Appendix A

Spectrophotometer Modifications

A spectrophotometer was modified to measure the transmittance of the E760 central calorimeter lead-glass blocks. Due to the length of the lead-glass blocks (see table 3.1), physical modifications to a spectrophotometer setup were necessary. Due to some inherent problems with the modified spectrophotometer setup, software modifications were also necessary. This appendix explains the necessary modifications.

There are two types of variable wavelength spectrophotometers: photo-diode array and conventional scanning. Photo-diode array spectrophotometers are advantageous since a whole spectrum can be measured within seconds instead of the longer time a conventional scanning spectrophotometer takes to turn a grating. A Hewlett-Packard 8451A Diode Array Spectrophotometer was employed in this work. The 8451A is capable of 2 nm resolution between 190 and 820 nm and the sample time is one second. Hewlett-Packard has also developed computer software which allows a personal computer (an IBM AT was used) to control the 8451A spectrophotometer through a HPIB interface bus.

A.1 Physical Modifications

Spectrophotometers are generally used in chemistry to measure absorbances of solutions contained in cuvettes. Therefore, the sample compartment for most spectrophotometers is about 12 cm and has an apparatus to hold cuvettes. Although some of the block widths could fit into the sample compartment, this was not feasible. Besides being awkward, the non-parallel side faces of the lead-glass blocks would have refracted the light away from the spectrophotometer detector slit. The lead-glass blocks have only two parallel faces: the front and back faces, which are 35 to 50 cm apart.

The 8451A spectrophotometer has a deuterium lamp as the source. To keep the modification of the 8451A sample compartment to a minimum, an external source has been substituted for the deuterium lamp. A Hamamatsu 75 watt Xenon arc lamp is used as the source; figure A.1 shows the spectrum. The shutter control is disconnected from the spectrophotometer's internal deuterium lamp and connected to a shutter for the Xenon light source.

A new sample compartment, 55 cm tall and 20 cm square, was built. There are holes at the top and bottom where fiber optics were mounted to the compartment. The adjustable top mount allows the distance between the fiber optic mounts to be set between 40 to 60 cm.

The source, the new sample compartment and the spectrophotometer are connected optically by two Applied Photonic Devices 2 m long quartz fiber optic cables,

Figure A.1: The xenon lamp spectrum that has been used in the modified spectrophotometer setup.

as shown in figure A.2. The connecting fiber optic cables are 660 μm core diameter, cladded and terminated SMA style. One fiber optic cable is connected to the xenon lamp source housing. The other end of the fiber optic cable is mounted in a collimating beam probe with a focal length of 19 mm, which is then mounted at the bottom of the sample compartment. Two other collimating beam probes are connected to the ends of the second optic fiber and mounted in the adjustable top mount of the sample compartment, making a collecting lens, and on a platform located in the spectrophotometer's sample compartment. Also located on the platform, a 1/4 wave adjustable mirror directs the collimated beam into a slit leading to the spectrograph, a grating which reflects the light onto a series of diodes.

The responses of the setup to different wavelengths of light have to be taken into consideration. The collimating beam probe consists of a plano-convex fused silica lens: focal length of 19 mm at wavelength 587 nm, according to the Oriel Optics Catalogue. The focal lengths at 335 and 644 nm wavelengths are 18.16 and 19.07 mm, respectively, using the refractive indices at these wavelengths from the catalogue. The finite size of the optical fiber (0.6 mm diameter) does not compensate for the 0.9 mm focal length difference for the two wavelengths. The opening angle of the optical fiber was found to be about 15°, whereas the lens from the focal point subtends an angle of 16°. Thus not all of the wavelengths will be emitted parallel to the axis of the beam probe for a fixed geometry. The light beam diverges within the sample compartment. Also, the angle of collimation is calculated to be 0.0315 (from the Oriel Catalogue, $C = d/f$ where C is the angle of collimation, d is the

