ACCELERATED

AND OUTDOOR

DURABILITY

TESTING OF

# organic materials

KETOLA/GROSSMAN EDITORS

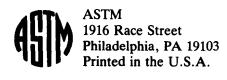


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Warren D. Ketola and Douglas Grossman, Editors

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The quality of the papers in this publication reflects not only the obvious efforts of the authors and the technical editor(s), but also the work of these peer reviewers. The ASTM Committee on Publications acknowledges with appreciation their dedication and contribution to time and effort on behalf of ASTM.

# **Foreword**

This publication, Accelerated and Outdoor Durability Testing of Organic Materials, contains papers presented at the symposium of the same name, held in Fort Lauderdale, FL on 19–20 Jan. 1993. The symposium was sponsored by ASTM Committee G-3 on Durability of Nonmetallic Materials and ASTM Subcommittee D01.27 on Accelerated Tests. Warren D. Ketola of the 3M Company in St. Paul, MN and Douglas Grossman of The Q-Panel Company in Cleveland, OH presided as symposium chairmen and are editors of the resulting publication.

# Overview

Recent advances in polymer chemistry and stabilization and increasing consumer demands for longer lasting products require use of faster, more reliable methods to evaluate durability of materials. However, there is no lack of controversy about how to properly test materials for resistance to sunlight, rain, and atmospheric acid and salt. Even when agreement is reached on a method of exposure, there is still controversy about how far one can go in drawing conclusions from the exposure data. Proper use of results from accelerated tests depends on understanding: (1) how the exposure stresses in the accelerated test match those found in field use and (2) impact of variability in results from accelerated tests and outdoor exposures.

In January of 1993, ASTM Committee G-3 on Durability of Nonmetallic Materials, in conjunction with ASTM Subcommittee D1.27 on Accelerated Tests for Paints, sponsored a symposium on Accelerated and Outdoor Testing of Organic Materials. This publication is a result of this January 1993 symposium. It covers research in characterization of exposure tests and new developments in exposure equipment and exposure test design. The following paragraphs provide a brief overview of how the results presented in the papers address important issues.

# **Characterization of Exposure Tests**

A good understanding of the primary exposure stresses to which materials are exposed during "natural weathering" is essential for explaining the mechanism and rate at which materials degrade and for providing a "target" environment for the development of appropriate laboratory accelerated tests. In order to make better use of exterior and laboratory exposure tests, it is important to fully characterize the level and consistency of the critical stresses (UV radiation, temperature, and moisture). Variation in stress level or intensity is the primary cause of variability in all exposure tests. As pointed out by Fischer, it is important to understand and characterize the variability in order to appropriately use the results from exposure tests for evaluation of new or modified products and for specifying the performance of existing products. The papers included in this section cover solar ultraviolet radiation, the effect of spectral distribution of the light source on degradation mechanism, use of radiant energy dose as a way to "time" exposures, the surface temperatures of materials subjected to exterior and laboratory accelerated weathering tests, and how glass transmission and seasonal variation in solar energy effect results for materials exposed to glass filtered sunlight.

Zerlaut provides an excellent review of current knowledge of the solar UV spectrum, how it is measured, and how and why it varies, particularly with atmospheric ozone levels. He also points out the need for monitoring solar UVB radiation and how laboratory accelerated tests that expose materials to high irradiance in the short wavelength UV-B region can significantly alter degradation mechanisms. Martin, et al. compare the methods used within the medical and agricultural communities for characterizing the effects of solar ultraviolet radiation with the measurement of broad band total UV typically used to characterize the effects of UV radiation in the coatings industry. They conclude that measuring spectral UV irradiance, material absorption spectra, and spectral quantum yield for pho-

todegradation to determine an integrated "total effective UV dosage" can provide a better approach to predicting service life of materials exposed outdoors.

