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Abstract

Laboratory ultraviolet (UV) chambers are widely used to obtain weathering data for a wide range of commercial polymer products including coatings, textiles, elastomers, plastics and polymeric composites. Although numerous improvements have been made in the design of UV chambers over the last 80 years, the reproducibility of the exposure results from these chambers have remained elusive. This lack of reproducibility is attributed to systematic errors in their design, operation, and control which prevent direct comparisons of the performance of materials exposed in the same environment, comparisons of the performance of the same material exposed in different laboratories, and the comparison of field and laboratory results. This paper describes an innovative UV chamber design based on integrating sphere technology that greatly reduces the magnitude of these errors, as well as provides additional experimental capabilities.

Introduction

Laboratory ultraviolet chambers (UV chambers) are the primary means for generating weathering data for a wide range of commercial products including coatings, elastomers, plastics and polymeric composites. (1). Over the years, numerous technical improvements have been made in the design, construction, and control of these UV chambers. However, the reproducibility of exposure results obtained from these chambers has remained elusive (2,3,4,5,6).

The reproducibility of UV exposure results are affected not only by variability in the material itself and its response to exposure, but also by *experimental* and *systematic* errors. Experimental errors and material variability, which are present in all experiments, are random in nature and can be compensated for through the use of proper experimental designs.

Systematic errors, on the other hand, are uncompensated, non-random sources of error which can strongly bias experimental results. Common sources of systematic errors associated with existing UV chambers include unnaturally high specimen temperatures, non-uniform spatial irradiance over the surface of a specimen, and temporal changes in exposure conditions. These errors can be minimized by standardizing test procedures, making changes in existing exposure equipment, or circumventing them through the use of alternate UV chamber designs. In this paper, an innovative UV chamber based on integrating sphere technology is presented, which has the potential to mitigate a number of these systematic errors. In addition, it will be shown that the use of an integrating sphere-based UV chamber allows for added capabilities and greater flexibility in experimental design.

Current Laboratory UV Weathering Instrumentation

Commercially available UV chambers began to appear circa 1920. Atlas Electric Devices[†] introduced its carbon arc “Fade-O-Meter” in 1918; several years later, Nelson (7) published preliminary exposure results for a UV chamber using a mercury arc lamp in 1922, and Buttolph

[†] Certain trade names and company products are mentioned in the text or identified in an illustration in order to adequately specify the experimental procedure and equipment used. In no case does such an identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the products are necessarily the best available for the purpose.

(8) patented several modifications to Nelson's UV chamber in 1924. UV chambers containing fluorescent lamps were introduced at a later date.

Numerous modifications in the early UV chamber designs have been made over the last 80 years. These modifications have been aimed at improving the repeatability and reproducibility of exposure results and include the development of UV sources with spectral distribution that more closely approximates the terrestrial solar spectrum (9,10,11,12), the employment of feedback-control devices for monitoring and controlling temporal changes in the radiant power, thus improving temporal stability of the source (13,14,15), and the introduction of three-tier exposure racks in xenon arc UV chambers, which served to improve the spatial irradiance uniformity over a specimen and between specimens. These changes have greatly reduced the variability in exposure results, but they have not fully resolved issues related to the lack of reproducibility (5,6).

Factors Affecting Reproducibility and Repeatability of Current UV Chambers

Common sources of systematic error found in current UV chambers include unnatural exposure conditions (e.g., high specimen temperatures, non-terrestrial wavelengths), non-uniform irradiance over the dimensions of a specimen and among specimens, the inability to accurately and precisely measure ultraviolet radiation dose, and temporal changes in exposure conditions. Of these sources of error, unnatural exposure conditions and spatial irradiance non-uniformity have been the focus of NIST researchers as those which could be mitigated by the use of a novel integrating sphere-based UV chamber.

Unnatural Exposure Conditions

The high current densities used in many arc lamps cause them to emit a substantial amount of energy in the visible and infrared regions (16,17). In a typical UV chamber, the light source is situated in close proximity to the specimens, thus it has been observed that exposed specimen temperatures can approach 60 °C (18,19). These temperature levels are generally not consistently reached in most outdoor applications.

In the current commercial technologies, improved temperature control can be achieved by removing the primary source of thermal energy, visible and infrared radiation, while maintaining

the photolytically effective ultraviolet radiation. This can be accomplished by introducing a heat controlling optical element (e.g., a dichroic mirror) between the light source and the specimen.

“Unnatural” chemistry in materials may also be induced by ultraviolet radiation with a wavelength less than 290 nm, which does not reach the earth’s surface (20,21). Efforts have been made by UV chamber manufacturers to eliminate these wavelengths by carefully researching light sources and utilizing cut-on filters. Although not a perfect match, certain grades of fluorescent lamps or xenon arc lamps equipped with borosilicate/borosilicate or CIRA/sodalime filters appear to be a close approximation to the solar spectrum (12).

