Light Stability Testing of Home and Personal Care Products

Q-Lab Corporation

Click here for morning presentation

Click here for afternoon presentation

Q-Lab Corporation

- Founded in 1956
- Specialize in material durability testing equipment and services

Westlake, Ohio Headquarters & Instrument Division

Bolton, England Q-Lab Europe

Shanghai, China Q-Lab China

Saarbrücken Germany, Q-Lab Germany

Q-Lab Outdoor Weathering Sites

What We Will Talk About

- Weathering Testing vs. Light Stability
- Common Light Spectra
- Natural Exposures
- Accelerated Testing
  - Xenon Arc Testing
  - Fluorescent UV Testing
- ICH Guidelines
- Best Practices and Practical Considerations

Weathering Testing

- Combination of sunlight, heat, and moisture
- Temperatures simulate realistic hot outdoor conditions
- Moisture (water spray or condensation) usually included

Light Stability Testing

- Simulation of sunlight or indoor lighting
- No moisture* or elevated temperatures
- Test temperatures often simulate typical indoor environment

*May control RH to reduce variability

Which Should I Use?

If you're not sure how your material will perform, and want to test it for every environment,

Run a Weathering Test

If your material only needs to perform in a controlled environment, or you are only interested in the effect of light on your product,

Run a Light Stability Test

Common Light Spectra

- Sunlight
  - Direct
  - Through Window Glass
- Commercial Lighting
- Home Lighting

Definitions

Irradiance

The rate at which light energy falls on a surface, per unit area; usually given as W/m^2

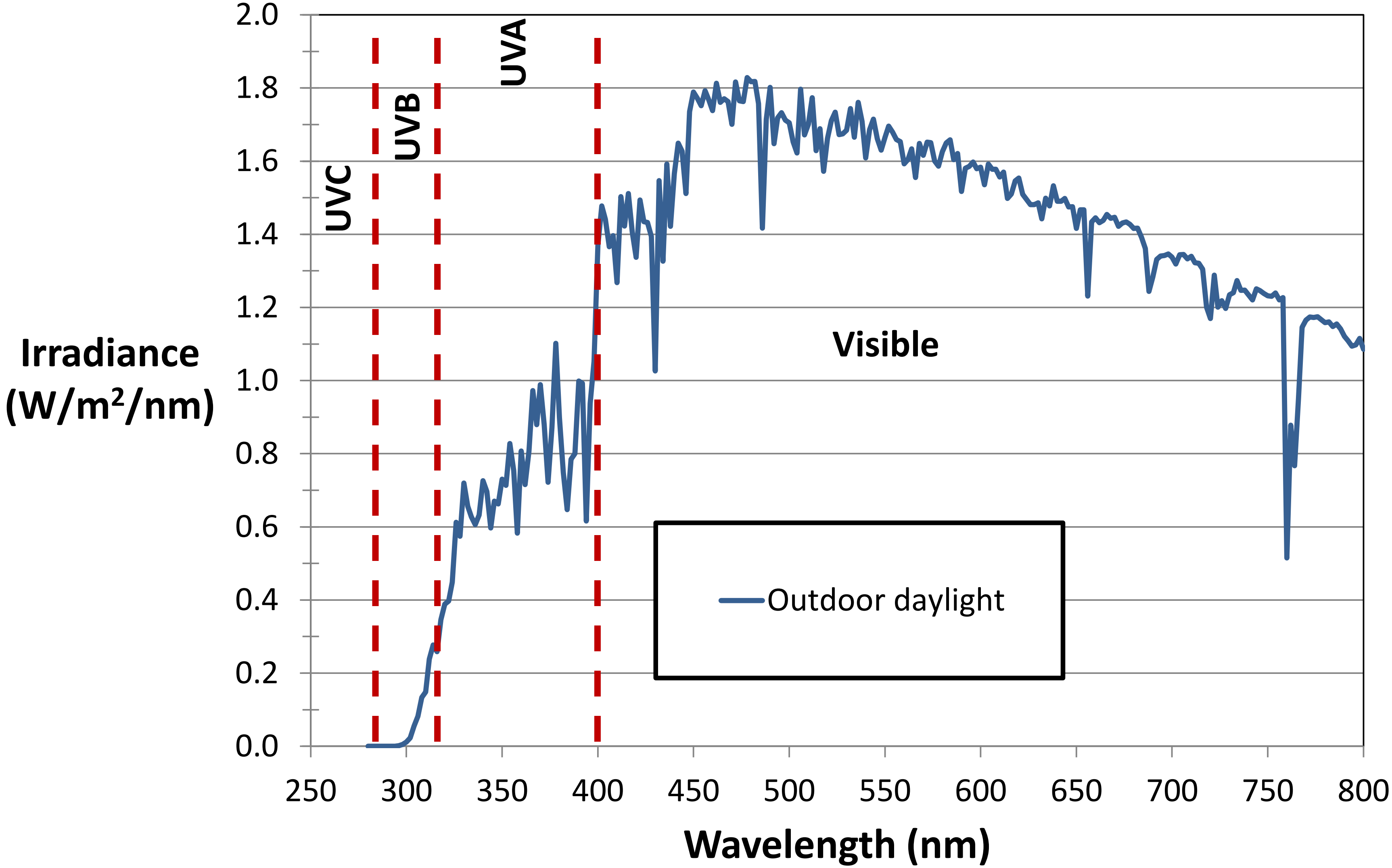
Spectral Power Distribution (SPD)

Graph of irradiance vs. wavelength

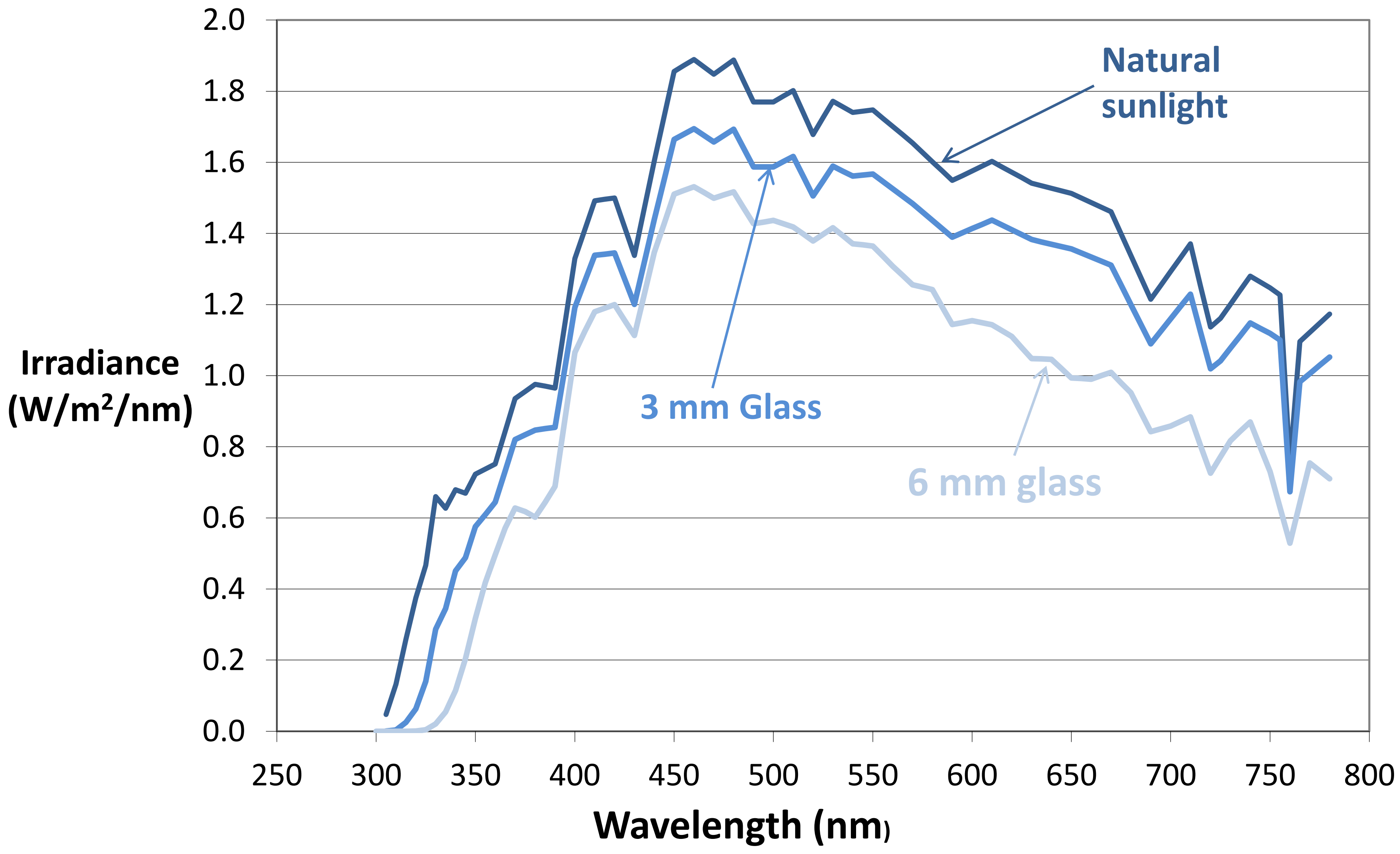
Radiant Dosage

Irradiance × Time; accumulated light energy exposure per unit area over a period of time *(more on this later)*