Searle discusses the interaction of UV radiation on polymers and shows how degradation mechanism depends on the spectral absorption of the material and the spectral emission properties of the light source. This is described by the activation spectrum, which also depends on specimen thickness and the presence of stabilizing additives in the polymer. She also shows examples where different spectral emission bands from the same light source can produce antagonistic effects (e.g., yellowing and bleaching) in polymers. Grossman shows that variations of over 200% in degradation are possible when identical materials exposed to identical dosage of UV radiation. The use of UV radiant exposure as a method for comparing exposures is only feasible when the temperature, moisture conditions, and the spectral power distribution of the light source used for the exposure test are identical, or where it can be shown that moisture and temperature have no effect on rate of degradation.

Fischer and Ketola compare the surface temperatures of a series of colored specimens measured in typical exterior and laboratory accelerated tests, and show that the temperature separation of colored specimens in outdoor exposures depends primarily on total solar irradiance. A linear regression procedure to predict temperature of different colors from black panel measurements provided excellent agreement with actual measurements. In laboratory accelerated tests, radiant heating from the light source determines the temperature separation between dark and light colors. This becomes especially important in the "high irradiance" devices now being used, where exposures at very high irradiance can actually produce slower degradation when surface temperature of light colored specimens is very low relative to that of a black panel used to control device operation.

Fischer provides a summary of an extensive series of round-robin tests conducted by ASTM subcommittee G3.03 to develop precision statements for ASTM standards G 23, G 26, and G 53, which describe carbon arc, xenon arc, and fluorescent UV exposures, respectively. The results show that identical tests performed in different labs show poor absolute reproducibility in terms of gloss loss, but excellent reproducibility in terms of relative rankings of durability. Fischer recommends that ASTM standards for these tests warn that conclusions can only be drawn relating to relative durability, not absolute durability.

There are two papers that deal with sources of variability in exposures to glass-filtered daylight. Ketola and Robbins quantify the variability in UV transmission of single strength window glass and show how it is related to iron content. They also show how UV transmission differences can affect the rate of photodegradation for materials exposed behind glass. Crewdson and Bahadur-Singh show how degradation of materials exposed behind glass is affected by seasonal variation in solar radiation and by differences in the window glass transmission. Their paper also provides an excellent illustration of how the use of multiple replicate specimens of each material can detect subtle differences between materials. This work forms the foundation for revising and improving ASTM G 24 and other standards describing exposures to glass-filtered daylight.

# **New Test Developments**

Equipment manufacturers continue to develop and modify exposure devices in an effort to provide faster, more reliable tests. The papers in this section describe some of the new equipment available and how it is being used. Robbins provides an excellent review of the development of solar concentrating exposure devices and gives a summary of recent advances in ASTM G 90 Fresnel concentrator type solar exposures. Suits describes work done within the ASTM D 35 committee to develop accelerated tests based on xenon arc and fluorescent UV exposure for assessing the durability of geosynthetic fabrics. This paper also discusses

problems with variability and meaningful interpretation of results. Fedor and Brennan describe irradiance control and high irradiance testing in fluorescent UV devices, and illustrate how this can improve reproducibility and shorten test times with UVA fluorescent lamps. A paper by Suga and Suga and a paper by Scott describe high irradiance xenon exposures and illustrate how they have been used to rapidly evaluate color stability of fabrics.

The next paper, also by Suga and Suga, describes a method for simulating acid rain corrosion using a modified salt-spray chamber and compares results from this test to those for tests at several sites located in Japan. Finally, the paper by Murray illustrates how results from laboratory accelerated tests in exposure procedures designed according to reliability theory can be used to estimate the service life of products expected to perform for many, many years. Murray's example is for testing compact discs, but illustrates the power of this approach, which is a significant departure from the traditional methods used to evaluate "weatherability" of organic materials.