Spatial Irradiance Uniformity

Ensuring spatial irradiance uniformity over the dimensions of a specimen and between specimens is a prime consideration in designing any optical system. Spatial uniformity is needed to determine that each specimen receives the same UV dosage. Spectral UV radiation dosage must be known in order to compare the performance of materials exposed in the laboratory and those exposed in the field (22,23).

Spatial irradiance uniformity is difficult to attain in current UV chambers due, in part, to the large surface area over which uniformity must be controlled. Factors affecting irradiance uniformity include non-uniform emittance from the lamps, reflectance from the specimen and walls of the chamber, and physical limitations imposed by the optical system (e.g., the geometry of the light source, dimensions of the specimens, and distance between the light source and the specimens).

Integrating Sphere Theory

The theory of integrating spheres, as well as their uses in a wide variety of applications, is well established (24,25,26). An integrating sphere is a hollow spherical chamber with an inner surface coated with a diffusely reflecting, or *Lambertian*, coating. Light entering an integrating sphere undergoes multiple diffuse reflections at the interior surface, resulting in a uniform field of light within the sphere. This collected light can then serve as a means of measurement or as a source of uniform illumination. This latter function will be utilized in the novel UV weathering device. Integrating sphere theory has its origin in the theory of radiation exchange between diffuse surfaces (27). Although the general theory can be complex, the symmetry of the sphere

simplifies the analysis. Consider the exchange of radiation between two differential elements of diffuse Lambertian surfaces A_1 and A_2 , with areas dA_1 and dA_2 as shown in Figure 1a. The fraction of the total flux leaving A_1 (Φ_{A_1}) and arriving at A_2 (Φ_{A_2}) is given by:

$$\frac{\Phi_{A_2}}{\Phi_{A_1}} = dF_{1-2} = \frac{\cos \theta_1 \cos \theta_2}{\pi S^2} dA_2 \quad (1)$$

where dF_{1-2} is known as the exchange factor. If these two surface elements are contained inside a diffuse sphere surface, and $S = 2R \cos \theta_1 = 2R \cos \theta_2$, as shown in Figure 1(b), then Equation 1 becomes:

$$dF_{1-2} = \frac{dA_2}{4\pi R^2} \quad (2)$$

Equation 2 is independent of the locations of the two elements and as well as the distance between the elements. This result is significant because it states that the fraction of flux received by A_2 is the same for every other point on the sphere surface.

If the differential areas dA_1 and dA_2 become finite areas A_1 and A_2 , then Equation 2 becomes:

$$dF_{1-2} = \frac{1}{4\pi R^2} \int_{A_2} dA_2 = \frac{A_2}{4\pi R^2} \quad (3)$$

Equation 3 is also independent of dA_1 , allowing it to be expressed as:

$$dF_{1-2} = \frac{A_2}{4\pi R^2} = \frac{A_2}{A_s} \quad (4)$$

where A_s is the total surface area of the sphere. Thus, the fraction of energy received by A_2 is proportional to the fraction of surface area that it takes up within the sphere.

The physical significance of the previous analysis is that every point on a sphere is equally illuminated by reflections from every other point. Theoretically, therefore, not only should the output intensity be uniform across the plane of an exit port, but every exit port should have the same spectral output as every other exit port. This property of integrating spheres will be exploited to produce an artificial UV weathering device with improved spatial irradiance uniformity. Thus, the use of an integrating sphere as a uniform UV radiation source can potentially resolve one of the major sources of systematic errors in current UV chamber design;

that is, non-uniform irradiance across the dimensions of a specimen and from specimen to specimen.

The throughput, at a given port, of an integrating sphere with multiple ports is given by:

$$\frac{\Phi_o}{\Phi_i} = \frac{\rho f_e}{1 - \rho(1 - f_{\text{tot}})} \quad (5)$$

where Φ_i is the input flux, Φ_o is the output flux, ρ is the average sphere wall reflectance, f_e is the fraction of the sphere surface area taken up by the exit port of interest, and f_{tot} is the total fraction of the sphere surface area taken up by all of the exit ports. This equation assumes that no portion of the input flux is directly incident on any of the exit ports. It is generally recommended that f_{tot} be less than 5% if sphere output uniformity is critical. If the input flux is known, the output flux of an actual sphere can be calculated via Equation 5.

In order for the above-mentioned equations to be correct and for an integrating sphere to be used successfully as an optical device, it is critical that the interior sphere surface scatter light in a Lambertian fashion over the wavelength region of interest. If the scattering is non-Lambertian, then the basic assumptions for the standard integrating sphere analysis are violated. The effects of non-Lambertian reflectance on the output of integrating spheres are presented by Hanssen (28).