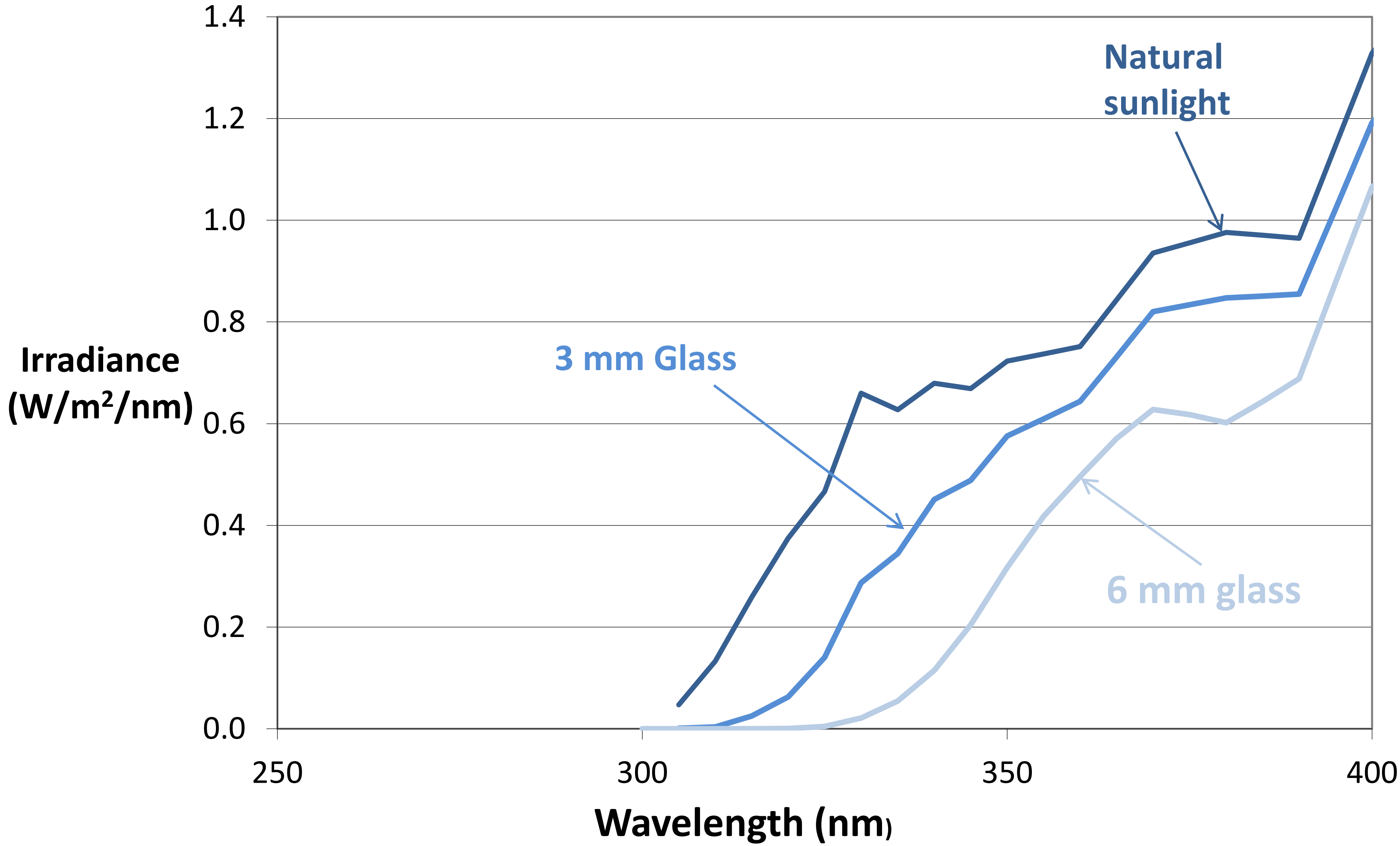
Summer Sunlight Spectrum



Sunlight through Window Glass

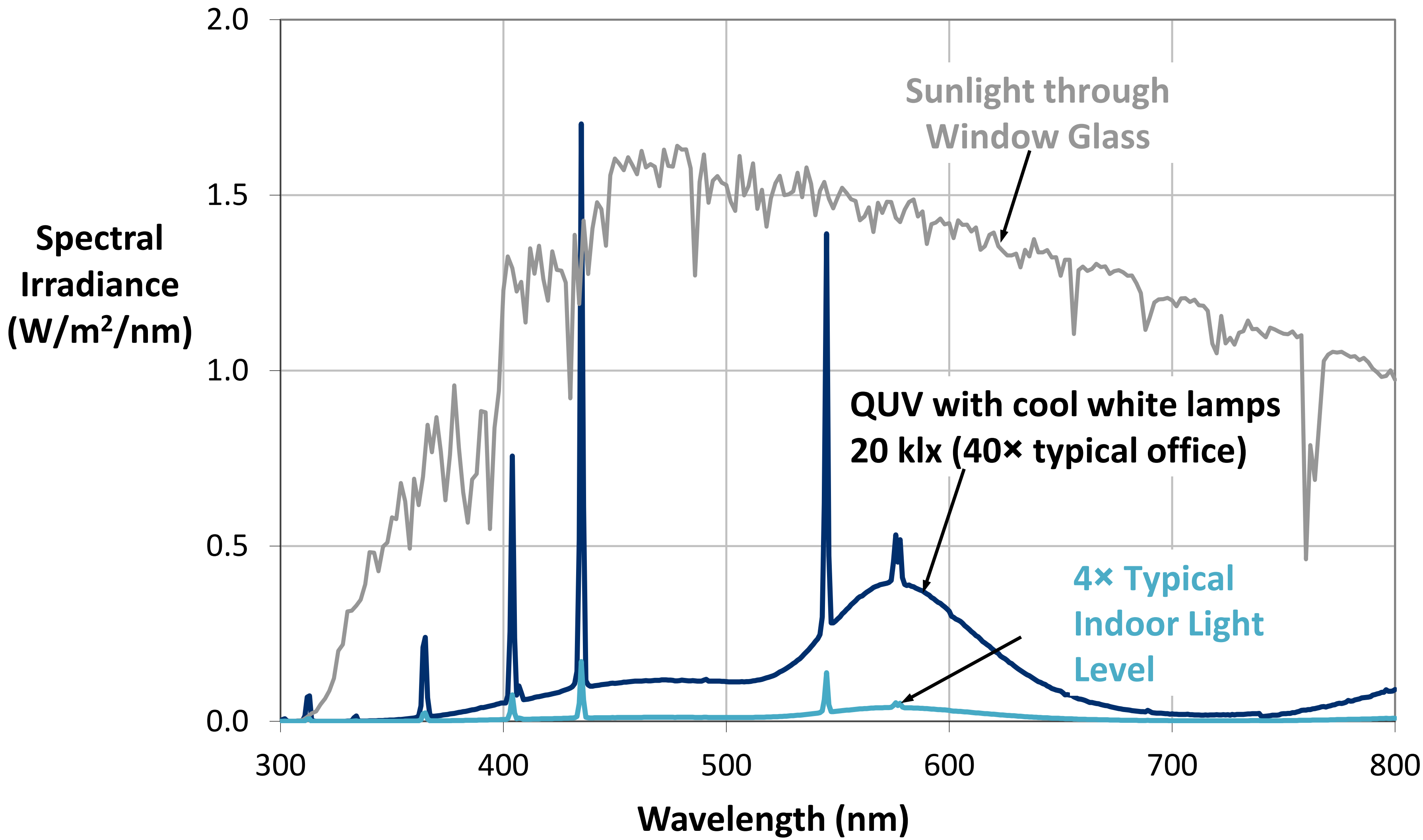


Sunlight through Window Glass

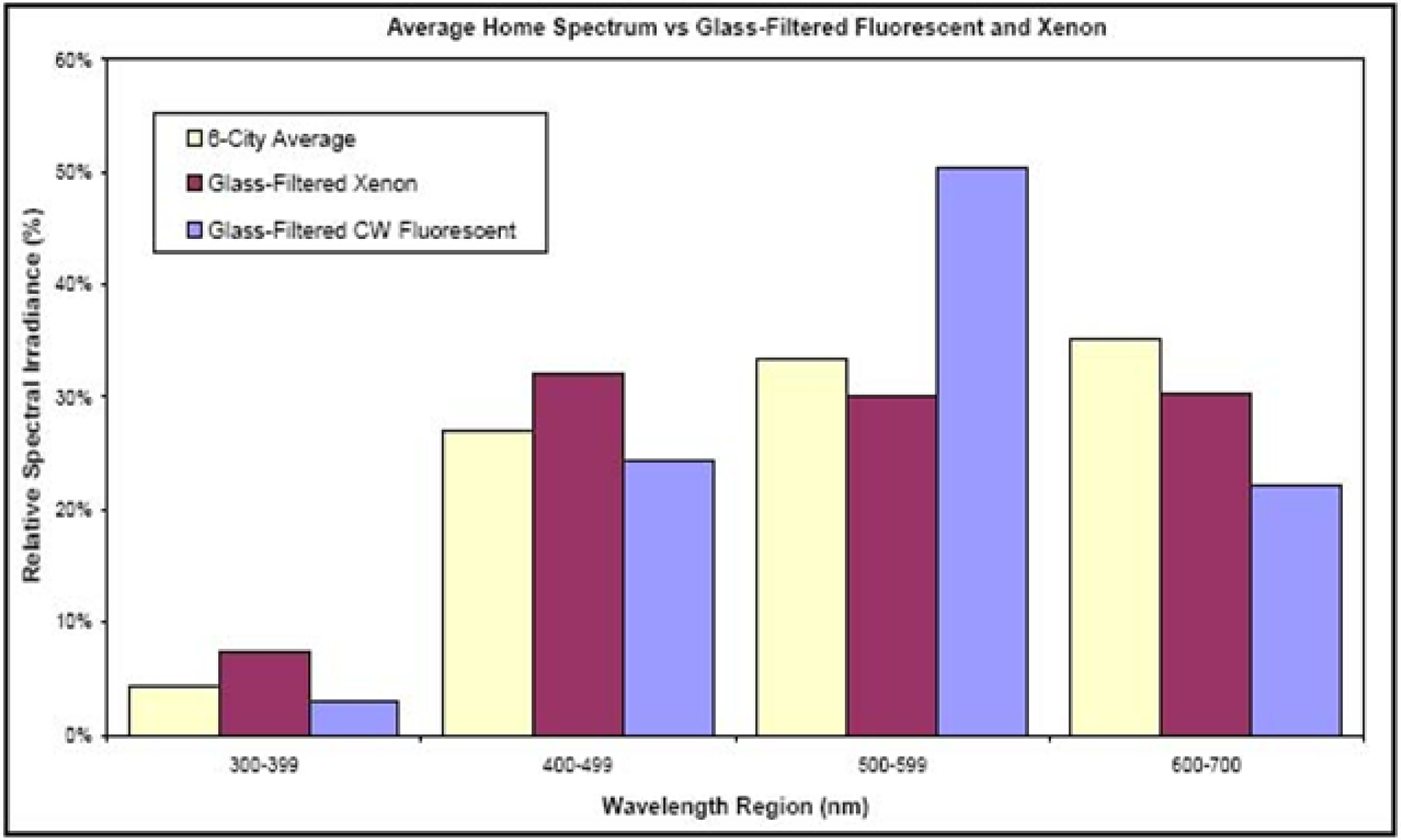


Interior Lighting

Commercial Indoor Lighting



Average Home Lighting



Even Though It Is Only 5% of Sunlight...

UV Light Causes Most Photodegradation!

Natural Exposures

Natural Exposures

In order to find out how your material will last in its service environment…

Put it in the service environment!

Natural Exposures

Benchmark Commercial Sites

South Florida, Arizona Desert

- Inexpensive
- Reliable
- Extreme environments create acceleration

At your own facility

- "Scientific Window Sill Testing"
  - Convenient
  - Easy to make frequent observations
- DIY Exposures

Natural Exposures

For many Fast Moving Consumer Goods (FMCGs), natural exposure testing at benchmark sites is very cost effective and can give you excellent data in a short amount of time

Accelerated Exposures

FMCGs can be tested for light stability in even shorted periods of time with accelerated testing, usually with xenon arc or fluorescent UV testers

Xenon Arc Testers

Xe-3-HC

Xe-1-BC

Q-SUN Xenon Test Chamber

Benefits of Xenon Arc Testing

- Realistic simulation of longwave UV and visible portion of sunlight
- Optical filters can simulate different kinds of glass
- Relative Humidity Control

Optical Filters

Daylight Filters

(exterior exposures)

Window Glass

(indoor exposures, textiles, inks, etc.)

Extended UV

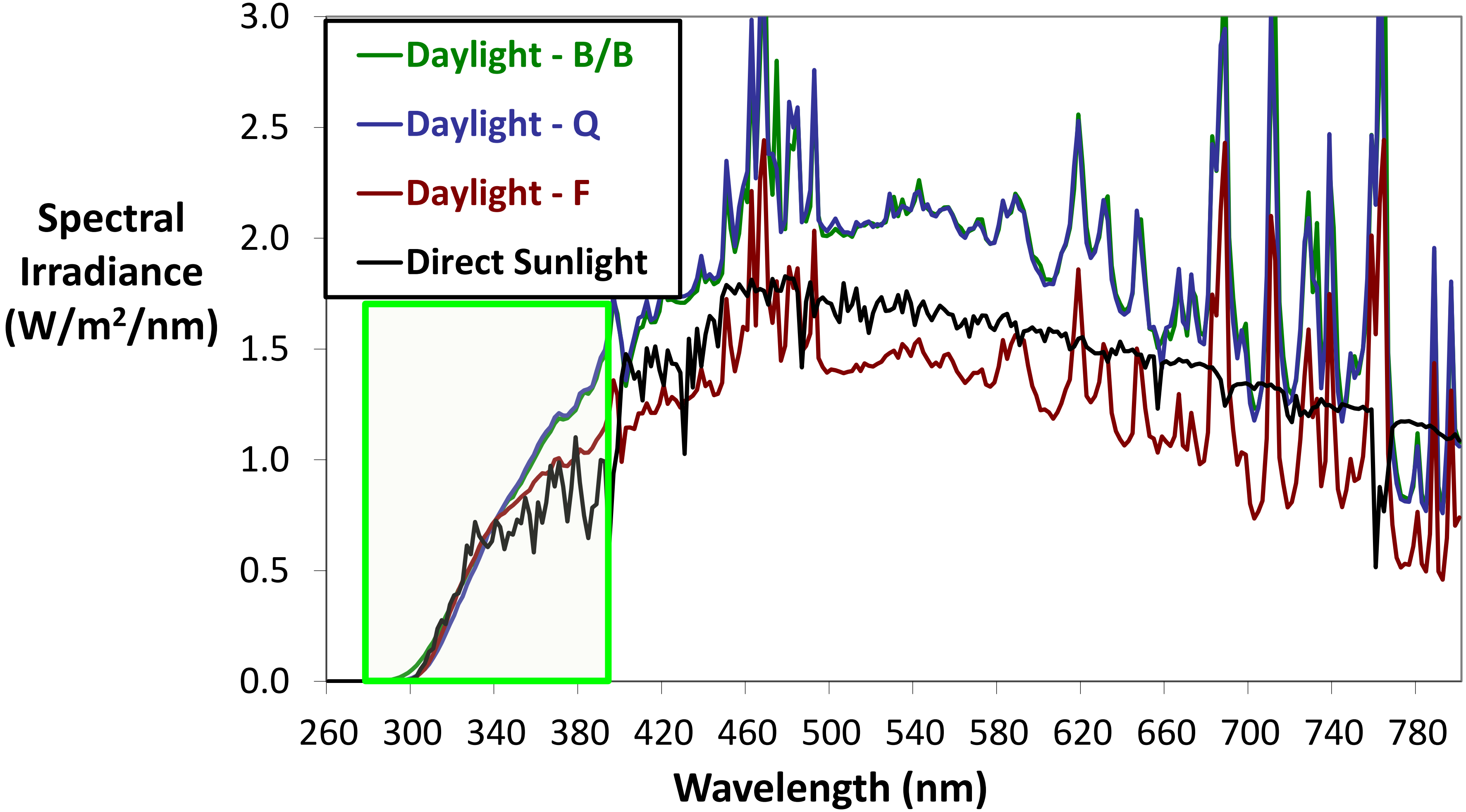
(automotive, aerospace, etc.)

Light Spectrum

- Critical factor in testing
- UV “cut-on” or “cut-off” wavelength
- Not all “Window” or “Daylight” filters are the same
- Commercial filter names create confusion