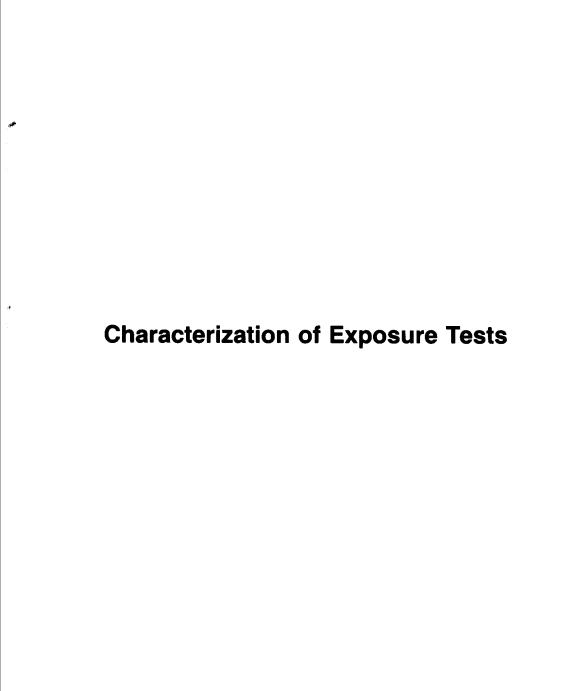
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SOLAR ULTRAVIOLET RADIATION: ASPECTS OF IMPORTANCE TO THE WEATHERING OF MATERIALS

REFERENCE: Zerlaut, G. A., "Solar Ultraviolet Radiation: Aspects of Importance to the Weathering of Materials," Accelerated and Outdoor Durability Testing of Organic Materials, ASTM STP 1202, Warren D. Ketola and Douglas Grossman, Eds., American Society for Testing and Materials, Philadelphia, 1994.

ABSTRACT: Constituents of the stratosphere and troposphere that serve to define the solar ultraviolet spectral energy distribution that is important to the outdoor deterioration of polymer-based materials are reviewed. Particular attention is devoted to the influence of ozone. . both from an historical sense, and on the basis of the latest information available on the relationship between ozone depletion and the spectral energy distribution in the so-called ozone cut-off region of the solar radiation spectrum. The significance of the historically accepted solar ultraviolet spectrum, as well as that predicted on the basis of, for example, 10 and 20% decreases in total column ozone, are discussed in terms of [1] correctly measuring ultraviolet radiation in the spectral region of importance to materials, [2] assessing the effects of solar ultraviolet radiation on such materials, and [3] simulating the spectral energy distribution of solar ultraviolet radiation in indoor exposure test scenarios.

**REYWORDS:** solar, ultraviolet, radiation, UV-B, radiometers, ozone, exposure, weathering, outdoor exposure, indoor exposure testing

## INTRODUCTION

The importance of the spectral distributions of ultraviolet radiation from natural sunlight, and by extension that of ultraviolet lamps and burners used to simulate the effects on materials of exposure to natural ultraviolet radiation, has received increased attention in recent years. This has been the result of a number of factors, not the least of which has been an increased understanding of the significance of spectra on materials degradation mechanisms and kinetics.

Two additional factors serve to greatly influence current and future application of ultraviolet technology to the exposure testing of organic materials such as plastics and polymeric coatings. One is the increased pressure placed on both outdoor and indoor exposure testing of the new classes of stabilized materials, and consequently the accurate assessment of the effects of such ultraviolet exposures. The second is

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the possibility that the current trend in ozone depletion may significantly affect the spectral power distribution (SPD) of ultraviolet radiation in the ozone-depletion region of the solar spectrum for the mid- to high-latitude regions of the world . . . an eventuality that may not be tempered by tropospheric anthropogens that might mitigate the degradative influence of short-wavelength ultraviolet radiation.

This paper attempts to address these issues, including the nature of tropospheric pollution as it affects the wavelength distribution of incoming solar ultraviolet radiation, and the fundamental response of materials to an increase in ultraviolet radiation in the ozone-depletion region. Also of importance are those measurement problems that must be addressed from the standpoint of currently accepted solar ultraviolet spectral energy distributions, and those that will result from further significant decreases in total column ozone.

