Light Stability Testing of Home and Personal Care Products
家居及个人护理品的光稳定性测试

Kobe Qu (瞿华盛)

Senior Technical and Marketing Manager

kqu@q-lab.com

Q-Lab Corporation

点击查看课程资料和视频回放。

What We Will Talk About

- Weathering Testing vs. Light Stability

耐候测试 vs. 光稳定性测试

- Common Light Spectra

常见光谱

- Natural Exposures

自然曝晒

- Accelerated Testing
  - Xenon Arc Testing 氙灯
  - Fluorescent UV Testing 荧光紫外
- ICH Guidelines ICH准则
- Best Practices and Practical Considerations 测试注意事项

Weathering Testing
耐候测试

- Combination of sunlight, heat, and moisture

阳光，温度和潮湿的协同作用

- Temperatures simulate realistic hot outdoor conditions

温度模拟实际户外条件

- Moisture (water spray or condensation) usually included

潮湿（水喷淋或者冷凝）通常被考虑

Light Stability Testing
光稳定性测试

- Simulation of sunlight or indoor lighting

模拟阳光或者室内光源

- No moisture* or elevated temperatures

没有潮湿，没有高温

- Test temperatures often simulate typical indoor environment

测试温度通常模拟典型室内环境

May control RH to reduce variability

**控制RH以减少湿度变化*

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

