus

The present system is based on an earlier instrument designed for measuring the thermal conductivity of powders in a vacuum (6) but has been altered considerably for the sake of cooling efficiency and compactness.

Fig. 1 shows a cross-sectional view of the chamber. The main parts comprising the system are: A, the outer chamber wall; B, the annular dewar or shroud with a capacity for one liter of liquid nitrogen; C, the combined constant temperature bath and sample holder; D, cold diaphragms; E, the calibration device; F, the chopper; G, the phase detector; H, the magnetic drive assembly; I, the BaF_2 window. The entire chamber and its contents except for the calibration device and the chopper were constructed of stainless steel. The annular dewar is supported only by J, the fill tube, at the top and K, three knife edge nylon supports, at the bottom, directly above the sample holder. This shroud provides a cold half space into which the powder surface radiates. It was filled automatically at 30 minute intervals from a 25 liter storage dewar by a simple clocked timed heater contained in it. The consumption under normal operating conditions is 25 liters in 24 hours.

The constant temperature bath is supported on the base plate by a lucite spacer directly above the throat of the diffusion pump. The bath has a removeable lid with a 1/4 inch depression in its surface which acts as a sample holder. The water inlet is placed at the bottom of the bath while the outlets are at four points along an edge groove concentric with the holder but above its lower surface. This construction does not allow an air bubble to form along the bottom of the holder that would reduce

thermal contact with the water. Both input and output temperatures of

the water flow are monitored. The difference was never more than 1.2° C.

The water was not recycled and was heated by an in-line 2500 watt heater.

The temperature could be varied continuously from the water main temperature

of about 20° to 100° C. The maximum variation over a 24 hour period was

$\pm 2^\circ$ C.

The calibration device is located approximately midway between the

sample surface and the chopper. Its function is to provide blackbody, or

more accurately, graybody emission at a known temperature. This is

accomplished by pumping ice water through the device. The device, shown

in Fig. 1, consists of a 30° half-angle cone machined into a slightly

larger copper cone about 1 inch long. This piece is hard soldered into

a hollow cylinder containing in-   and outlets for cooling water. The

base of the inverted cone is masked with a 1/2 inch diameter diaphragm

slightly larger than the f-16 beam width at that point. The inner surfaces

are coated with Krylon Primer. The device can be inserted laterally into

the measuring beam, from outside the chamber, obscuring the sample surface.

The chopper is a piece of blackened, round aluminum stock, 1 1/2 inches

in diameter, through which a square opening has been cut in such a way

that the circumference of the chopper is equally divided. For an infinitely

small beam the theoretical chopped output would have a square waveform.

However, in this system the beam width at the chopper is about 1/4 inch

in diameter resulting in a trapezoidal waveform. The chopper is supported

by L,bakelite rod shafts,ending in N,Barden corp. Teflon race ball bearings.

The bearings are in the vacuum but remain at the temperature of the outside

wall. These bearings have functioned for several hundred hours already

without need of replacement. Attached to one shaft is a second chopper
which interrupts a light-photocell arrangement to provide a phase reference.
The extreme end of the shaft holds a magnet. Directly adjacent is the
.020 inch thick stainless steel housing wall of the bearing assembly.
Opposite to the interior magnet is mounted M, the exterior 3600 rpm
synchronous chopper motor and drive magnet.

The signal chopper is cooled by radiation alone and a cold diaphragm
is placed directly above the chopper to limit any radiation from outside
the f-16 beam. Another diaphragm, thermally coupled to the nitrogen shroud,
is placed between the calibration unit and the sample surface to isolate
the two.

In this system a 2mm thick powder sample comes to thermal equilibrium
in 4-6 hours.

Detection System

The flux emitted from the surface into the cooled cavity passes
through the adjacent cold diaphragm and is chopped at 120 Hz. The
resulting modulated flux passes out through the second cold f-16
diaphragm and is transmitted out of the chamber through the .060 inch
thick BaF_2 vacuum window. The transmission of the material is fairly
uniform across the 8-13μ region falling off rapidly beyond 13μ.