### THE SUN

# Extraterrestrial Solar Radiation

The sun radiates to earth as a 5777°K full radiator (Plankian) [1]. This color temperature is defined by the sun's outermost layer, the photosphere, which is responsible for the visible radiation that reaches the earth [2]. However, the ultraviolet region having wavelengths less than 400 nm is more closely represented by a 4500°K full radiator. The lower color temperature of the sun's observed ultraviolet radiation is a result of the cooler outermost layer of the photosphere, since the ultraviolet radiation generated deep in the photosphere is absorbed prior to reaching the surface. The extraterrestrial solar spectrum is presented in the topmost curve of Fig. 1.

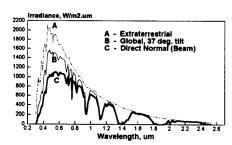


Figure 1 Solar Spectral Energy Distribution for Air Mass 1.5 vs Air Mass 0. After ASTM E 891, ASTM E 892, ISO 9845, and Frölich E Werhli (Ref. 5)

The sun's full-radiator continuum in the ultraviolet region is modified further due to absorption by metal ions such as Mg, Fe, Cr, Mn, etc., which are products of the nuclear reactions that sustain the solar processes . . and which diffuse into the photosphere. This results in absorption lines, termed Fraunhofer lines, that impart distinct character to the ultraviolet spectrum reaching the earth's atmosphere. Except for Rayleigh scattering and broad band absorption due to both stratospheric and tropospheric ozone, and anthropogenic constituents of the troposphere, the Fraunhofer-band

'n

structure (shown in Fig. 3) is spectrally unchanged in the ultraviolet radiation that reaches an earthbound observer.

# Terrestrial Solar Radiation

The currently accepted solar constant is  $1377\pm7~Wm^{-2}$  [3]. About 27% of extraterrestrial radiation is attenuated by the earth's atmosphere, resulting in what is known as a **one-sun** global irradiance of  $1000~Wm^{-2}$ ; this is also the approximate value of the integrands of several standard reference spectra that have been published in recent years.

These spectra, which are reproduced as curves B and C of Fig. 1, are taken from ASTM Standard Tables for Terrestrial Solar Spectral Irradiance at Air Mass 1.5 for a 37° Tilted Surface (E 892) and ASTM Standard Tables for Terrestrial Direct Normal Solar Spectral Irradiance for Air Mass 1.5 (E 891), respectively. It is instructive to note that these two ASTM standards have been promulgated as international standards by both the International Electrotechnical Commission (IEC) and the International Standards Organization (ISO)[4,5].

The effects of stratospheric ozone on the solar spectrum-- Ozone exists in gaseous form in measurable quantities from 0 to 50 km altitude, but is concentrated in a highly dynamic belt that varies in thickness from about 18,000 to 30,000 meters. The lower limit of this belt varies in altitude above the earth from 20,000 to 30,000 meters (20 to 30 km). The thickness varies from morning to night, from day-to-day, and from season-to-season.

I have plotted the tabular data of Leckner [6] on a log-normal chart in Fig. 2 to depict the extraordinarily strong absorption that ozone exhibits for ultraviolet radiation. The absorption coefficient is plotted in reciprocal centimeters as a function of wavelength; the three major ozone absorption regions are identified.

The Hartley region is characterized by the strongest absorption coefficient ranging from 10 reciprocal centimetres at 300 nm wavelength to 38 at 290 nm. This accounts for the fact that we normally term the solar ultraviolet cut-on wavelength as being 300 nm; also, because of the very low photon flux in this region, it was not until recently

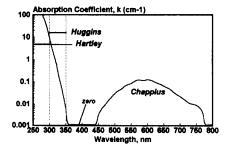


Figure 2 Ultraviolet Spectral Absorption Coefficient for Ozone. Adapted from Iqbal (Ref. 1) after Lechner (Ref. 6)

region, it was not until recently that spectroradiometers have been constructed to accurately measure the region shorter than 300-nm wavelength.

The region from 300- to 350-nm is termed the Huggins band and is the region that is most critical to normal weathering processes . . . except that large changes in the Hartley band due to stratospheric ozone depletion will significantly affect the lifetime of materials as well as both flora and fauna. Diurnal changes in the stratospheric ozone layer are easily observed by making solar radiation measurements with a good spectroradiometer set to monitor a specific wavelength in the 320-nm region. Indeed, it is in the Huggins region that spectral measurements are made to compute total column ozone (although the Chappuis region can also be used for this purpose). In this respect, if all the ozone in the stratosphere were condensed to liquid, the normal thickness would vary between 0.3 and 0.35 cm (indicating just how powerful an absorber gaseous ozone is).