In the range of photolytically active wavelengths for polymeric materials, namely 290 nm to approximately 400 nm, materials with the highest Lambertian reflectance are based on barium sulfate powder or polytetrafluoroethylene (PTFE). The reflectance of pressed PTFE powder has been studied by Weidner and Hsia (29), and was measured to be > 0.98 in the region from 250 nm to 2000 nm. In recent years, a solid, machinable PTFE material has been developed having the high reflectance of the powder, but with greater durability and resistance to temperature, moisture and corrosive chemicals. PTFE maintains its reflectance indefinitely under normal laboratory conditions if not contaminated with organic substances and does not need to be repacked or recoated (30). In the event that reflectance does decrease over time due to surface contamination, the material can be sanded or vacuum baked (31) to regenerate its original reflectance.

Integrating Sphere Based UV Chamber

Integrating Sphere

NIST researchers have developed a novel UV chamber based on a 2 m diameter integrating sphere and equipped with a high intensity UV light source, shown in Figure 2. The sphere is constructed from modular panels, allowing individual panels to be removed as needed for modification or repair. The exterior shell of the sphere is aluminum, and the interior surface is lined with PTFE panels. The sphere currently contains thirty-two 11.2 cm diameter ports, and a 61 cm diameter top port to accommodate the UV light source.

UV Light Source

The lamp system partnered with the 2 m sphere is a microwave-powered, electrodeless lamp system with an output that is rich in the region between 290 nm and 400 nm. A total of six lamps are utilized, symmetrically arranged around the top port of the sphere, with a calculated total output flux of 8400 W. A multiple lamp system, in contrast to a single lamp, allows the response of a material to various irradiance levels to be evaluated, thus providing opportunities to test the law of reciprocity. Because the light source is located outside the sphere, it is easily accessible for adjustments and repair. Dichroic reflectors in the lamp housings remove 80 % to 90 % of the infrared and visible output from the lamps, and a cut-on filter is also installed in the optical path in order to remove radiation below 295 nm.

Specimen Chambers

To study the durability of materials used in outdoor applications, it would be advantageous to uniformly irradiate specimens under a variety of exposure conditions. This could be accomplished by equipping each port with a specimen chamber in which temperature, relative humidity, mechanical loads, UV flux, and other factors can be independently controlled.

Because each chamber is independent of the others, a multiplicity of environmental conditions can be evaluated in a given experiment. While the UV irradiance would be identical between the ports, narrow band-pass filters or neutral density filters can be installed at the exit port to study the effect of a narrow wavelength region or to adjust the intensity, respectively. For instance, it would be possible to expose specimens at one exit port to 25 °C, 30 % relative humidity, polychromatic light; whereas, at another exit port, specimens could be exposed to 50

°C, 95 % relative humidity, and 290 nm radiation. Because the specimens at a given port are independent of all other specimens, and are also physically separated from the sphere and the lamp system, individual specimens can be entered into or taken out of test without disturbing the remaining specimens.

The capability to apply mechanical stresses, either cyclic or static, to the specimens while they are undergoing UV exposure can also be achieved, as shown in Figure 3. Other unique exposure environments can also be created, including temperature cycling (including freeze/thaw), humidity cycling, and acid rain.

Conveyance and collimation of the highly uniform radiation from the sphere exit port to the above-described specimen chambers is accomplished with minimal loss of uniformity and intensity with the use of non-imaging optical devices. Such non-imaging optical devices are also referred to as compound parabolic concentrators, Winston cones, and cone concentrators, and are considered to be more efficient than conventional image-forming optics in concentrating and collecting light. These devices date back to the 1960's and were once used for solar collection; a detailed treatment of this subject is given by Welford and Winston (32). In the application at hand, they will be used to collimate the diffuse output from the sphere exit port to a divergence angle of 20° and transfer it to the specimen surface.

Preliminary Measurements

The output spectrum for the UV light source with the dichroic reflectors and cut-on filters in place is shown in Figure 4. It can be seen that output below 290 nm is virtually non-existent. In the event that wavelengths > 400 nm are found to be critical in the photodegradation of certain materials, the dichroic reflectors can be replaced with conventional reflectors, thus retaining a greater proportion of the visible and infrared spectrum.

Spectral measurements of the UV light source output were taken as a function of time, using a UV-visible spectrometer equipped with a collection sphere. The coefficient of variation for 700 independent spectra taken over every ½ h for approximately 500 h was found to be on the order of 1 %. These measurements are routinely taken during the time that the UV light source is in operation, and are used to quantitatively assess the quality of the UV output, which may be affected by changes not only in the lamps themselves, but in the transmittance or reflectance of

any material in the optical path. This includes the reflectance of the dichroic elements, the sphere wall, and the interior of the cone concentrator, and the transmittance of the cut-on filter.