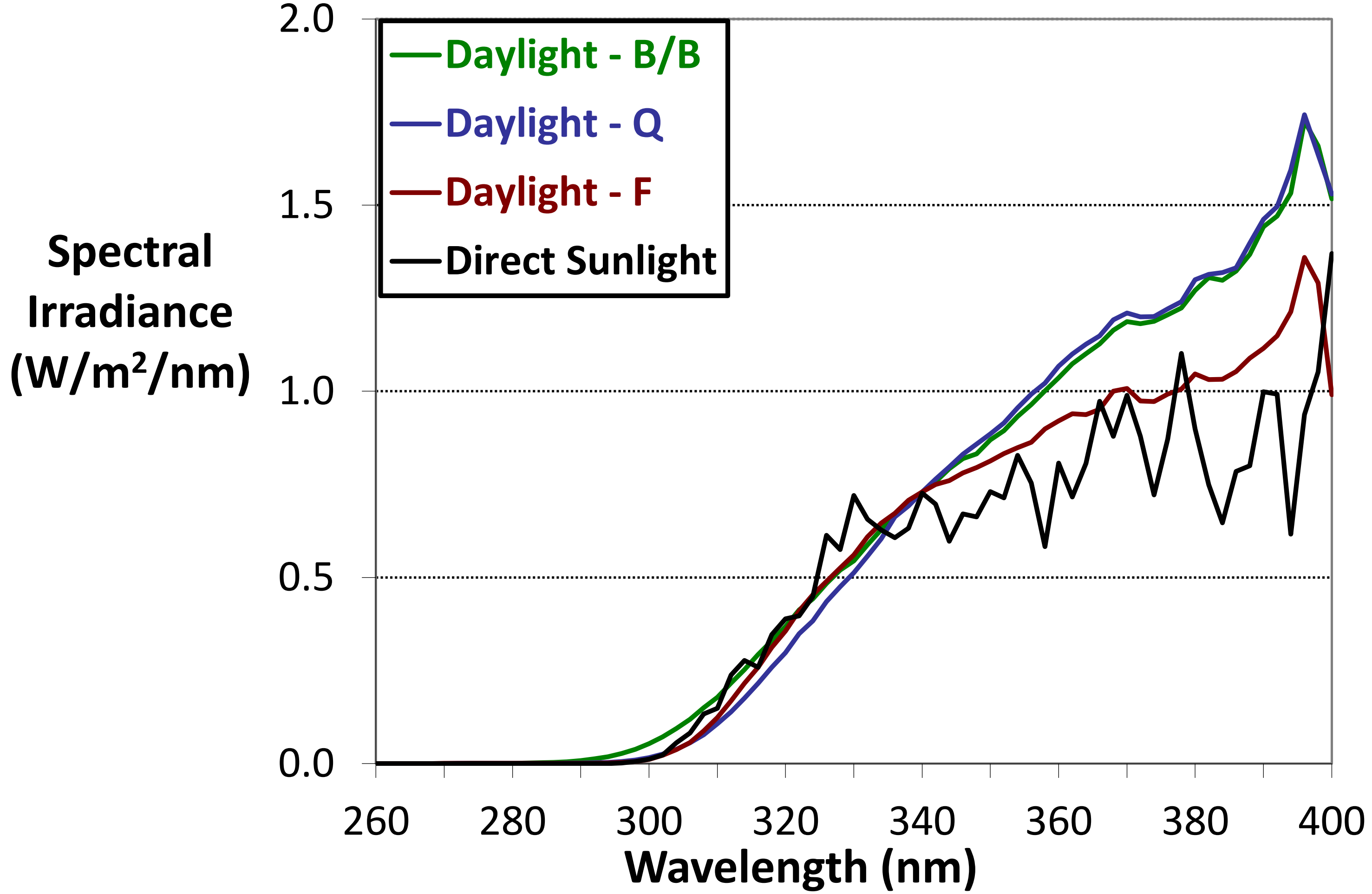
Xenon Arc with Daylight Filters

UV and Visible



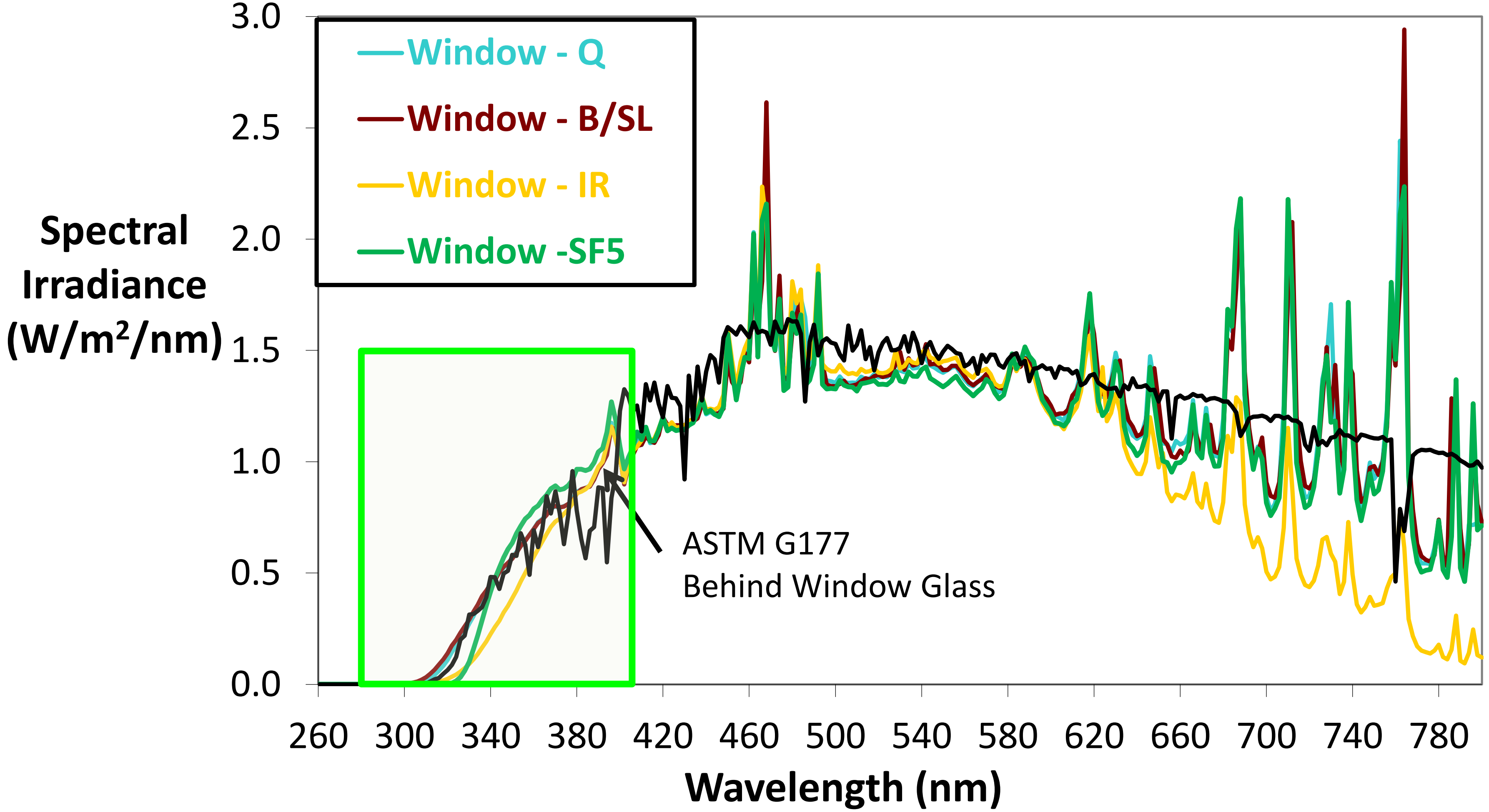
Xenon Arc with Daylight Filters

UV spectrum



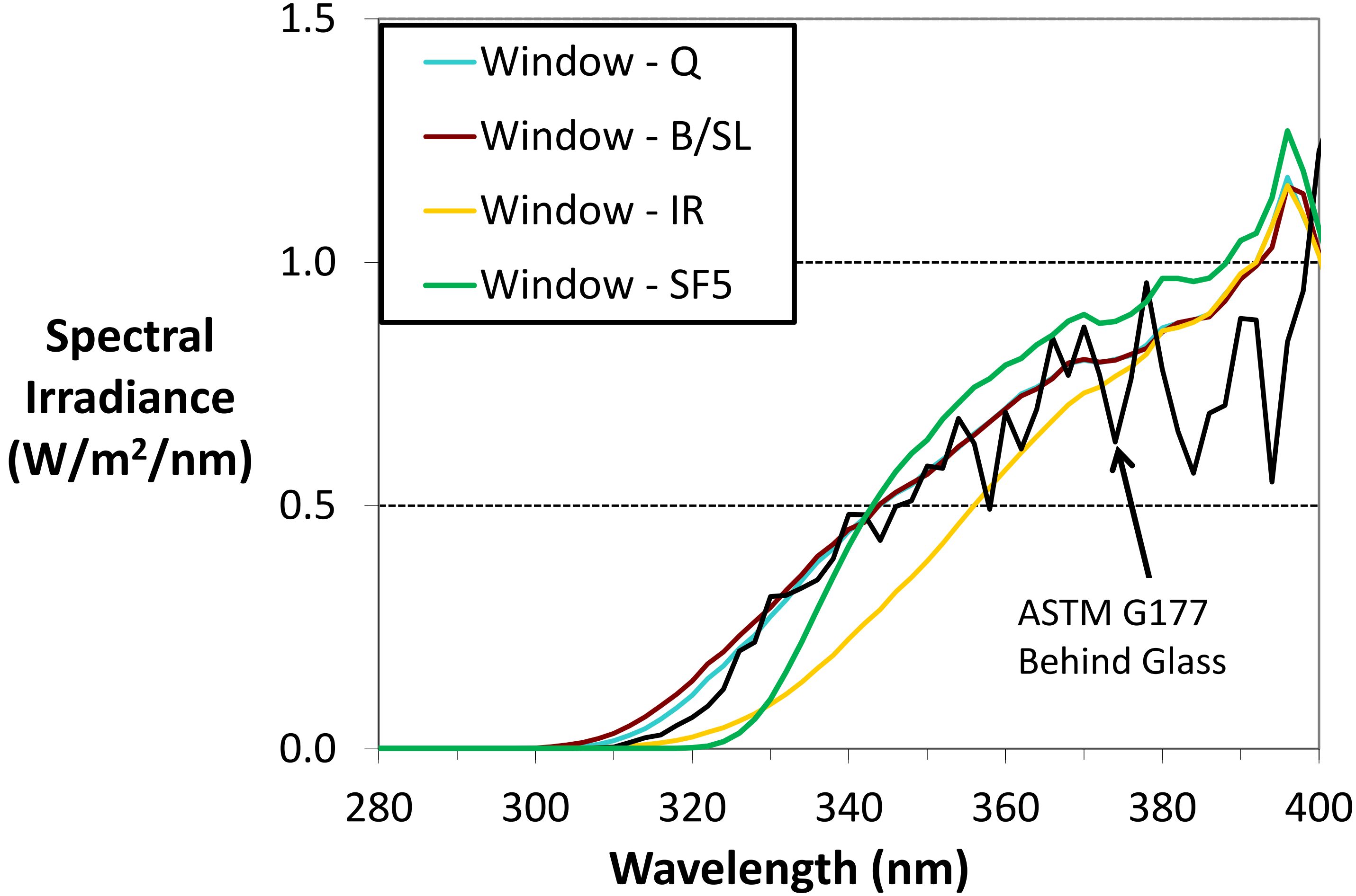
Xenon Arc with Window Filters

UV and Visible



Xenon Arc with Window Filters

UV Spectrum



Irradiance Control

- Narrow Band
  - 340 nm
  - 420 nm
- Total UV (300-400 nm) Wide Band
- Global (300-800 nm) – not recommended
  - Shorter wavelengths cause more photodegradation
  - Lamp aging can cause more than 50% reduction in critical UV wavelengths

Irradiance Control Point Conversion

Example: **Window B/SL** Filter

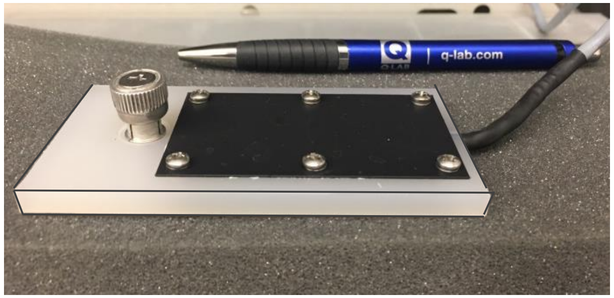
Control Point	Irradiance
340 nm	0.35 $W/m^2/nm$
420 nm	0.79 $W/m^2/nm$
TUV (300-400 nm)	40 W/m^2

These conversion factors only apply for this particular filter

Temperature Control

- Black panel
  - Hotter than ambient in sunlight
  - Not necessarily same as specimen temperature
  - Exists for test repeatability and reproducibility
- Chamber air
  - Controlled somewhat independently
  - More relevant for some applications
- Chiller System
  - Removes heat to allow normal indoor temperatures inside xenon arc test chamber

Black Panel Temperature Sensors

Panel	Construction	ASTM Designation	ISO Designation
	Black painted stainless steel	Uninsulated Black Panel	Black Panel
	Black painted stainless steel mounted on 0.6 cm white PVDF	Insulated Black Panel	Black Standard

** White Panel versions of the above are available but far less commonly used*

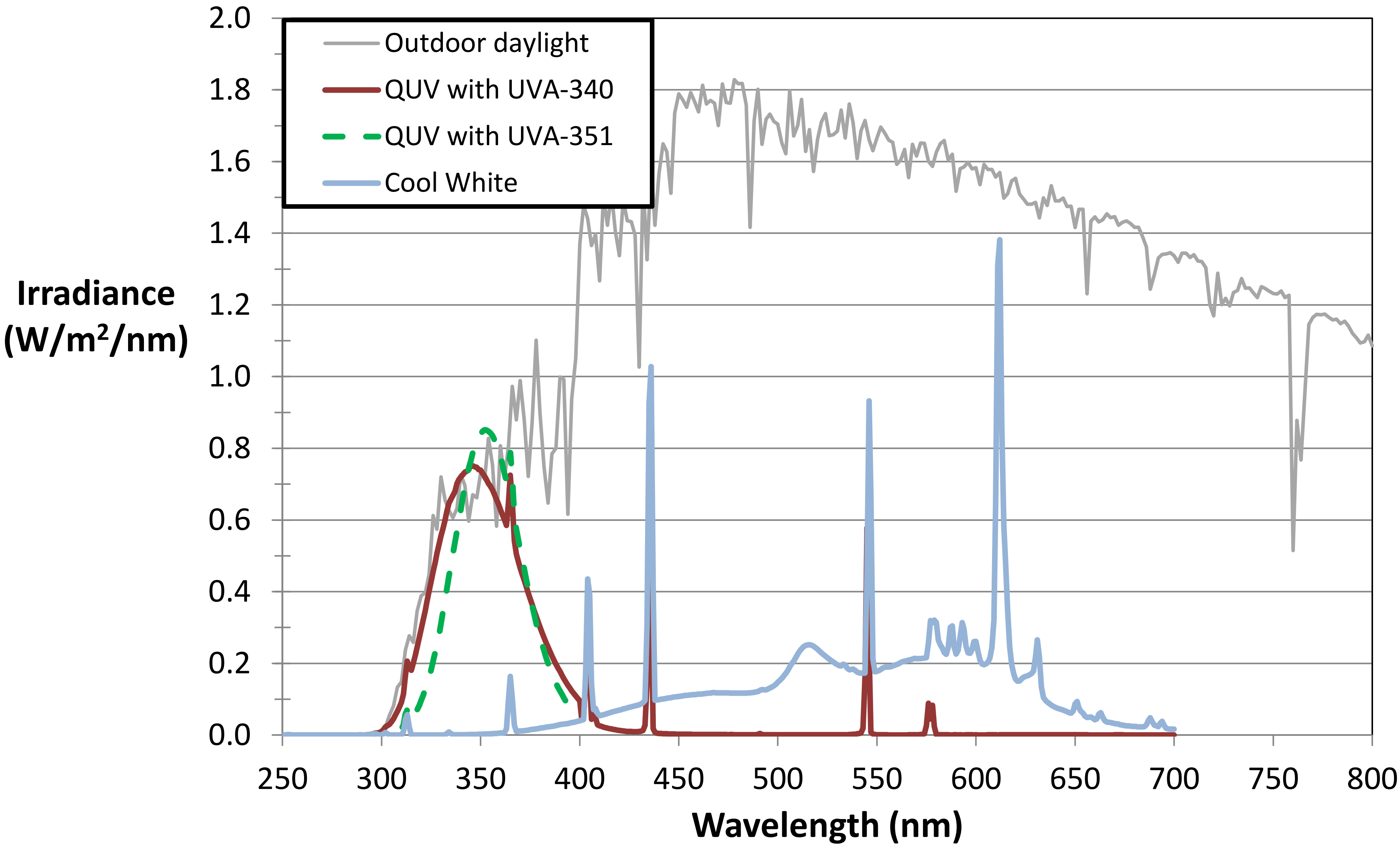
Fluorescent UV Testing

QUV/se Weathering Testing and QUV/cw Light Stability Testing Chamber

Benefits of Fluorescent UV Testing

- Lower-cost solution
- Highly repeatable and reproducible spectrum
- Cool White lamps are an excellent reproduction of commercial lighting
- Very easy to use