A typical solar ultraviolet spectral distribution, measured at New River, AZ, is presented in Fig. 3. The drop-off in irradiance below 335-nm wavelength is due to the Huggins-band; it changes disproportion-ately throughout the day, and from day-to-day. The diurnal and seasonal

<sup>&</sup>lt;sup>2</sup>Which is discussed in greater detail in a subsequent section.

intensity of the radiation at wavelengths longer than 335 nm is much more constant and is governed primarily by air mass. Air mass is defined by the latitude, declination and sidereal hour, as well as altitude, i.e., atmospheric pressure, and at sea level is determined as the secant of the sun's zenith angle (see, for example, Fig. 7).

The Chappuis band, with an absorption of 10<sup>-1</sup> reciprocal centimetres, although weak, accounts for the flattening of the global, relative to the extraterrestrial solar spectra, in the 500- to 700-mm wavelength region, as shown in Curves A and B of Fig. 1.

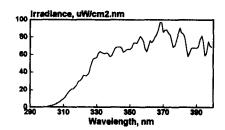


Figure 3 Solar Ultraviolet Spectral Energy Distribution (Air Mass 1.21, Hemisphrical Normal Incidence, July 7, 1989). Obtained using the DSET CMP-II Spectgroradiometer.

The ozone cut-off region-- Petit [7] in 1932 and Coblenz and Stair [8] in 1937 reported the existence of ultraviolet radiation below 295 nm. However, these early spectral measurements suffered from poor instrumentation and questionable band passes, making their data less reliable than data obtained using more modern instrumentation and calibration schemes.

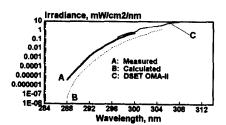


Figure 5 Ultraviolet Spectral Irradiance in the Ozone-Cutoff Region. Adapted from Koskowski et al (Ref. 10)

The computations and measurements of Bener [9] served to provide the first accurate description of the availability of ultraviolet radiation in the socalled ozone cut-off region at wavelengths shorter than 330 nm. Using Bener's graphical data, 3 a sample of which is reproduced in Fig. 4, it can be observed that there is indeed no evidence of an absolute cut-off of ultraviolet radiation at, for example, 300-nm wavelength. Rather, there is a continuum that varies in relation to total column ozone and the atmospheric pressure-adjusted air mass.

The work of Bener was first confirmed by Kostkowski and his coworkers at the National Bureau of Standards in measurements obtained with a specially constructed spectroradiometer in mid-June 1980 at Gainesville, Florida [10]. Their experimental data, which I have plotted in Fig. 5, is compared to their predicted spectrum obtained by mathematical modelling for a specific total column ozone and aerosol concentration based on the extraterrestrial spectrum (such as the one given in Fig. 1). Superimposed on this plot are typical data obtained with a diode array spectroradiometer at New River, AZ [11].

More recently, a large number of spectral measurements in the ozone cut-off region have been reported. This activity is largely in

<sup>&</sup>lt;sup>3</sup>These data represent a composite of several years of Paul Bener's measurements, observations and computations at WMO's Physikalisch-Meteorologisches Observatorium Davos; Davos, Switzerland [9].