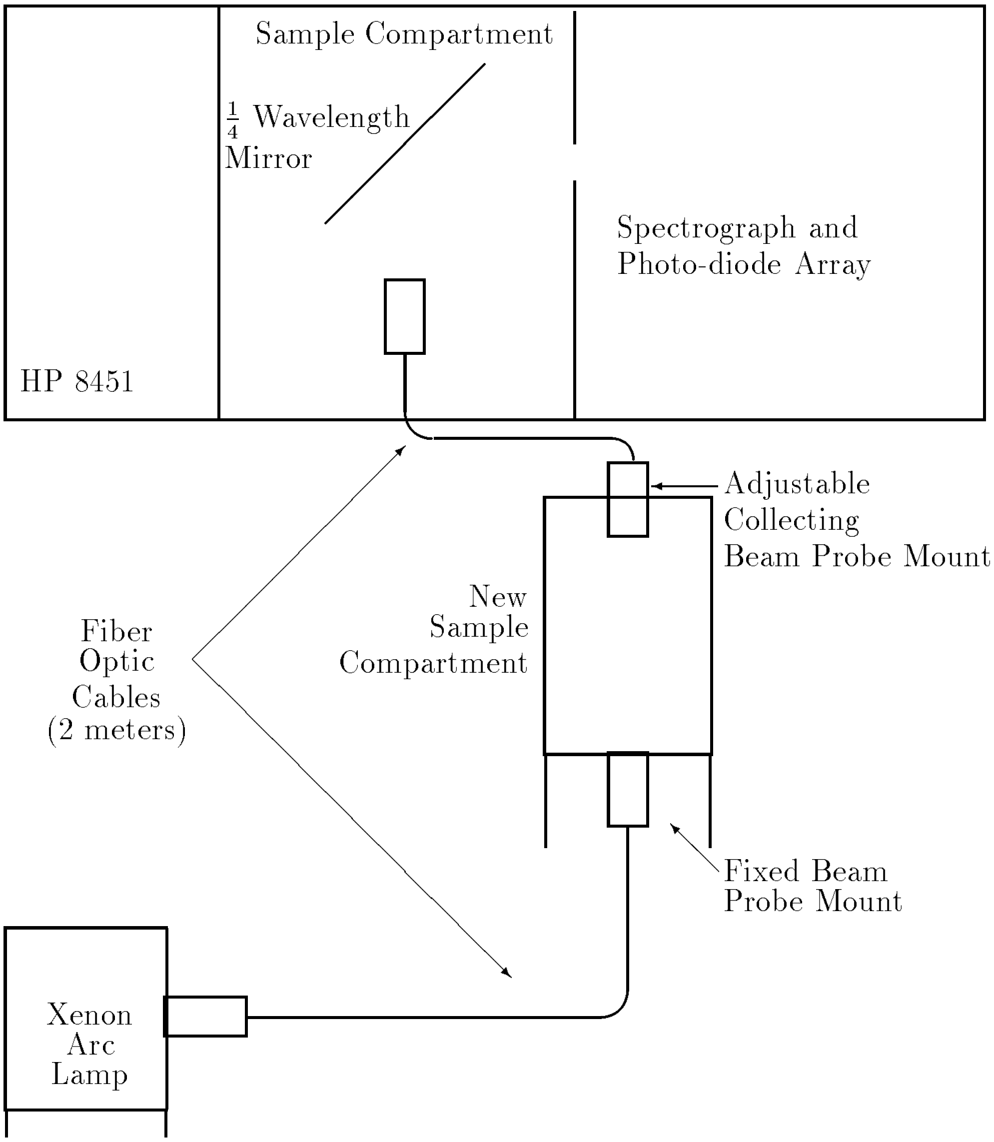

Figure A.2: The modified spectrophotometer setup (shown not to scale).

diameter and f is the focal length). Using the angle of collimation and Snell's Law, the diameter of the light spot at the collecting lens is 0.78 mm smaller for wavelength 589 nm through 40 cm of F2 lead-glass when compared to 40 cm of air. The F2 lead-glass effectively focuses the light. By the time the light reaches the collecting lens, the spot is significantly larger than the collecting lens. The ratios of the areas of the collecting lens and beam spots with 40 cm of air (F2 lead-glass) is calculated to be 0.74 (0.90).