Fluorescent UV Light Spectra



ICH Guidelines

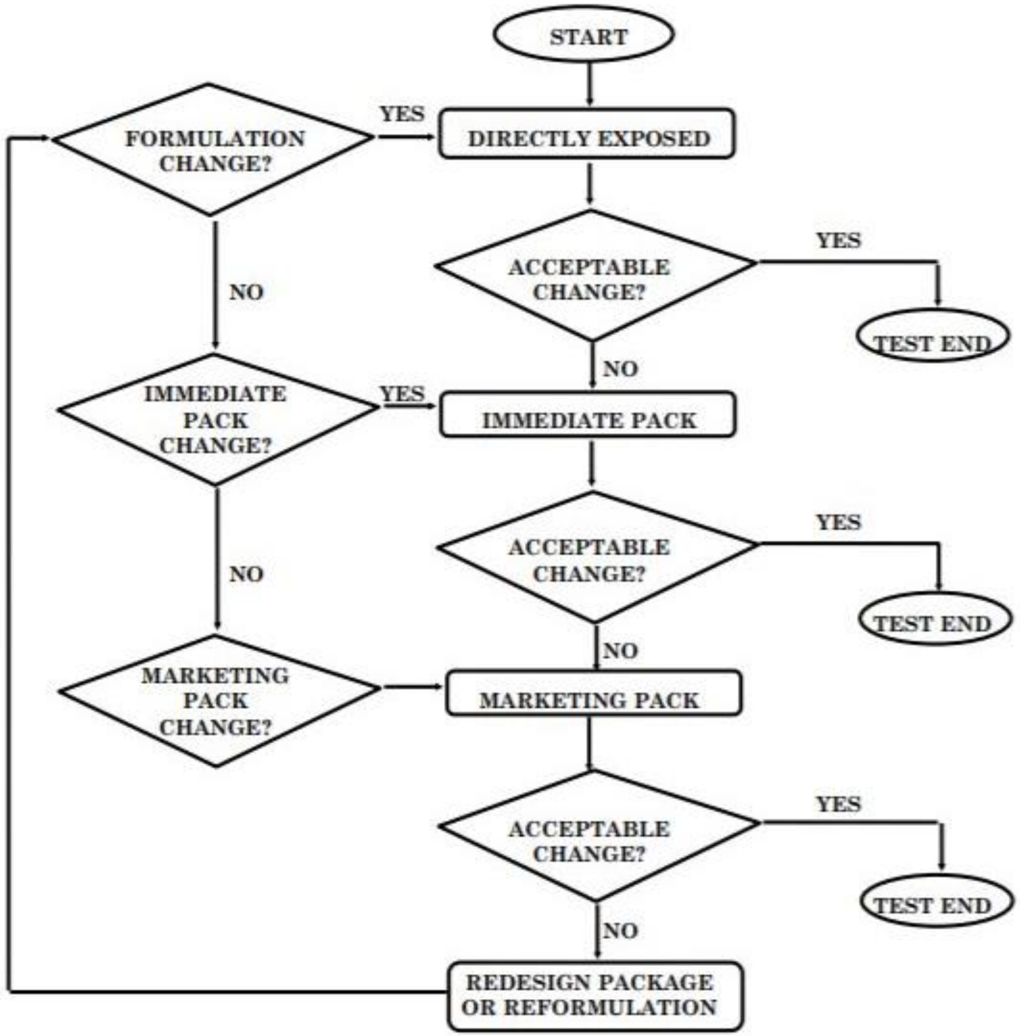
International Conference on Harmonization: Guidelines

for the Photostability Testing of New Drug Substances and Products

ICH Guidelines

- Joint effort of U.S., European, Japanese regulatory agencies
- New products and drug substances should not exhibit “unacceptable change” when exposed to light
- Two exposure options are available

ICH Guidelines Flowchart



ICH Guidelines

Two Exposure Options

1. D65/ID65 light source*
    - “artificial daylight fluorescent lamp combining visible and ultraviolet outputs, xenon, or metal halide lamp”
    - Wavelengths below 320 nm may be filtered

2. Cool white fluorescent and “near ultraviolet lamp”

** ICH Guidelines cite ISO 10977 on photographic films and prints, which is withdrawn and replaced by ISO 18909. They refer to CIE 15, Recommendations on Colorimetry.*
CIE 85 Solar Spectral Irradiance would have been a better choice for lightstability tests.

ICH Guidelines
Radiant Exposure

Exposures are based on UV ***radiant*** *dosage* and ***illuminance*** ** dosage*

**Illuminance is a measure of visible light that takes irradiance dosage and applies the human photopic response curve*

ICH Guidelines
Radiant Exposure Criteria

Two exposure values must be reached:

1. 1.2 million lux-hours (per m^2) *minimum (*visible light by definition*)*

2. 200 Watt-hours UV (per m^2) *minimum*

– These do not correspond specifically to either the D65 or ID65 reference light source

- *No single light source can meet the visible light exposure conditions without significant "over-exposure" of the UV portion*
- *"Over-exposure" is perfectly acceptable*

Value 1: Calculating Lux-hours

Irradiance (W/m^2) at each wavelength

×

Photopic Response (lumens/W) at wavelength

=

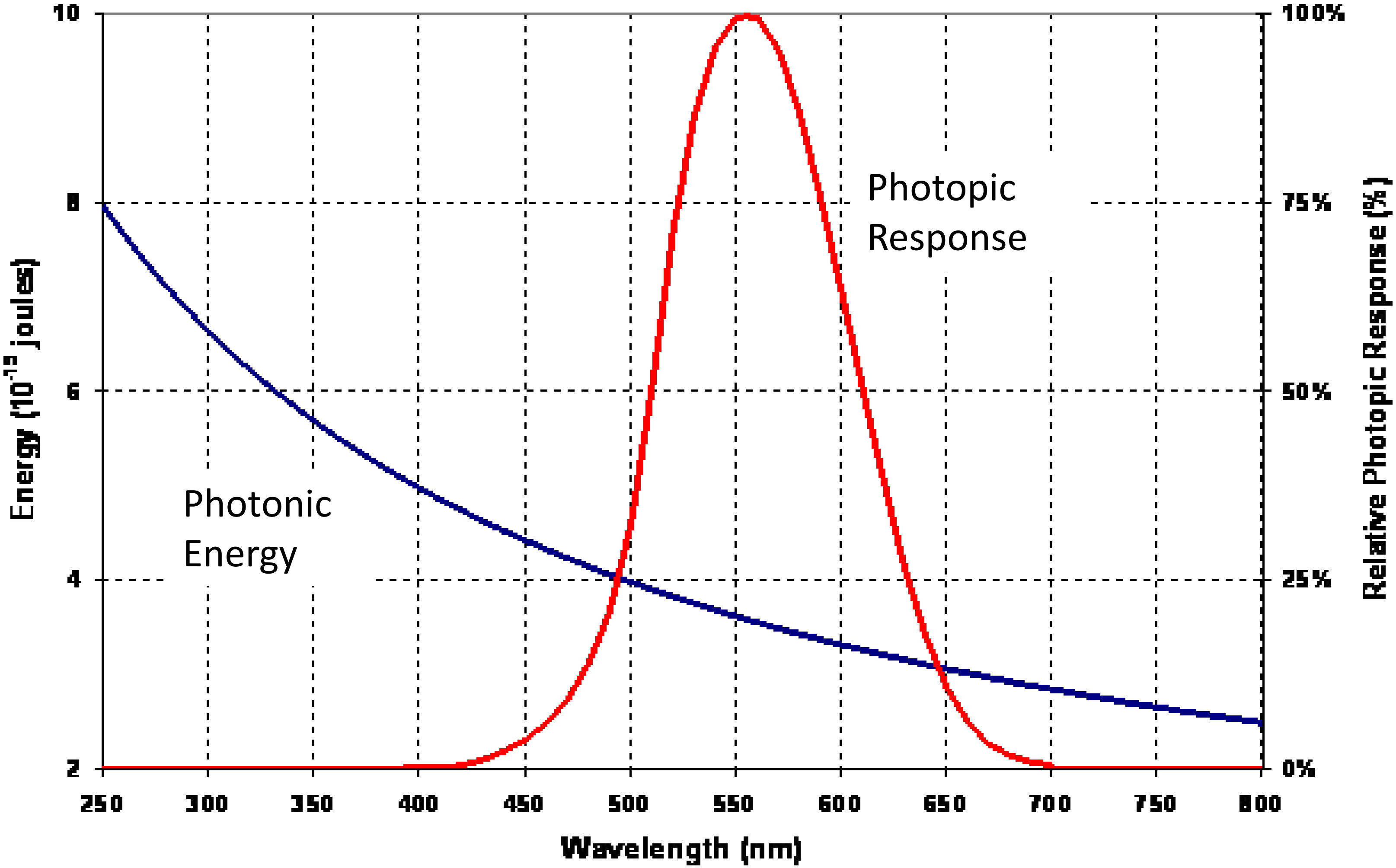
Illuminance ($lumens/m^2$) or lux

Example:

Wavelength (nm)	*Photopic Response (lumens/W)*		*Irradiance (W/m^2)*		*Illuminance ($lumens/m^2$)(lux)*
555	**683.00**	×	**0.33**	=	**227.2**

Now, sum up the value at each wavelength and multiply this number by exposure time in hours

Photopic Response & Photonic Energy

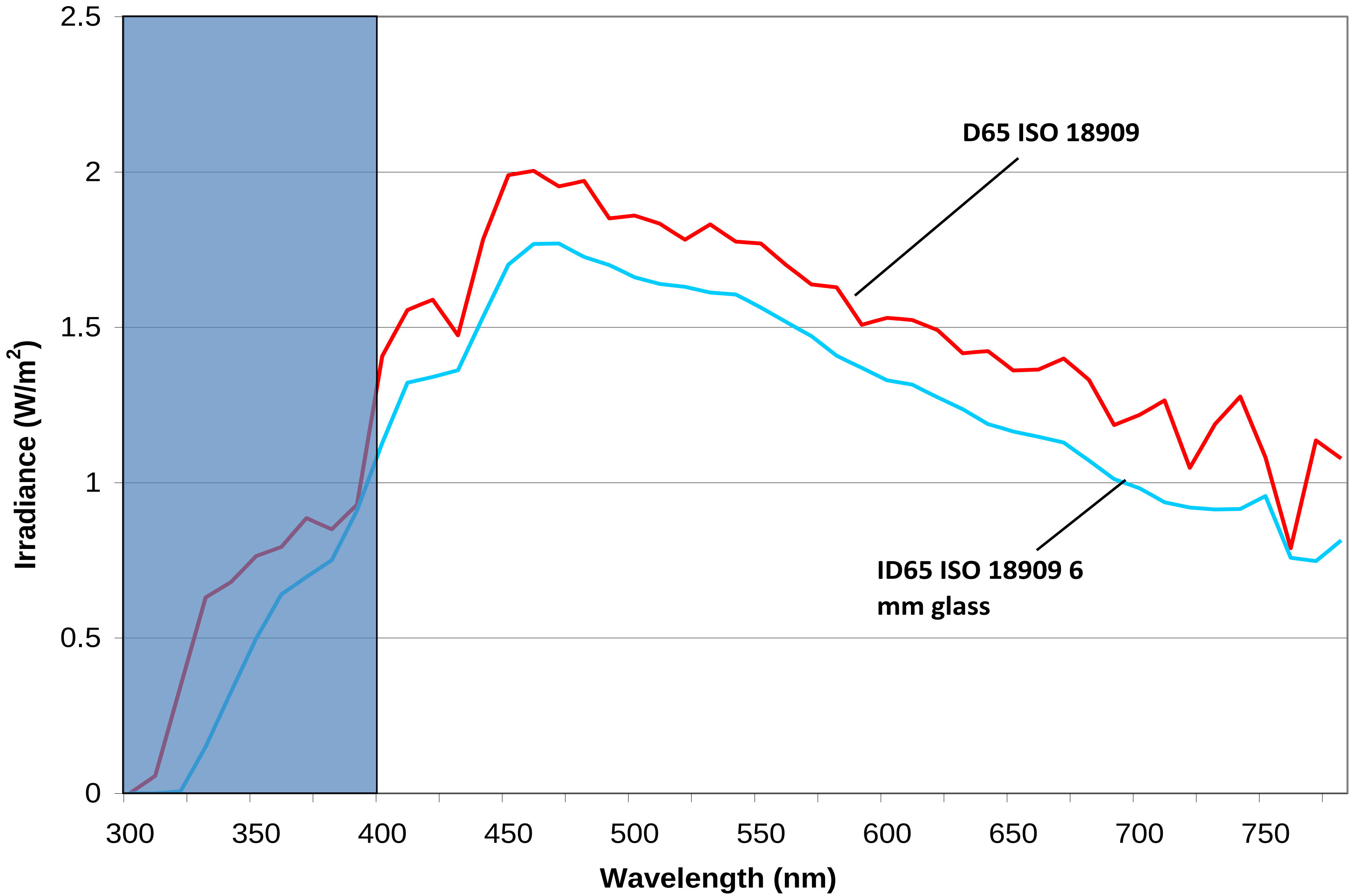


Value 2: Calculating TUV Watt-hours

- SPD data gives you irradiance (W/m^2) at each wavelength
- Sum irradiance at wavelengths 300-400 nm (Total UltraViolet or "TUV")
- Multiply this number by exposure time measured in hours