The useful wavelength range of this apparatus is only limited by the
vacuum window material used.

The emerging flux is fed into a modified Ebert-Fastie (7) grating
spectrometer coupled with a liquid hydrogen cooled, mercury doped germanium

detector. The signal is amplified and synchronously detected using the
phase reference signal generated by the phase detector coupled to the
chopper shaft. The DC output signal is displayed on a chart recorder and
simultaneously digitized and punched on paper tape. The flux received
at the detector is the difference between the combined flux from the sample,
the diaphragms, the window and the spectrometer on one half chopper cycle,
and the flux from the cold chopper, the diaphragms, the window and the
spectrometer on the other half cycle. The net signal produced is then
actually the difference between the fluxes from the sample and the chopper.
Since the chopper is cooled by radiation to a temperature (approximately
100° K), which is below the minimum detectable temperature of the system,
it acts as a zero reference level. The signal which is amplified is that
from the sample (or calibration unit) alone. The detector receives energy
through a 7.8 μ long wave pass filter and the spectrometer operates in
the first order. The spectral resolution averages about .07 μ. The
peak signal to noise ratio, for a sample having a surface temperature of
180° K, is about 60:1.

Emittance Measurements

Fig. 2a shows the computer output for a sample of quartz powder,
grain size 70-150 microns. The two smooth curves correspond to blackbody
curves 5° K apart. It is obtained by matching theoretical blackbody curves
to the sample on the basis of flux vs. wavelength from the sample plotted
on the same scale. It is used to compute the relative emittance shown
in Fig. 2b according to eq (1).

A detailed discussion of the experimental results of this investigation
will be published elsewhere.

Acknowledgments

References

1. Conel, J. E., Infrared Thermal Emission from Silicates, Jet Propulsion Laboratory Technical Memorandum, No. 33-243, (1965).

2. Lyon, R. J. P., Evaluation of Infrared Spectrophotometery for Compositional Analysis of Lunar and Planetary Soils: Part II Rough and Powdered Surfaces, NASA Contract No. NASr-49(04),(1964).

3. Lyon, R. J. P., Analysis of Rocks by Spectral Infrared Emission (8 to 25 microns), Econ. Geol., 60, 715-736, (1965).

4. Van Tassel, R. A., and I. Simon, Thermal Emission Characteristics of Mineral Dusts in "The Lunar Surface Layer". J. Salisbury and P. Glaser, (eds.), Academic Press, 469-481, (1964).

5. Launer, P. J., Regularities in the Infrared Absorption Spectra of Silicate Minerals, Am. Min. 37, 774-784, (1952).

6. Watson, K., and C. A. Bauman, Apparatus to Measure the Thermal Conductivity of Powders in Vacuum from 120° to 350° K, Rev. of Sci. Inst., Vol. 34, No. 11, 1235-1238, (1963).

7. Fastie, W. G., A Small Plane Grating Monochromotor, J. O. S. A., 42, No. 9, 641-647, (1952).

Figure 1. Emittance apparatus. A, outer chamber wall; B,liquid N_2 shroud; C, constant temperature bath; D, cold diaphragms; E, calibration device; F, signal chopper; G, phase reference; G1, lamp; G2, chopper; G3, photo-diode; H, drive magnets; I, BaF_2 window; J, liquid N_2 fill tube; K, nylon knife-edge supports; L, bakelite drive shaft; M, synchronous motor; N, Teflon race bearing.

Figure 2a. Emission spectrum of 70-150 micron size quartz powder with closest fitting blackbody curve. Upper curve is a Planck function 5° K higher in temperature.