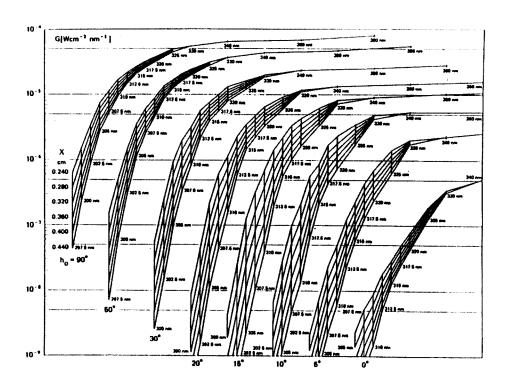


Figure 4 Spectral Distribution of Ultraviolet Solar Intensity for the Annual Mean Amount of Atmospheric Ozone at Different Latitudes and for an Assumed 50% Decrease in the Annual Mean Ozone Amount. Taken from Bener (Ref. 9)4

response to international emphasis on the measurement of ultraviolet radiation in the UV-B region<sup>5</sup> as a result of the reported decrease in total column ozone for high-latitude and polar regions of the world. Typical of these are the spectra presented in Fig. 6. These spectral data were obtained by McElroy [12] using the very sophisticated Brewer Ozone Spectroradiometer.

It is important to observe, from an examination of these and other available data, that the spectral irradiance for low zenith angles (i.e., at solar noon) is approximately 1  $\mu \text{W.cm}^{-2}.\text{nm}^{-1}$  at a wavelength of 300-nm. As a result of these observations, I have for the past several years defined the solar ultraviolet cut-on region as being the "wavelength region that lies at wavelengths less than that at which the irradiance at solar noon is 1  $\mu \text{W.cm}^{-2}.\text{nm}^{-1}$  for conditions of near-normal incidence." Hence, the solar cut-on wavelength generally can be defined as being 300 nm.

The full-size chart can be obtained from the author of this paper.

 $<sup>^5</sup> Ultraviolet$  radiation is classed as UV-A (320- to 400-nm), UV-B (290- to 320-nm) and UV-C (below 290-nm wavelength).

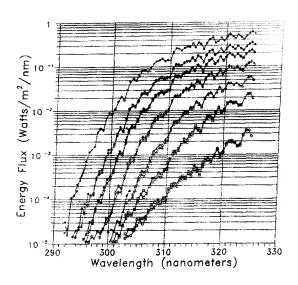


Figure 6 Global UV-B Irradiance Measured using a Brewer Spectroradiometer, Toronto, June 25, 1989. Courtesy of T. McElroy (Ref. 12)

# ATTENUATION OF SOLAR RADIATION BY THE TROPOSPHERE

# The Trophosphere and Stratosphere

The troposphere is generally considered to represent that portion of the atmosphere extending from sea level to an altitude of about 15 km in the tropics, and 10 km in the polar regions. The tropopause separates the stratosphere from the troposphere and is that region where increasing altitude no longer results in decreasing temperature, thereby giving rise to great turbulence and severe storms [13].

The stratosphere is separated from the mesosphere by the stratopause, which resides at an altitude of approximately 50 km. It is in the stratopause, and the upper stratosphere (also termed the chemosphere), where the pro-duction of ozone takes place.

# Extinction, or Attenuation of Ultraviolet Radiation

General Considerations— Ultraviolet radiation is absorbed by anthropogens such as  $NO_x$ ,  $SO_z$ , elemental carbon  $(C_e)$ , and other pollutants, such as ozone, that are generated in the troposphere. While the shorter-wavelength ultraviolet is scattered out of the beam component by gaseous molecules through Rayleigh scattering, the longer-wavelength ultraviolet is attenuated by moisture particles as a result of Mie scattering. These attenuation processes are highly variable on a temporal basis, are often site-specific with respect to their general effectiveness, and account for the differences observed in the measurement of ultraviolet

radiation recorded at various exposure sites having different climatic patterns.

To provide greater insight into these processes, and their significance to the weathering of materials, it is important to clarify the concept of the direct versus diffuse components of sunlight. Extinction, or attenuation, of sunlight, describe processes that occur largely in the direct, or beam, component as shown in Fig. 7. Until ultraviolet radiation has been scattered out of the beam component into the hemispherical

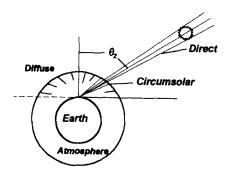


Figure 7 Direct, Diffuse and Circumsolar Components of Solar Radiation (Showing Concept of Air Mass). AM = secant  $\theta_s$ .