$40\ W/m^2 \times 10$ hours = 400 W-hours/m^2

Total **UV** Exposure (TUV, 300-400nm)



ICH Guidelines
Temperature

Temperature is not specified, however …

- Thermal degradation should be evaluated separately in heat aging tests, not during lightfastness testing. Therefore, testing at normal room temperature ranges is desirable
- Room temperature testing requires chilling the air circulated through the chamber

ICH Guidelines
Performing Option 1

- Q-SUN Xe-1BC
- Window – Q Filter (ID65 3 mm glass spectrum
- 420 nm irradiance control point, 1.10 $W/m^2/nm$
- Chamber Air temperature control, 25 °C

ICH Guidelines
Option 1

Test duration

- Run test for 13.1 hours
- 650 Watt-hours UV (225% more UV than required)
- 1.2 million lux-hours

To reduce the UV exposure, run in two parts

- Part 1: Run until 200 W-hr/m^2 TUV exposure, using Window-Q Filters
- Part 2: Add a UV Blocking filter, recalibrate, and run to achieve 1.2 million Lux-hours (no additional TUV)

Irradiance & Test Time

Option 1, Q-SUN with Window-Q

Irradiance @ 420 nm	Hours	Lux-hours	TUV Dosage (Watt-hr/m^2)
0.50 W/m^2	28.9	1.2 million	647
0.60 W/m^2	24.1		
0.70 W/m^2	20.7		
0.80 W/m^2	18.1		
0.90 W/m^2	16.1		
1.00 W/m^2	14.5		
1.10 W/m^2	13.1		

Multiple pathways to reach the specified exposure criteria

ICH Guidelines

Option 2

Step 1: QUV with cool white lamps

Set Point: 20,000 lux

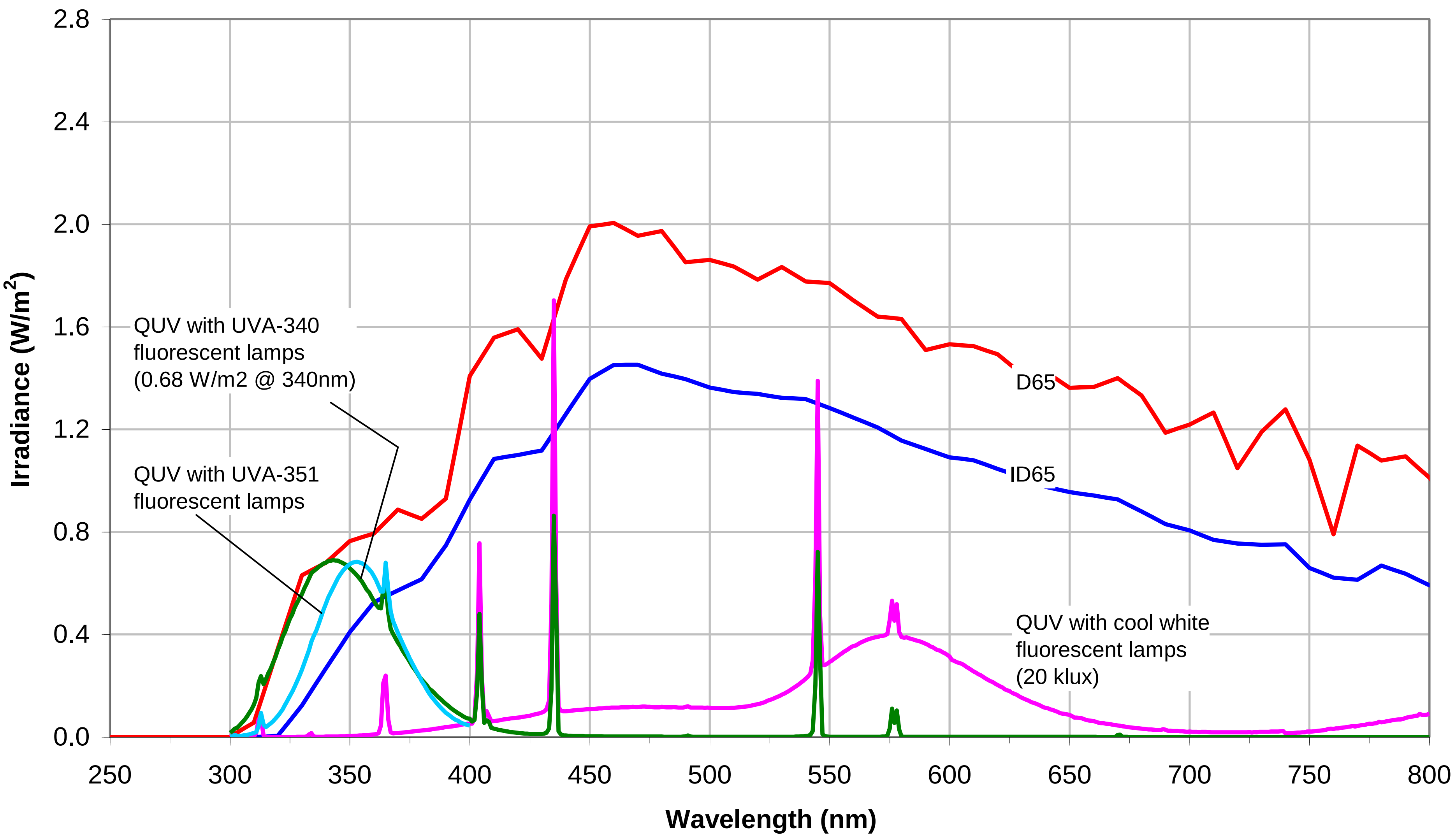
Time: 60 hours

Step 2: QUV with UVA-351 lamps

Set Point: 0.55 $W/m^2/nm$ @ 340 nm

Time: 4 hours

QUV Light Spectra and ICH Guidelines



Best Practices and Practical Considerations in Light Stability Testing

Best Practices And Practical Considerations

1. **Perform natural exposures**
   - Necessary for understanding accelerated results
   - Does lab test correctly rank material performance?

Miami outdoor exposures

Best Practices And Practical Considerations

2. **Test until failure** (forced degradation)
   - Required for drug products
     - Identify impurities resulting from photodegradation
     - Determine degradation pathways
   - Necessary for developing rank order performance

Best Practices And Practical Considerations

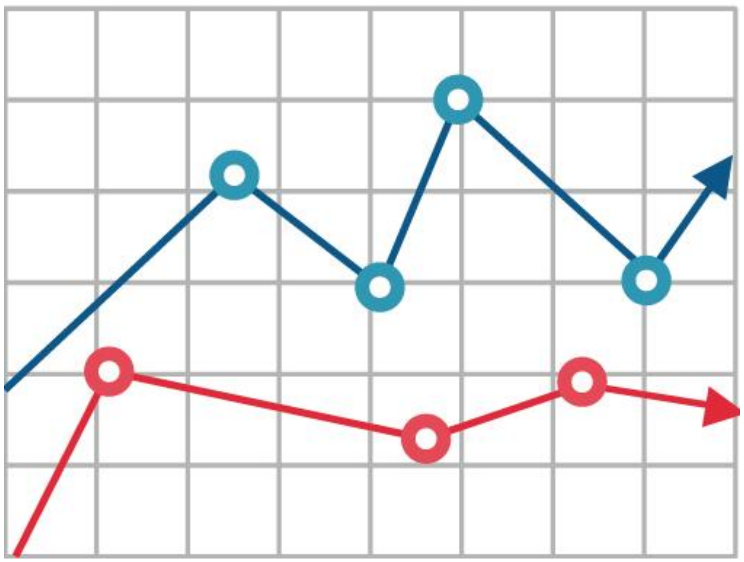
3. **Expose a control with your test specimen**
   - Use Control Material of Known Durability
     - Outdoor performance
     - Lab performance
   - Similar Composition to Test Material
   - Similar Degradation Mode to Test Material

Benefits of a Control

- Compare performance of control to a known material
- Allows confidence in lab exposure
- Assure that laboratory tester is operating properly

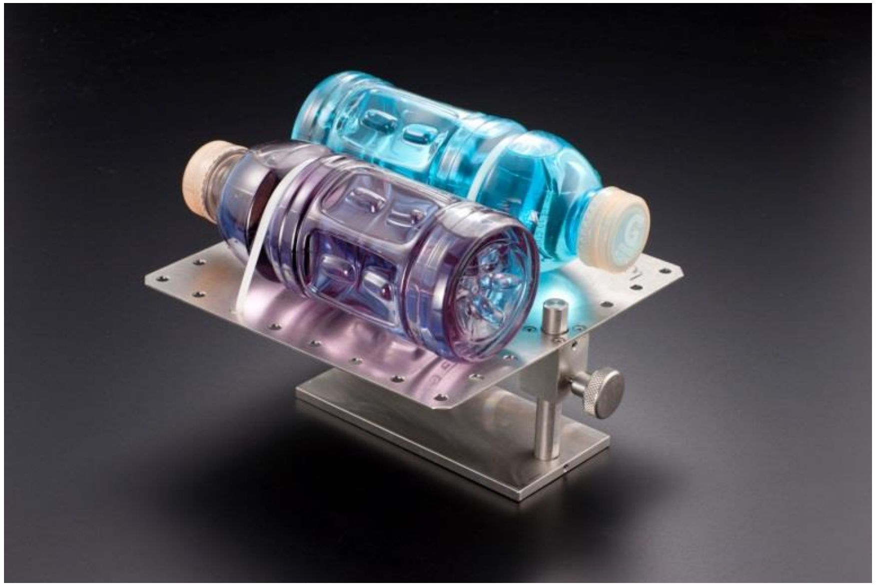
Correlation using Control Materials

- Consider developing a new products that must be lightfast, but ingredients to make it lightfast are expensive.
- One can select a known good control product and included control with all new product testing
- Test both materials for a specified duration.
- If new product performs worse than the control, reformulate to have greater lightstability
- **Retest!**



Best Practices And Practical Considerations

4. Test your product "In the package" in order to simulate the actual service environment.



Whole Product Testing

Q-SUN Xe-3

Q-SUN Xe-1

Best Practices And Practical Considerations

5. Use realistic temperatures to prevent unrealistic failures

Testing with a chiller system allows for higher irradiance while maintaining cool temperatures

Q-SUN Specimen Capacity

Q-SUN Xe-3HC

$3200\ cm^2$

Q-SUN Xe-1BC

$1100\ cm^2$

Correlation

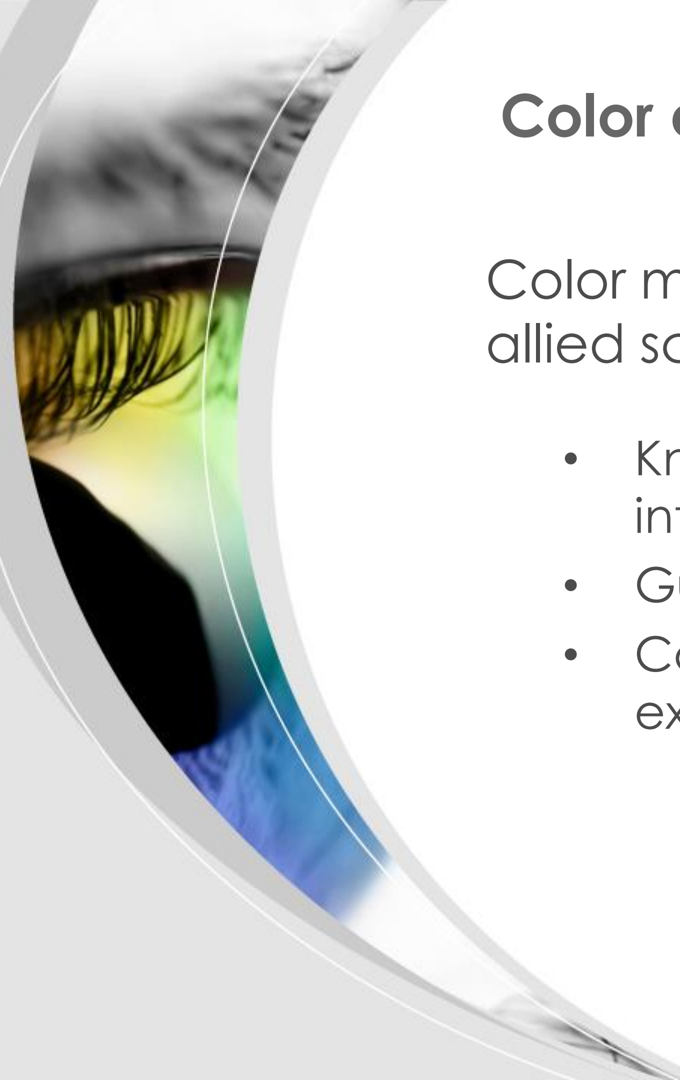
5. Use realistic temperatures to prevent unrealistic failures

Testing with a chiller system allows for higher irradiance while maintaining cool temperatures

Questions?