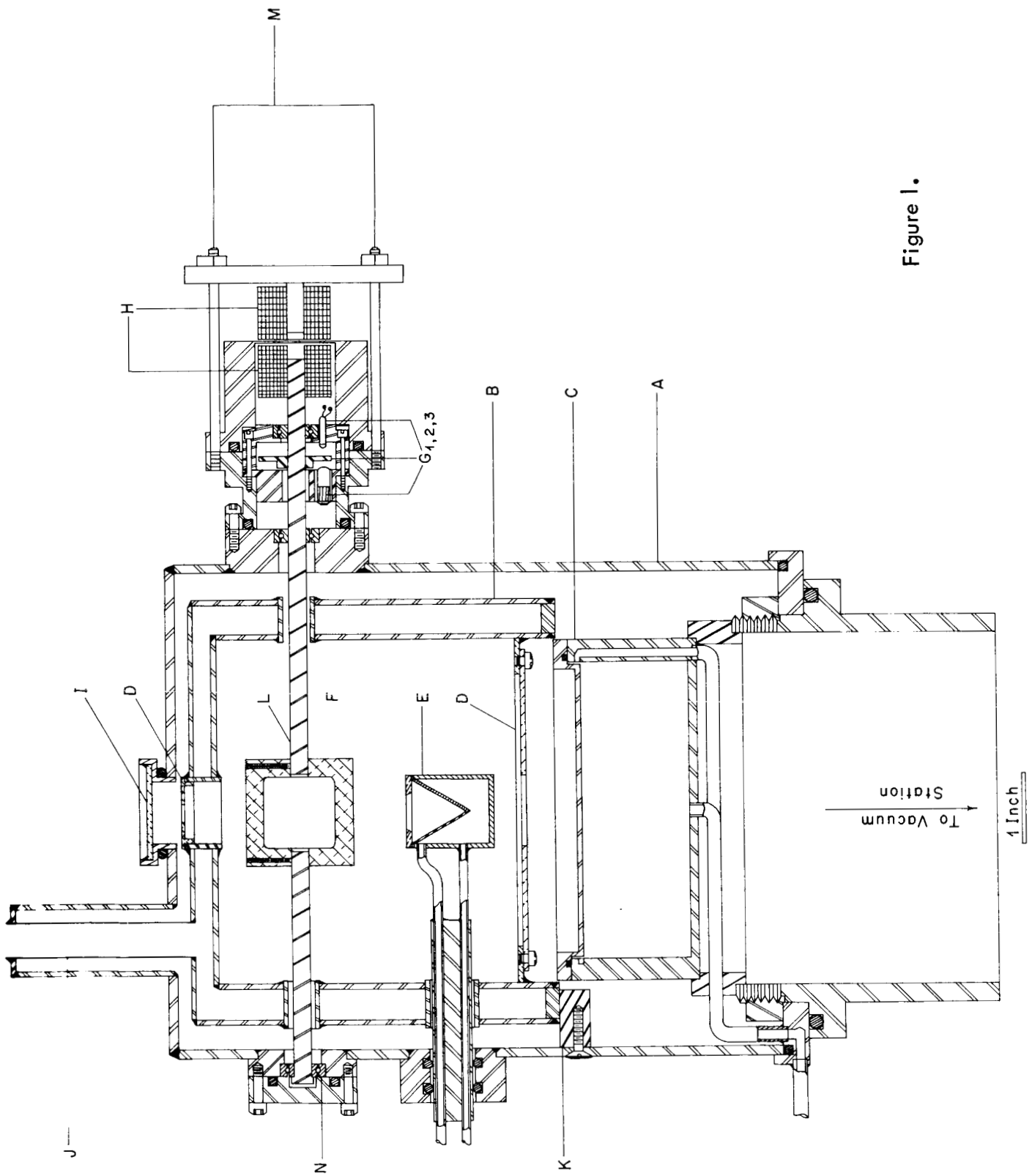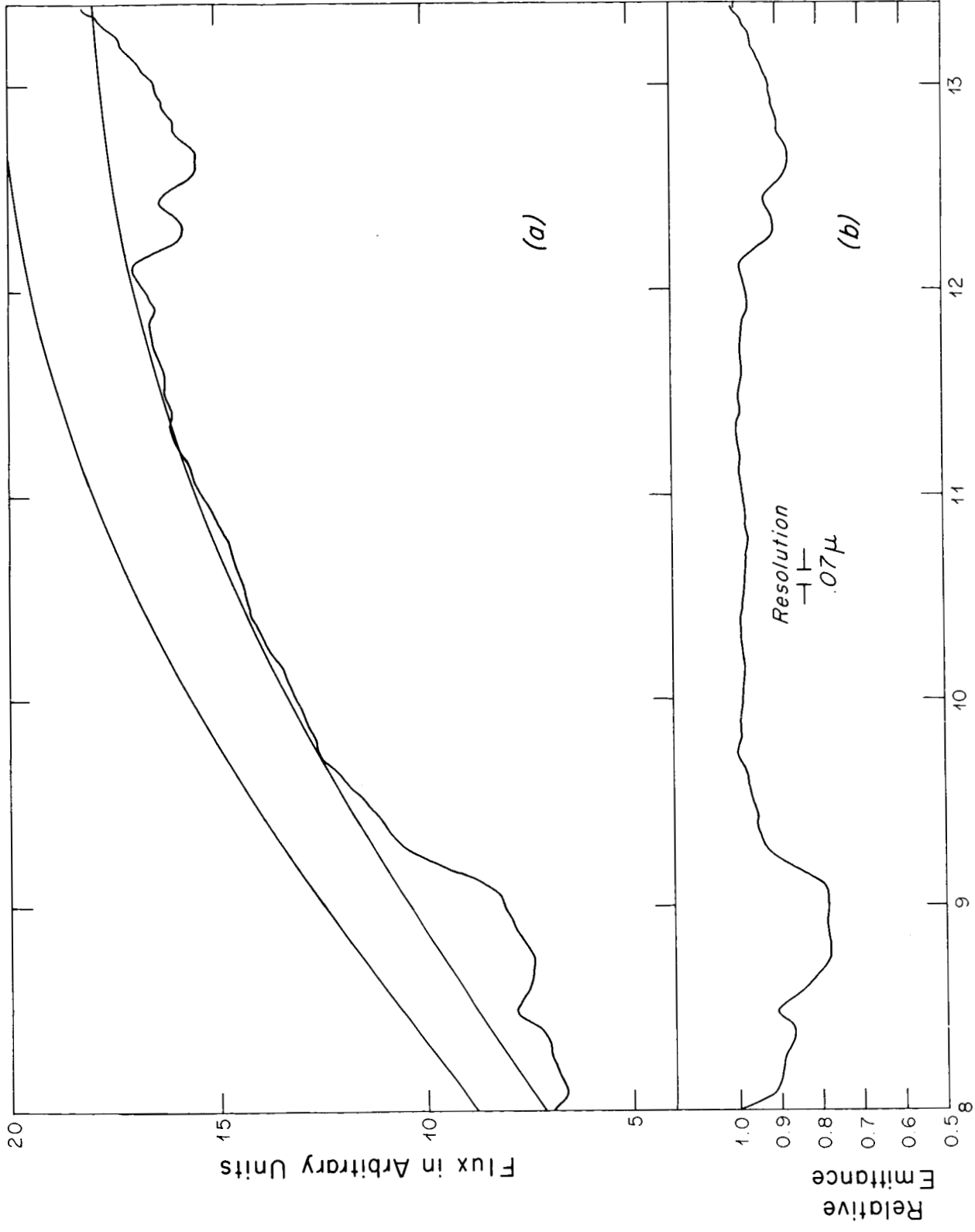Figure 2b. Relative emittance of the quartz sample calculated according to equation (1).

Figure 1.

To Vacuum
Station

1 Inch

Resolution
⊤
⊥
.07 μ

(a)

(b)

Wavelength in μ

Flux in Arbitrary Units

Relative Emittance

Figures 2a and 2b.