UV-visible measurements were taken at 17 points (illustrated in Figure 5a) at the output end of the cone concentrators with all 6 lamps running at 100 % power. A representative uniformity profile is shown in Figure 5b, revealing that the uniformity over the measured points within the exposure area is 92 % or better. Also, Figure 6 shows that UV-visible spectra taken at the center point of 14 different cone concentrators are virtually non-distinguishable. The data from this series of measurements leads to the conclusion that both the intra-port uniformity and inter-port irradiance uniformity of the integrating sphere output are high.

The sphere system is heavily instrumented with thermocouples to ensure that overheating of the sphere does not occur. Temperature profiles as a function of time at various positions in the sphere are shown in Figure 7. In general, temperatures are quite stable and do not exceed 35 °C.

Conclusions

UV chambers play an important role in comparing or predicting the performance of polymeric construction materials and determining the effect of different weathering factors on the performance of a construction material. Although significant modifications have been made in current UV chamber designs, controlling the systematic errors and thus the reproducibility of exposure experiments using these chambers has remained elusive. An integrating sphere UV chamber design is being developed which appears to be capable of mitigating known sources of systematic errors.

The improvements offered by an integrating sphere UV chamber over conventional weathering instrumentation are summarized as follows:

1. Spatial irradiance uniformity.

As a uniform radiation source, an integrating sphere is capable of minimizing errors due to non-uniform irradiance over the dimensions of a specimen and from specimen-to-specimen. Spatial irradiance uniformity does not depend on the light source, the age of the light source, or batch to batch variability, but, instead, it is controlled by the physics of integrating spheres.

2. Removal of radiation below 290 nm.

By positioning a cut-on filter after the dichroic mirror, radiation below 290 nm can be removed from the radiant flux. Moreover, interference or cut-off filters can be positioned in front of each specimen so that each specimen can be uniquely irradiated using any combination of wavelengths.

3. Removal of visible and infrared radiation from the radiant flux

High specimen exposure temperatures can be minimized by removing most of the visible and infrared portions of the radiant energy flux emitted by the light source prior to entering the sphere. By removing most of the visible and infrared radiation emitted by the light, the temperature within the chamber can easily be maintained at slightly above room temperature.

4. Simplicity in design and easy accessibility to the light source, exposure cells and specimens.

The light source, exposure cells, and specimens are located on the exterior of the integrating sphere and, thus, they are readily accessible even while an experiment is in progress.

5. Experimental Flexibility

Finally, use of an integrating sphere provides the opportunity to simultaneously and independently expose a multiplicity of specimens each to its own exposure environment. This can be achieved by positioning specimens in individual exposure cells and uniformly irradiating the specimens by projecting the output from the exit ports with the use of non-imaging optical devices. The environmental and operating conditions within each exposure cell can be uniquely selected.

Acknowledgments

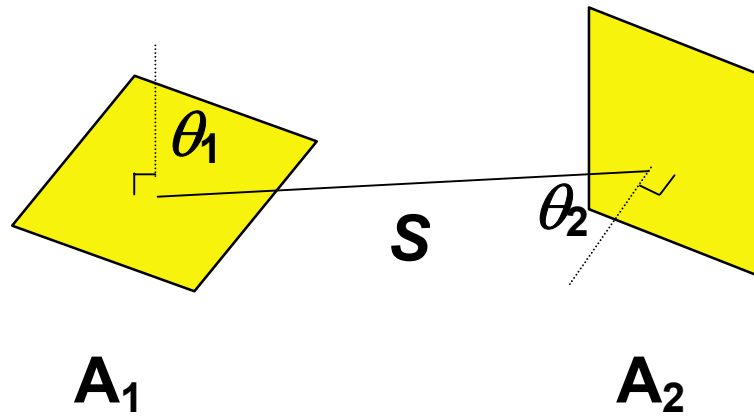
This project was conducted under the auspices of the NIST Coatings Service Life Prediction Consortium, which includes as its industrial members Atlas Electric Devices, Dow Chemical, PPG, Akzo Nobel, Millennium, Atofina and Sherwin Williams. Federal agency participants

include the Federal Highway Administration, Forest Products Laboratory, National Renewable Energy Laboratory, and the Air Force Research Laboratory.

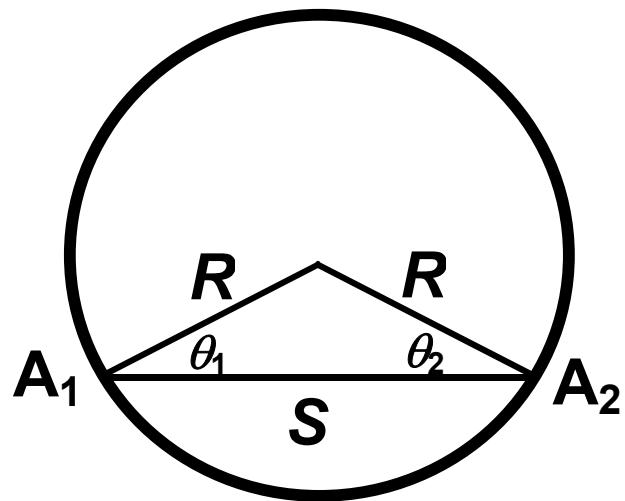
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(a)



(b)

Figure 1: (a) Exchange of radiation between two diffuse surface elements, and (b) Exchange of radiation in a spherical enclosure.