The world’s true measure of color

HunterLab



Color and Weathering Sciences

Color measurement and weather testing are allied sciences:

- Knowledge and simulation of light and how it interacts with the product is critical
- Guided by and conform to global standards
- Color measurement tests the effects of exposure to light, temperature, and moisture

Key Learning Objectives

1. What is Color Measurement?
   - Colorimetry, the science
   - Quantifying and communicating color

2. Implementing color measurement and Best Practices

3. Applications challenge: measuring liquids and powders in the personal care industries

The world's true measure of color
HunterLab

Applications Challenge

Challenges for Color Measurement – Home and Personal Care Products

Difficult to measure because:

1. Opaque and transparent characteristics
   - Light is transmitted and scattered
2. Color observed in reflectance and transmittance
3. Sample forms and structure vary:
   - Semi-solids, powders, gel forms
   - Liquids, aerosols
   - Surface area to be measured
4. Sample preparation
5. Sample presentation

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What is Colorimetry?

Colorimetry

- Evaluates color as the human eye
- Combines and defines the illumination and human observer into the color assessment
- Incorporates the entire visible region for analysis – like your eyes
- Uses color scales to describe, quantify, and communicate colors

Colorimetry

- Colorimetry is a globally accepted method
- Defined by ASTM, DIN, ISO, CIE (and others)
- Can correlate instrumental color measurement to the visual assessments
- Can define any color with tristimulus values that incorporate the light source and human observing situation to describe the color or appearance of an object

To Define a Color

Three essential parameters to define, describe, and understand color:

1. **Light source** / illuminating condition
2. How the **object** / sample interacts with the light
3. The **observer** or observing situation

The world's true measure of color

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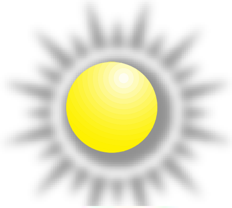
COLOR PERCEPTION

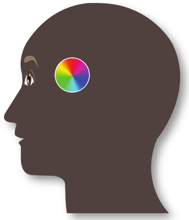
Things Required To See Color

A **Light Source**

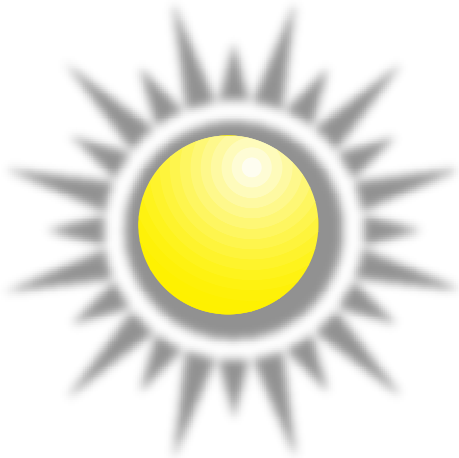


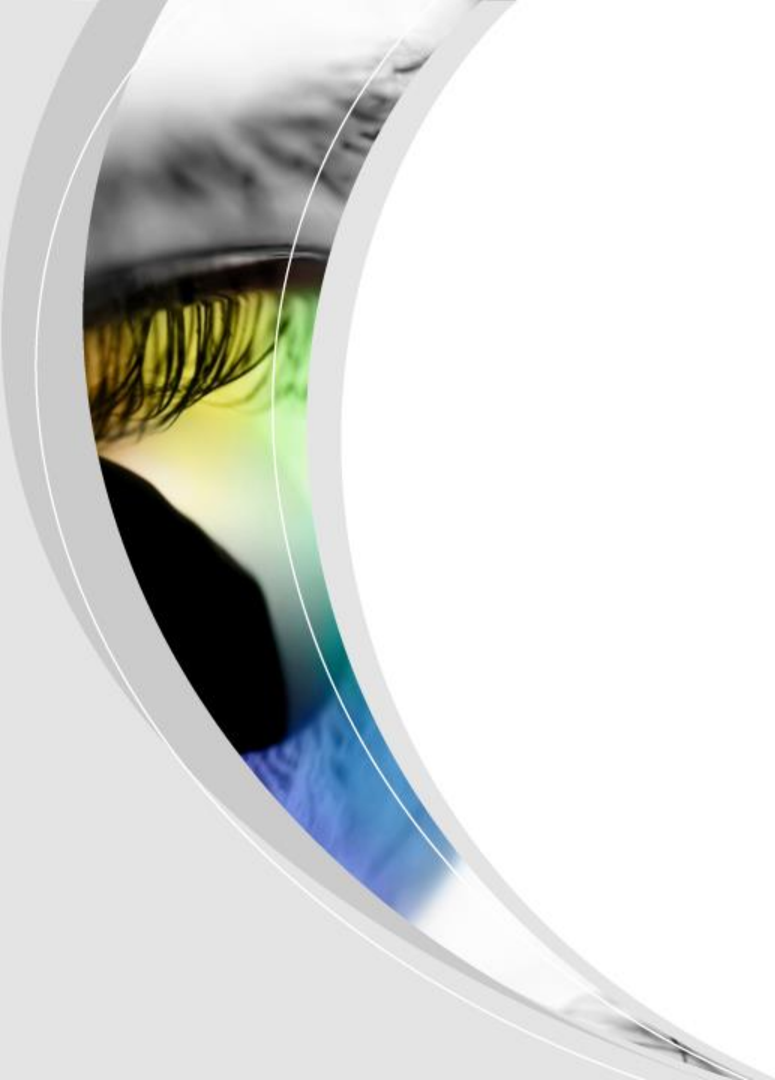
An **Object**

An **Observer**

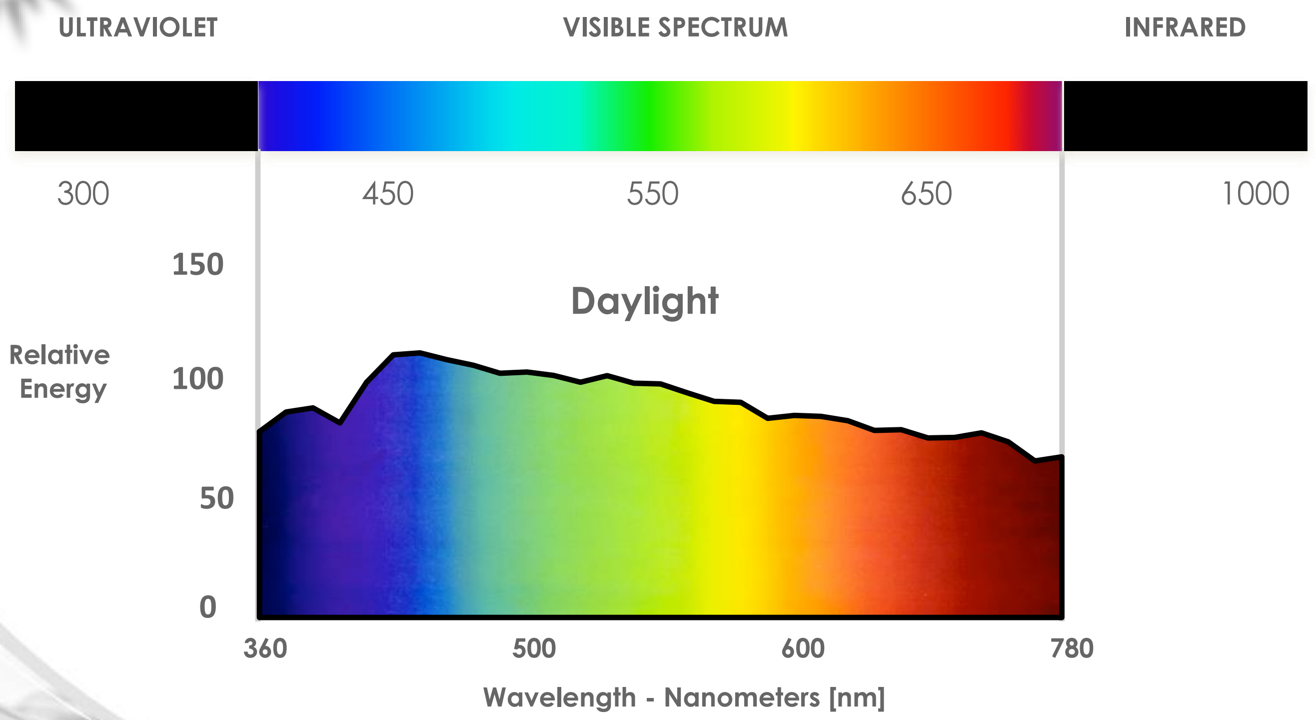


Light Source





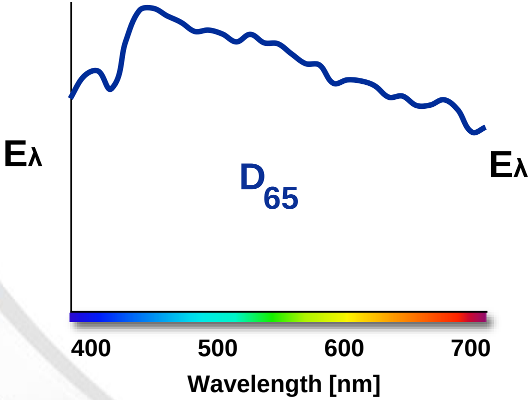
Spectral Power Distribution of Sunlight

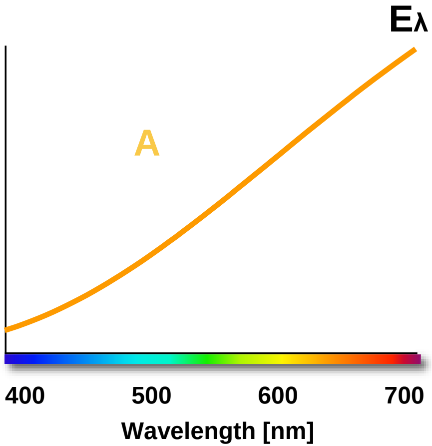


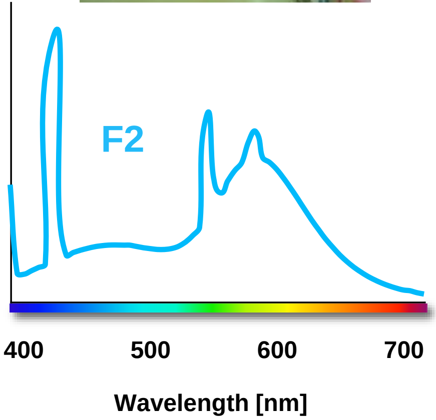
Light Source versus Illuminant

Source

Illuminant







Object

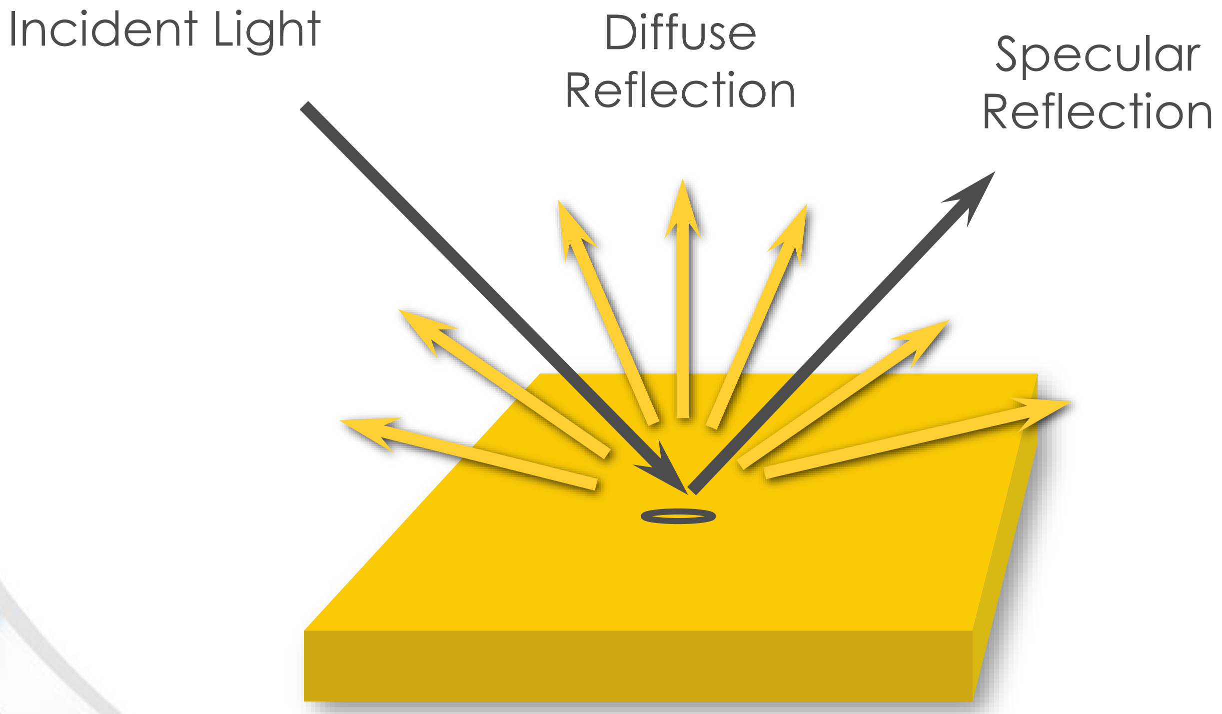

COMMERCIAL
ARM & HAMMER
THE STANDARD OF PURITY
Pure Baking Soda
America's #1 Trusted Baking Soda Brand
Ideal for Restaurants and Institutional Kitchens
NET WT. 1 LB. (454g)


GOLD BOND
HEALING
MEDICATED
SINCE 1908
ORIGINAL STRENGTH
BODY POWDER
Triple Action Relief
Cooling • Absorbing • Itch Relieving
Menthol 0.15%