Achromatic lenses were investigated as a possible solution to the non-parallelism of the collimated beam. Literature research showed that a commercially available achromat might be a small improvement compared to the collimating beam probes. An achromat was purchased to be used in conjunction with the collecting collimated beam probe. There, however, was no improvement and it was decided to only use the collimated beam probes.

Generally, one would like to compare the sample lead-glass blocks to a known reference. Ideally, a short piece of the same material is used as the reference, but the focusing effect is greater for the longer pieces of lead-glass when the collecting lens is a fixed distance from the emitting lens. For a reference piece of lead-glass that is 25% of the length of a lead-glass block, the collecting lens can not be moved far enough to ensure the same beam spot size for both lead-glass pieces. Air is another choice for the reference. Again, the collecting lens could not be moved far enough to compensate for the focusing effect of the lead-glass. In either case, there would need to have been a very reliable method to determine the spot size and reproduce the size reliably at the collecting lens. Calculations suggested that the lens placement has to be accurate to 0.5 mm to be within 5% of the same spot size. The use of a set screw does not allow for the needed placement accuracy. Keeping the collecting lens fixed, corrections to the spectra are needed to compensate for the difference in light spot sizes and, therefore, intensities.

A.2 Software Modification

Transmittance is calculated in terms of the ratio of intensities through the sample, corrected for dark current, with respect to a reference. For a fixed collecting lens, *transmittance* of a 40 cm lead-glass block with respect to air is greater than 100% due to the focusing. It was decided to correct for the difference in spot sizes through software. Since the spot size differs for different wavelengths, a correction factor has to depend on wavelength.

A direct way to find a wavelength dependent factor was to change the spot size in air by moving the collecting probe, as the spot size would change between lead-glass and air measurements. While the collecting probe was moved, the intensities were recorded every 1 to 2 mm and compared to the greatest recorded intensity (the smallest spot size–smallest separation distance). A total of sixty-one air intensity spectra were recorded representing the full range of separation distances. The intensity of wavelengths 600, 450 and 350 nm were reduced over the course of travel to 54.3%, 53.4% and 52.3% ($\pm 0.5\%$) of their greatest values, respectively, showing a small wavelength dependence. The intensity values of the spectra at 600 nm were divided by the 600 nm intensity value from the spectrum taken when the beam probe separation was smallest. These intensity ratios are used to correct for the focusing, see below.

The procedure to measure the transmittance of the F2 lead-glass blocks with the spectrophotometer is as follows. After the instrument has been allowed to warm up, dark current and air reference measurements are made and recorded with the sample compartment empty. A lead-glass block is then placed in the sample compartment. While the lead-glass block is rotated about its axis, intensities at several wavelengths are sampled continuously. Up to 5% fluctuations of the intensities are observed during a full rotation of a lead-glass block. The changing intensities can be explained as due to the end faces of the lead-glass block not being parallel; the light is not at normal incidence at the exit face, causing the spot not to be centered upon the collecting lens. The smaller wavelengths are affected more than the green part of the spectrum of interest. Six wavelengths' intensities are continuously monitored as the lead-glass block is rotated once and then turned to the orientation which results in the greatest intensity values. Next, an entire intensity spectrum from 300 to 600 nm is measured and recorded. The ratio of the air to lead-glass intensities at 600 nm is calculated. The closest ratio for 600 nm intensities from above (air spectra with the beam probes at different separation distances) to the air/lead-glass ratio indicates which of the sixty-one spectra is used to modify the air spectrum. The dark current (intensity) spectrum is subtracted from both the lead-glass sample and modified air reference spectrum before taking the ratio which is defined as the transmittance for the block. A sample transmission spectrum is shown in figure 3.7. The method forces the transmittance at 600 nm to be 100% for the lead-glass.

A.3 Discussion

The HP8451A transmittance measurement is good to better than 1%. If a lead-glass block is oriented the same each time (when the intensity is greatest for all of the wavelengths), then the transmission spectrum is reproducible with less than 2% variation for all wavelengths. The modifications allow for reproducible results and have been used to compare many of the central calorimeter's lead-glass blocks. The characteristics of the the lead-glass blocks' spectra are discussed in section 3.2.2