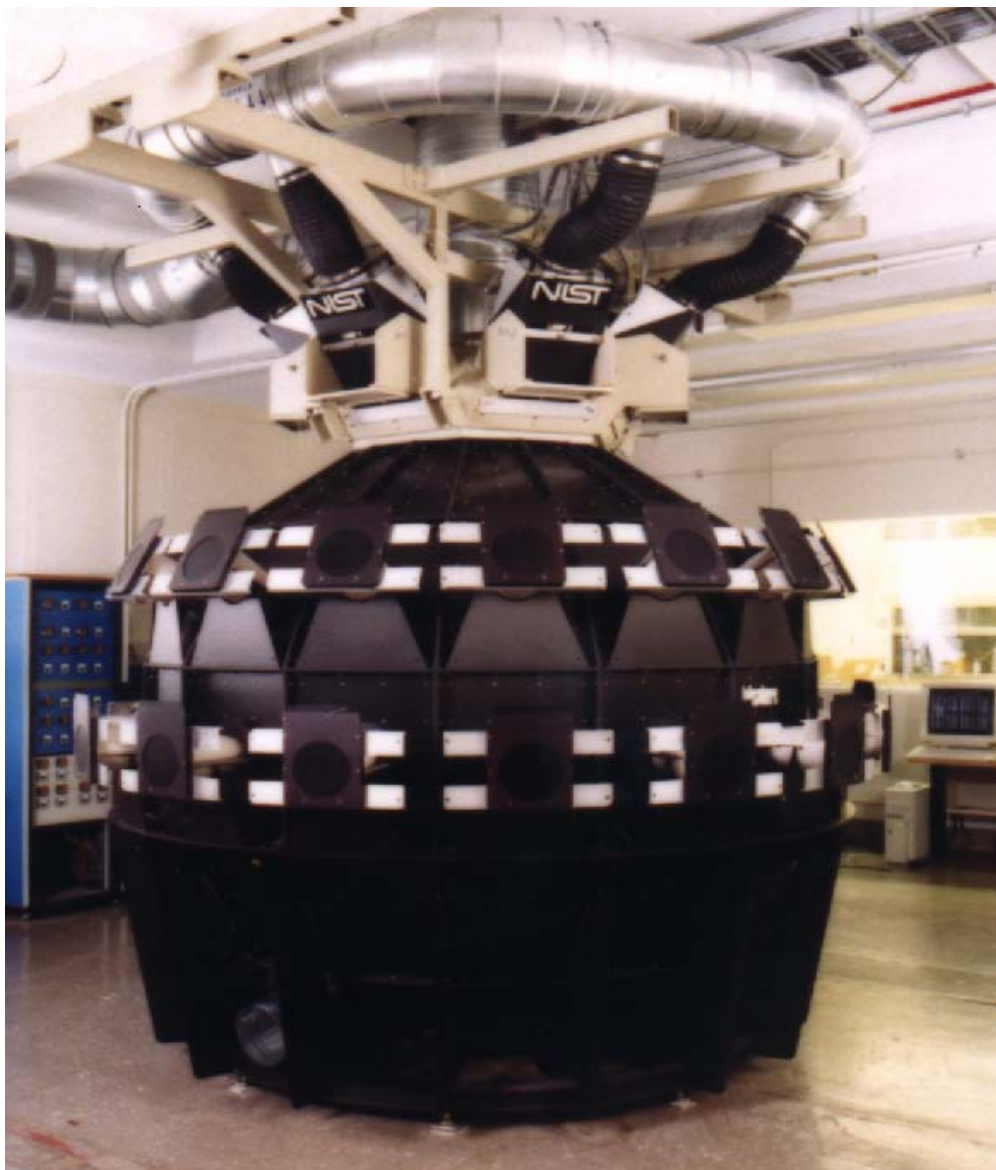


Figure 2: NIST 2 m integrating sphere, shown with UV light source.