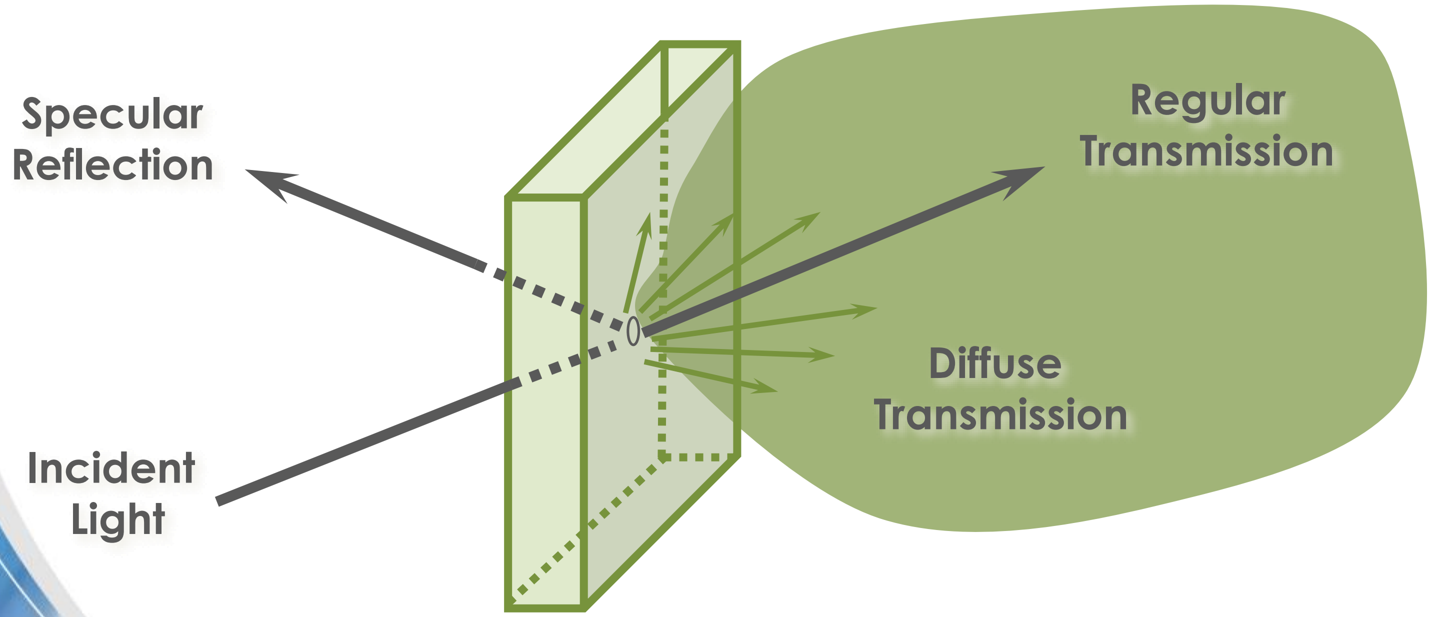
Object

- Objects modify light:
  - Reflect
  - Transmit
  - Absorb
  - Scatter
  - Fluoresce
- Colorants in the object selectively absorb some wavelengths of light while reflecting or transmitting others

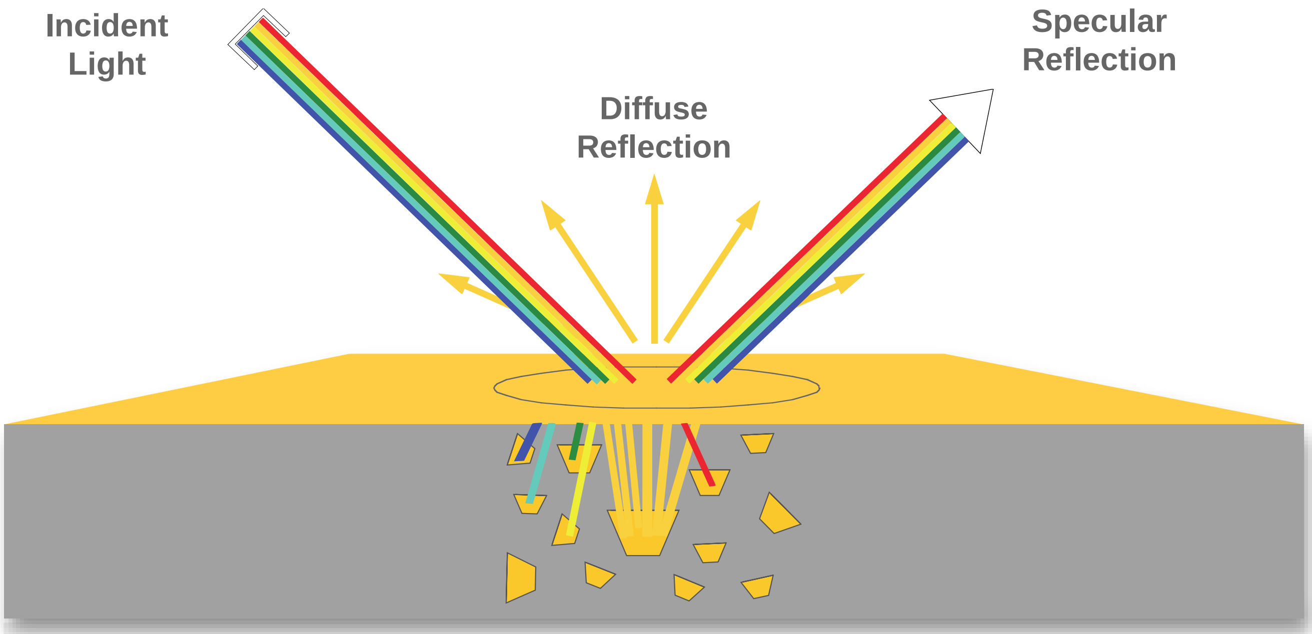
Reflectance of Light



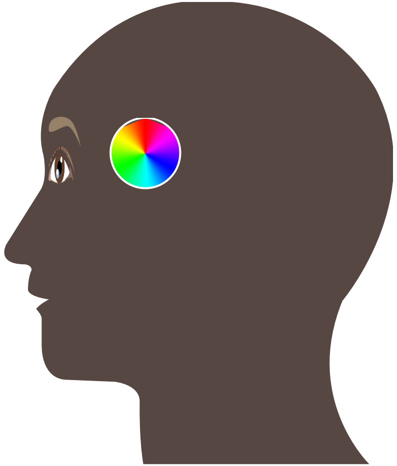
Transmission of Light



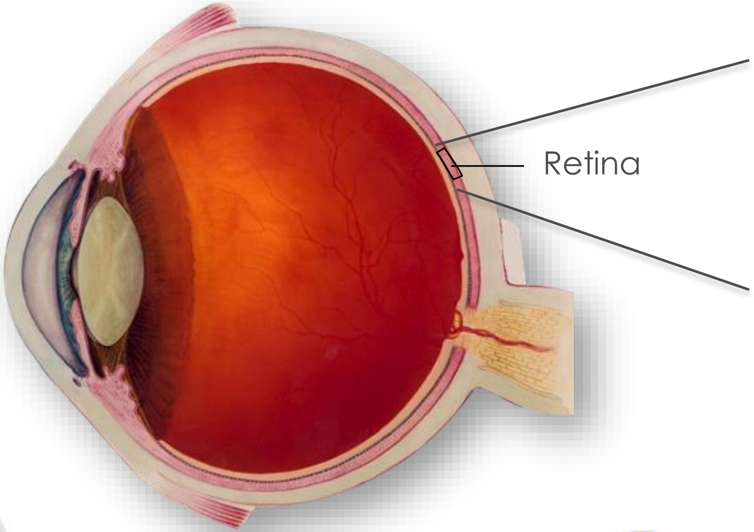
Light Interaction with Yellow



Observer

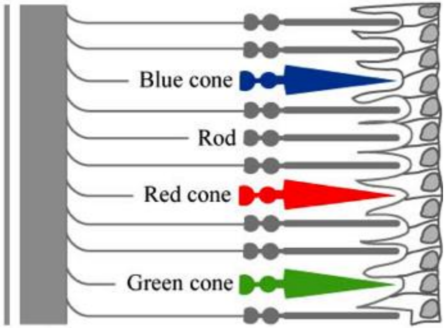


The Human Eye

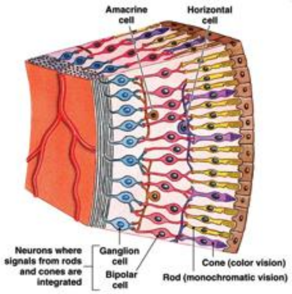


Retina

Light



Light



Retina

Human Observer

- **Rods** in the eye are responsible for low light vision (including night vision).
- **Cones** in the eye are responsible for color vision and function at higher light levels.
- The three types of cone sensitivities are **red**, **green** and **blue**.

CIE Standard Observers

- CIE 1931 **2°** Standard Observer
- CIE 1964 **10°** Standard Observer
- These functions quantify the red, green and blue cone sensitivity of the average human observer

Three Elements to See Color:

Summary: The three elements of the visual observing situation to see color:

- The **Light Source** is a user-selected CIE illuminant
- The **Object** is quantified by measuring the reflectance or transmission
- The **Observer** is represented by a CIE Standard Observer

The world's true measure of color
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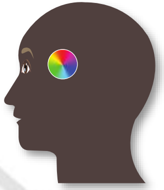
COLOR MEASUREMENT

Required to See or Measure Color

To See Color

Light Source

Object

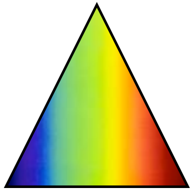


Observer

To Measure Color

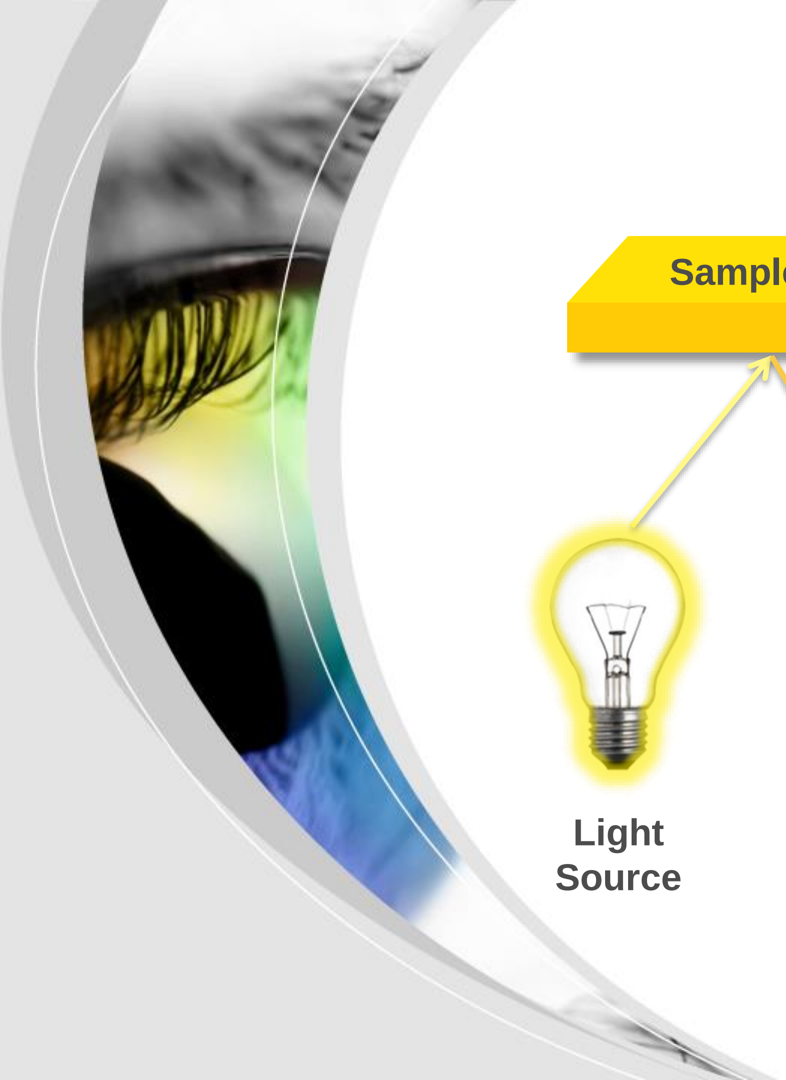
Light Source

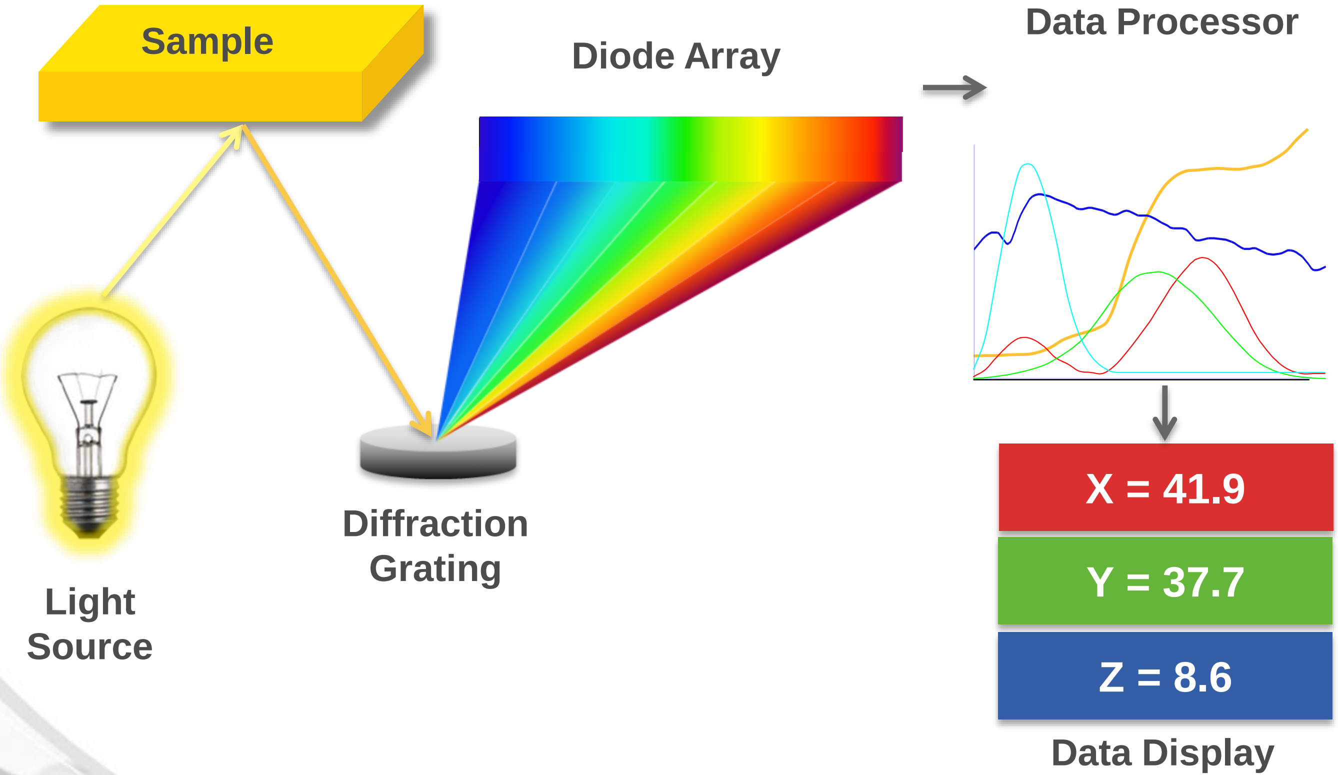
Sample



Spectrophotometer

Measuring Color





How is Color Communicated?