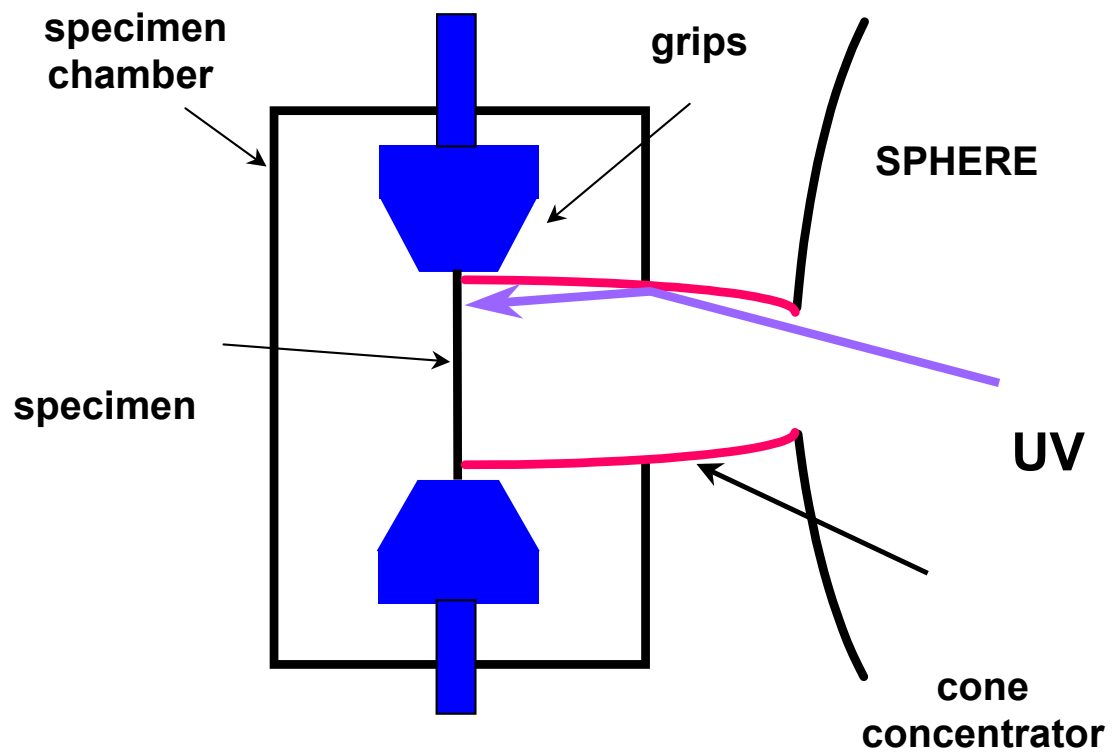


Figure 3: Proposed specimen chamber with capability for specimen loading.

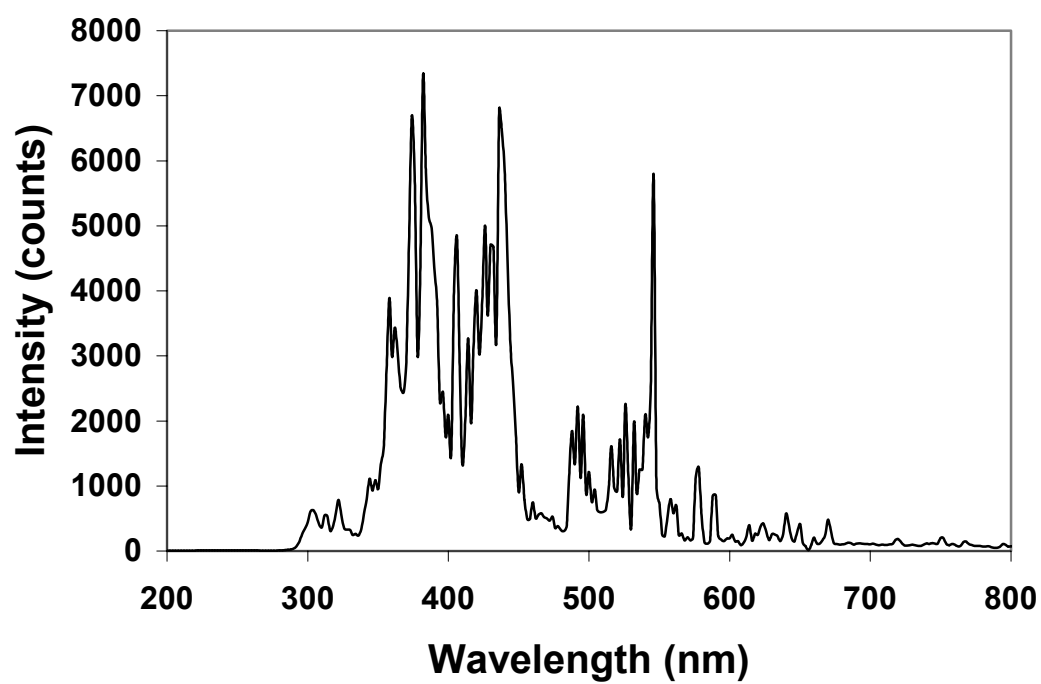
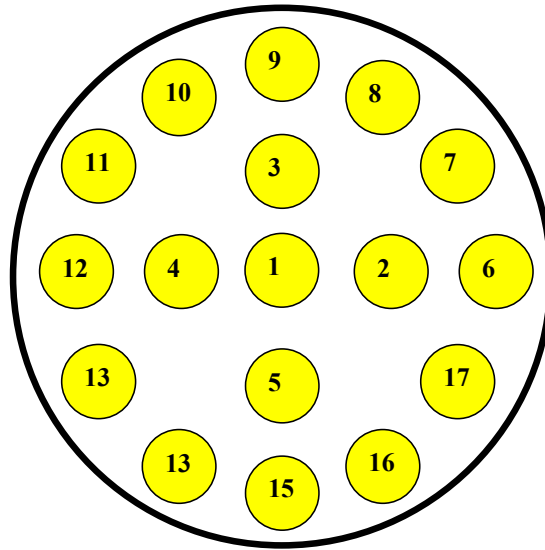
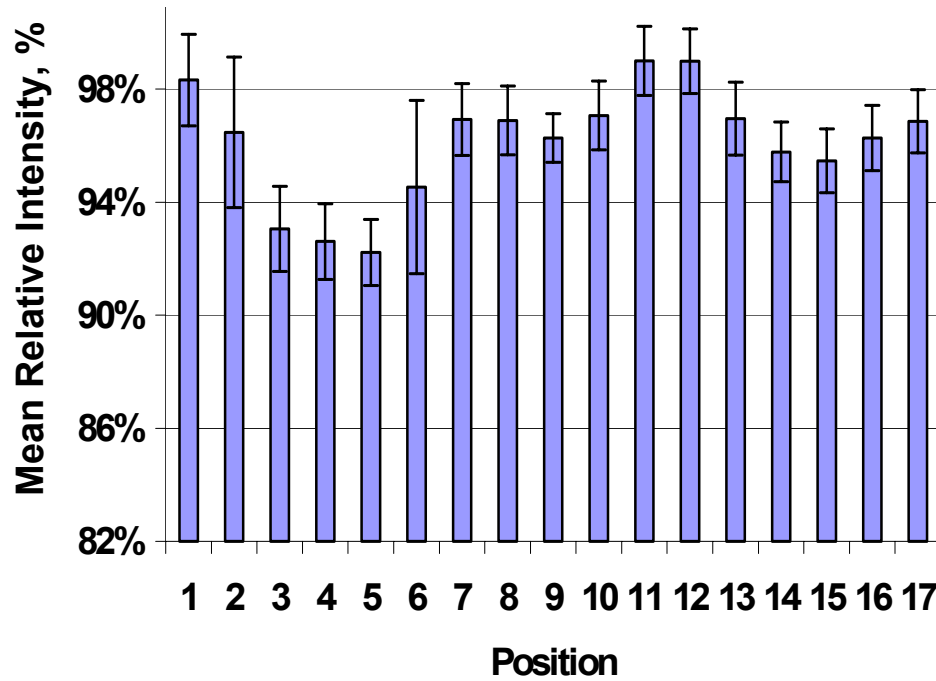


Figure 4: Output from UV light source equipped with dichroic reflectors and cut-on filter.



(a)



(b)

Figure 5: (a) Position of measurement point at end of cone concentrator, and (b) Uniformity of sphere output at exit end of cone concentrator as a function of position. Error bars represent \pm one standard deviation.

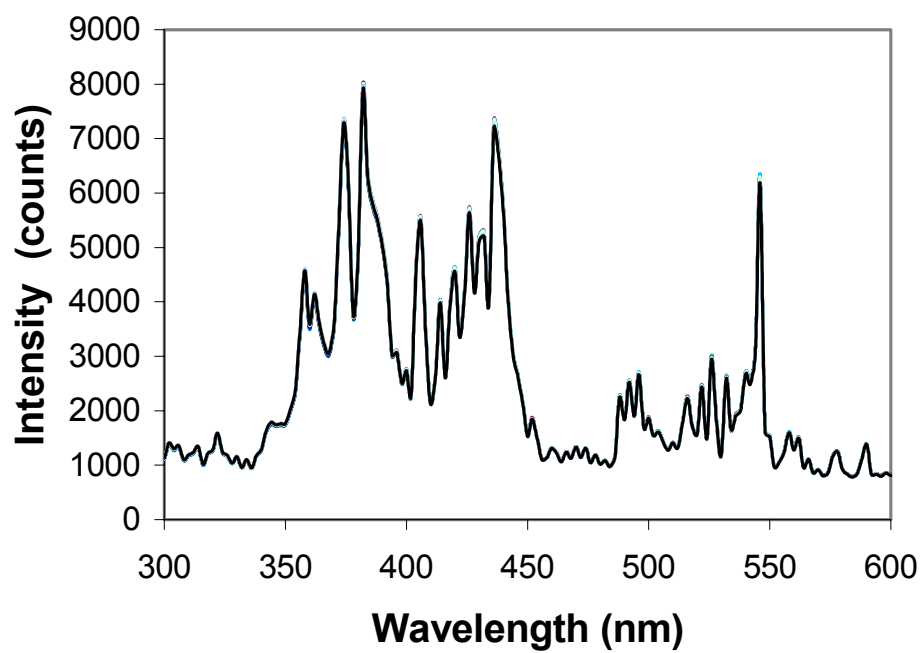


Figure 6: Sphere output at center points of 14 non-equatorial ports, superimposed.