"Yellow"
Measured Values

X = 41.9

Y = 37.7

Z = 8.6

Color Scales

In terms of object color, X, Y, Z values are not easily understood. Other color scales have been developed to:

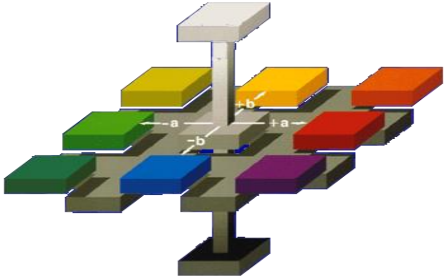
- Better relate how we perceive color
- Simplify understanding
- Improve communication of color
- Better represent uniform color differences

L,a,b Values

L,a,b values relate directly to how the eye sees color and are calculated from the X,Y,Z values

L - lightness or darkness,

0 is complete black, 100 is complete white



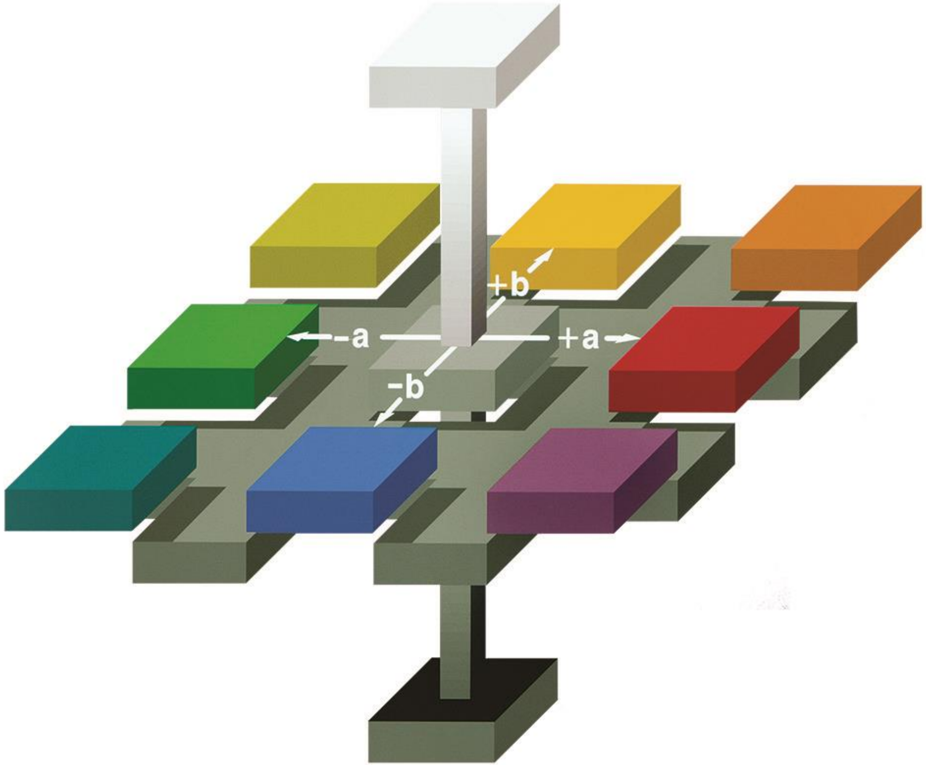
a - redness or greenness

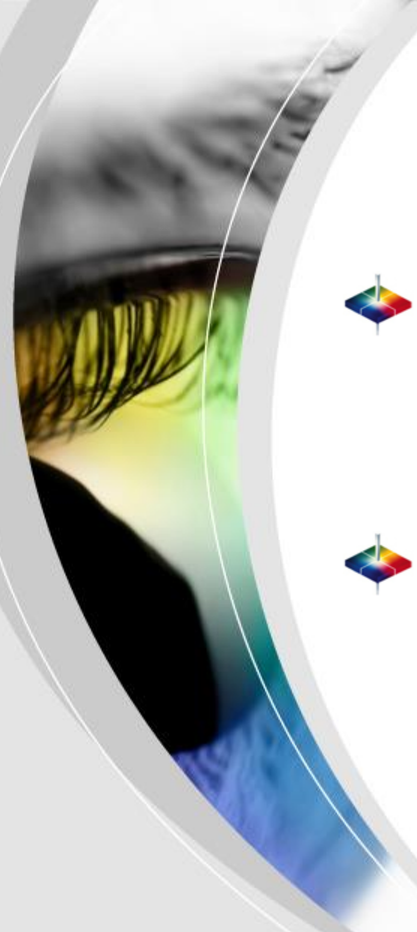
+a values are red, -a values are green

b - blueness or yellowness

+b values are yellow, -b values are blue

L,a,b Color Space





Hunter L, a, b Color Space

- All colors can be represented in L, a, b rectangular color space.
- The following slide shows where “yellow” falls in Hunter L, a, b color space.

Hunter L, a, b Values for “Arm and Hammer Yellow”

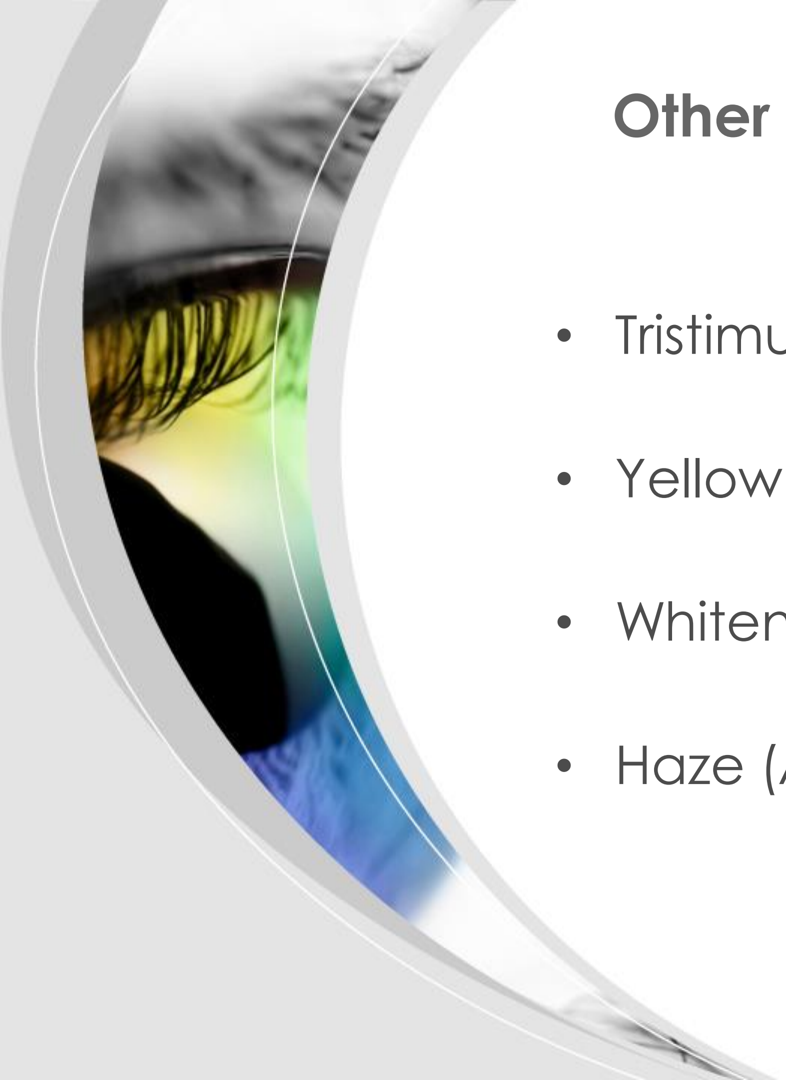
L = 61.4

a = + 18.1

b = + 32.2

L, a, b Color Scales

- There are two popular L,a,b color scales in use today: **Hunter L,a,b** and **CIE L*,a*,b***.
- While similar in organization, a color will have different numerical values in these two color spaces.
- **The current CIE recommendation is to use the CIE L*,a*,b* scale**



Other Common Color Scales

- Tristimulus Color, color difference (L*a*b*, ΔE*)
- Yellowness Indices (APHA, ASTM YI, CIE)
- Whiteness Indices (ASTM WI, CIE)
- Haze (ASTM, turbidity)

The world's true measure of color
HunterLab

Applications, Sampling and Best Practices

Challenges for Color Measurement – Home and Personal Care Products

Difficult to measure because:

1. Opaque and transparent characteristics
2. Light is transmitted and scattered through the samples
3. Color observed in reflectance and transmittance
4. Sample forms and structure
5. Sample preparation
6. Sample presentation

Opaque Application: Sampling Techniques

- Measure a largest sample area practical in consistent sample vessel
- Illuminate the product circumferentially to average out non-uniformity
- Average measurements, rotate
- Sealed optics

Translucent Application: Sampling Techniques

1. Make the sample:
   - ***Thinner*** for transmission measurements
   - ***Thicker*** for reflectance measurements
2. Control the background
   - White recommended – but be consistent!
3. Average measurements (rotate, re-fill)
4. Largest area view is best

General Guidelines

Color Scale: CIELab (L*, a*, b*)

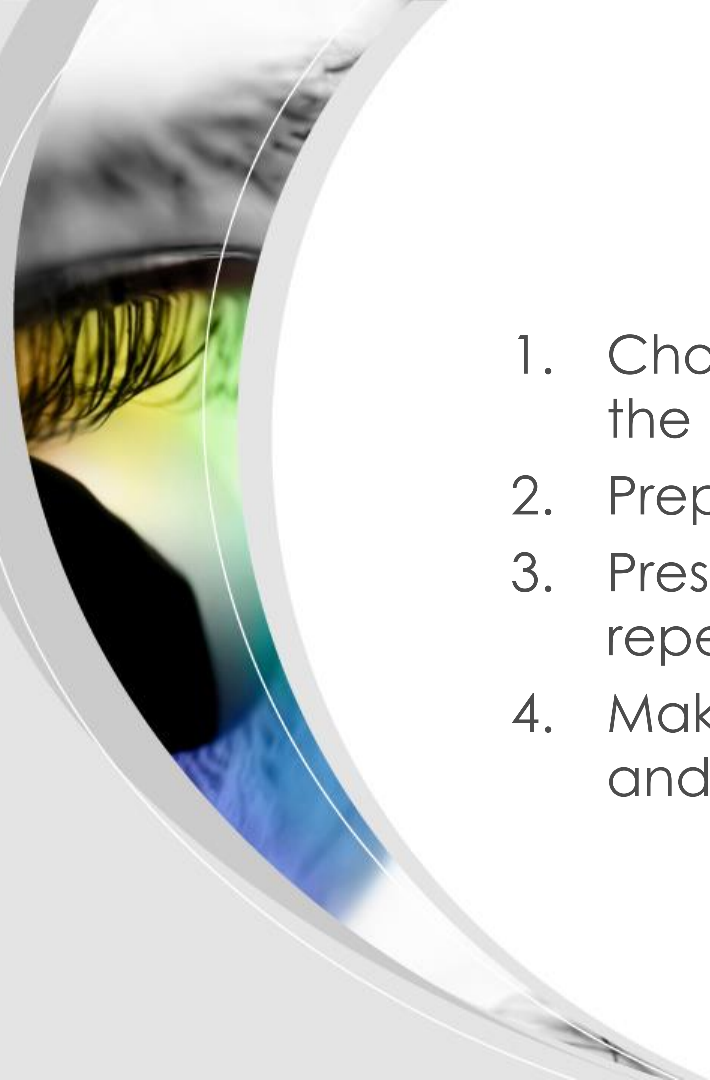
Illuminant: D65

Observer: 10 degree

Tolerances: ± 1 (min), ± 5 (max)

Method:

- Varies for reflectance (solids/powders) or
- transmission (liquids/solutions)



Best Practices

1. Choose samples that are representative of the product and manufacturing
2. Prepare samples the same way each time
3. Present the samples to the instrument in a repeatable manner
4. Make multiple preparations of the sample and average measurements

Establishing a Specification

- **YOU** define the color quality relationship
- The measurement system includes:
  1. Customer acceptance parameters
  2. Raw material inspection
  3. Process variables and variation
  4. Business considerations
- Internal systems may be tighter

Thanks

Additional info available from:

HunterLab web site:
www.hunterlab.com

Email questions to:
paul.barnes@hunterlab.com