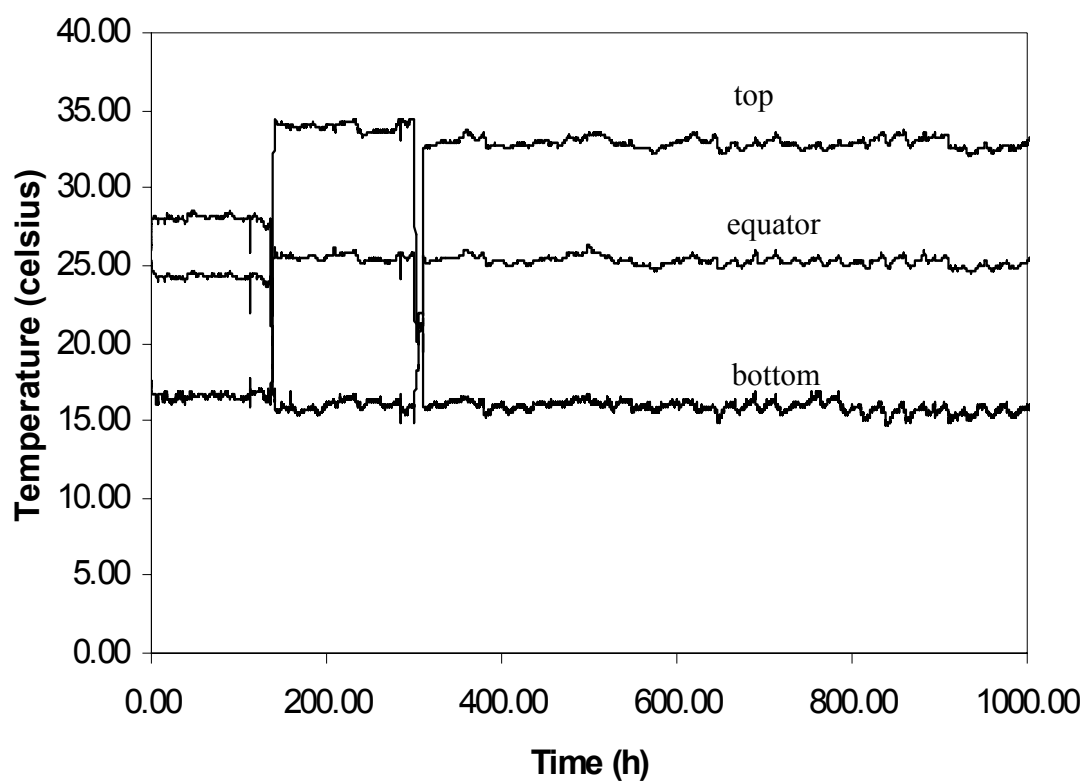


Figure 7: Temperature profiles in integrating sphere.

Dr. Joannie Chin is a Materials Research Engineer at the National Institute of Standards and Technology (NIST). She received her B.S. in Polymer Science from Case Western Reserve University, and Ph.D. in Materials Engineering Science from Virginia Polytechnic Institute and State

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Mr. Eric Byrd is a Research Chemist at the National Institute of Standards and Technology in Gaithersburg, MD. Mr. Byrd received his B.S. degree in 1980 from Shepherd College, Shepherdstown, West Virginia, and an M.S. degree from American University, Washington DC. His research activities include the study of degradation of solar collector materials and protective coatings, particularly focusing upon the identification of mechanisms of degradation and the use of analytical techniques for early detection of degradation. Analytical tools used include scanning electron microscopy, optical microscopy, thermography, thermal methods of analysis, spectrophotometry, and chromatography.

Mr. Ned Embree is a senior Physical Science Technician at the National Institute of Standards and Technology. He joined NIST in 1974, working on the aluminum wire connection overheating project. Since that time, he has been the senior technician on numerous projects and programs including those related to solar energy, roofing, adhesives, and ultraviolet degradation of organic coatings. In performing his tasks, he has designed, constructed and tested many novel testing devices. In 1987, while serving as a project team member, he received an R&D 100 Award for his part in the invention of an infrared emission surface profilometer.

Dr. Jonathan Martin is Group Leader of the Polymeric Building Materials Group at the National Institute of Standards and Technology. Dr. Martin received his B.S. and M.S. degrees from Penn State, and his Ph.D. in materials science and engineering from Washington State University. He joined NIST in 1979, and assumed the responsibilities of Group Leader in 1994. Dr. Martin's major interest and major area of responsibility at NIST is the application of reliability and life testing analysis and nondestructive testing and appearance techniques to building materials.