如果你不确定你的材料的表现，想评估在任何环境下的变化，
请使用耐候老化测试

- 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

如果你的材料仅仅是在一个固定的环境，并且只关心光作用在你产品上的变化，
请使用光稳定性测试

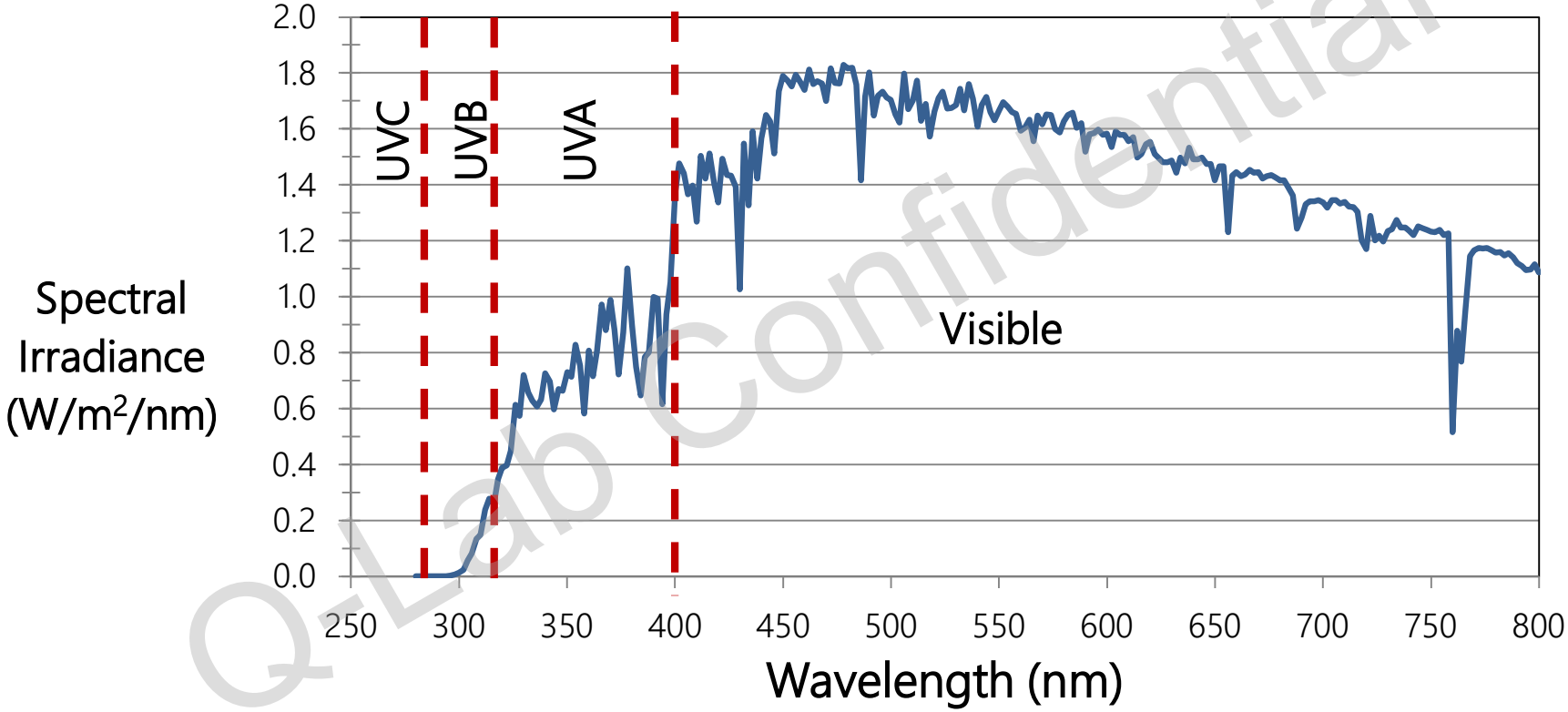
Common Light Spectra

- Sunlight 阳光
  - Direct 户外直射
  - Through Window Glass 透过窗玻璃

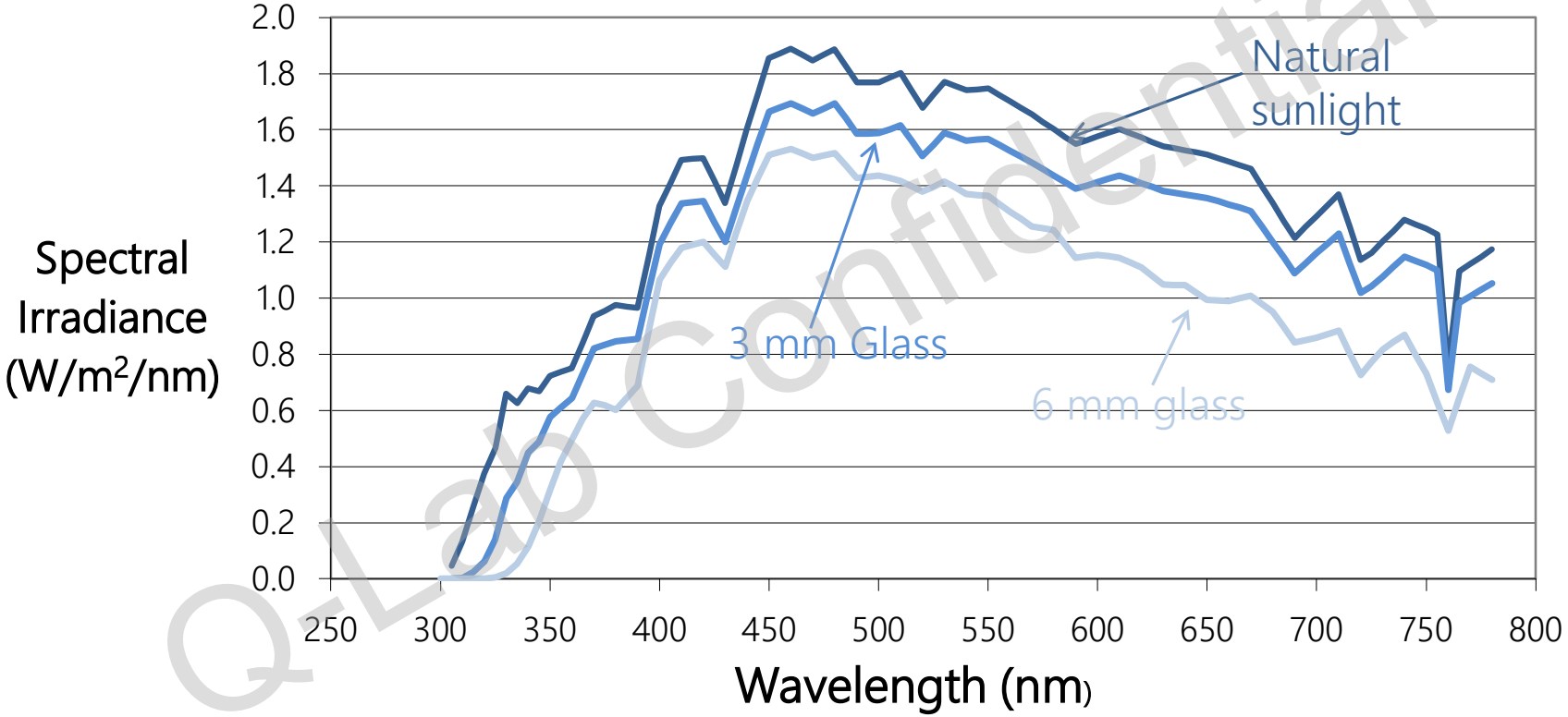
- Commercial Lighting
商超内光源

- Home Lighting
室内光源

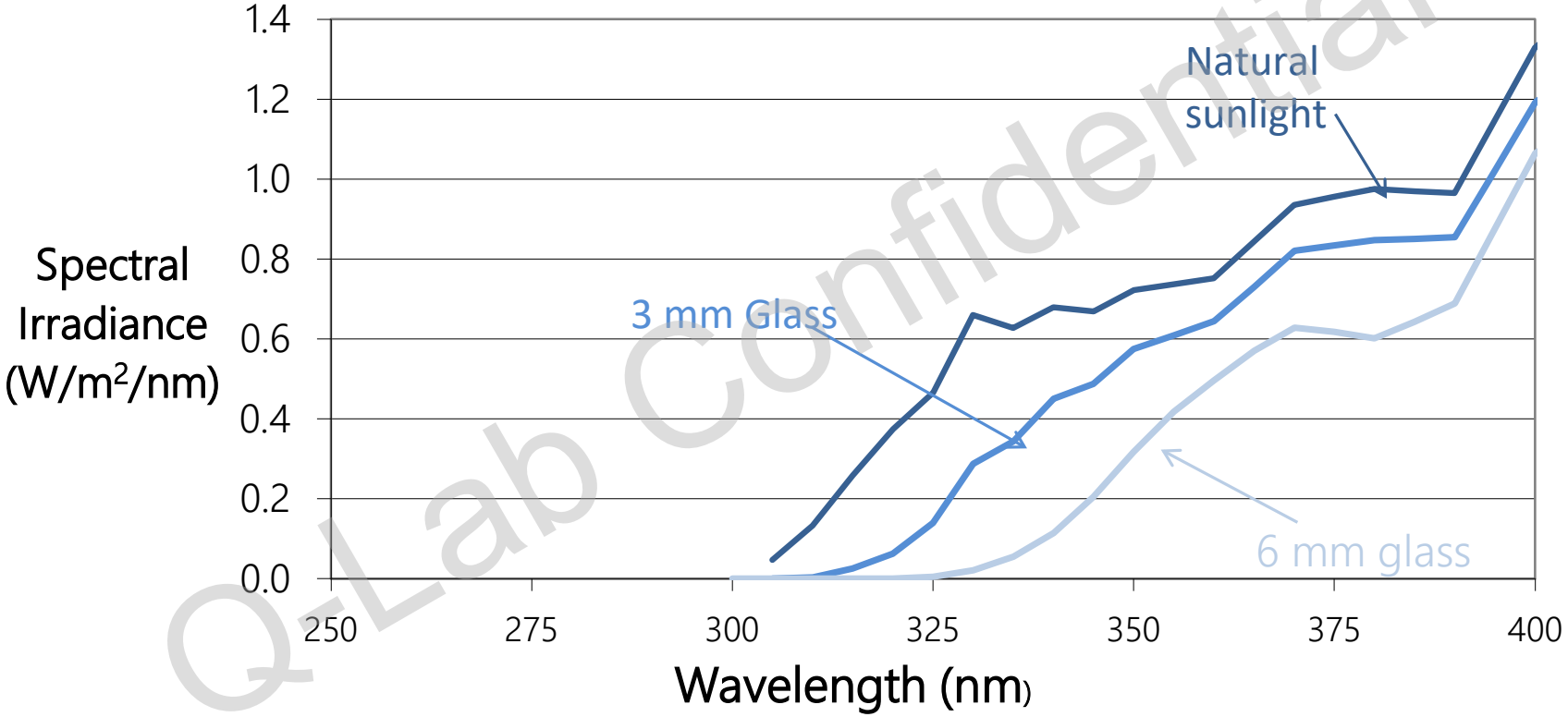
Summer Sunlight Spectrum



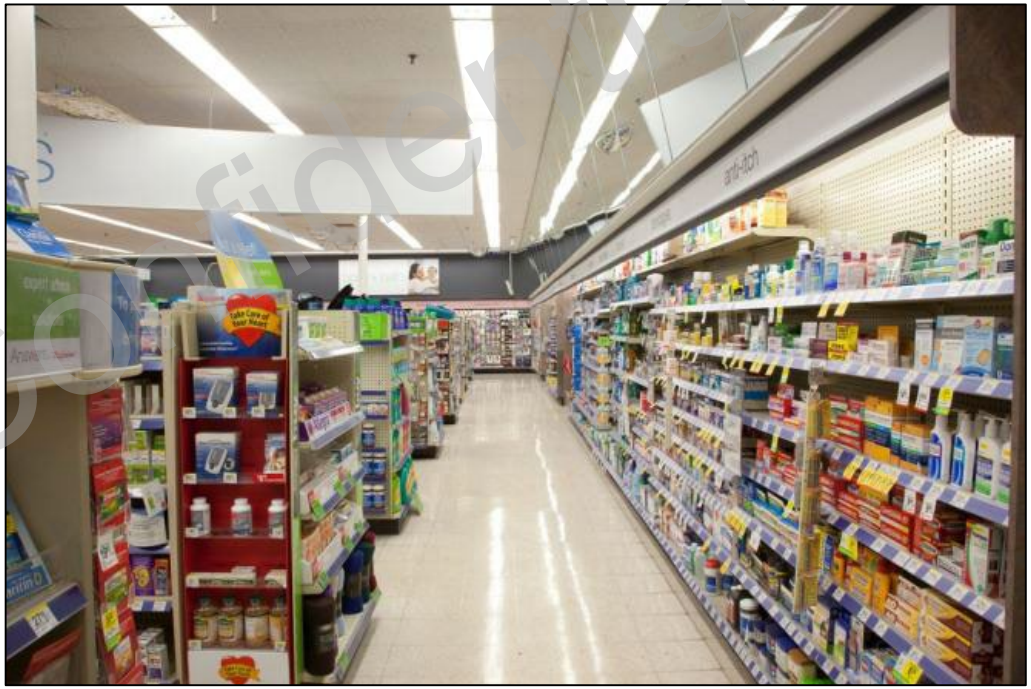
Sunlight through Window Glass



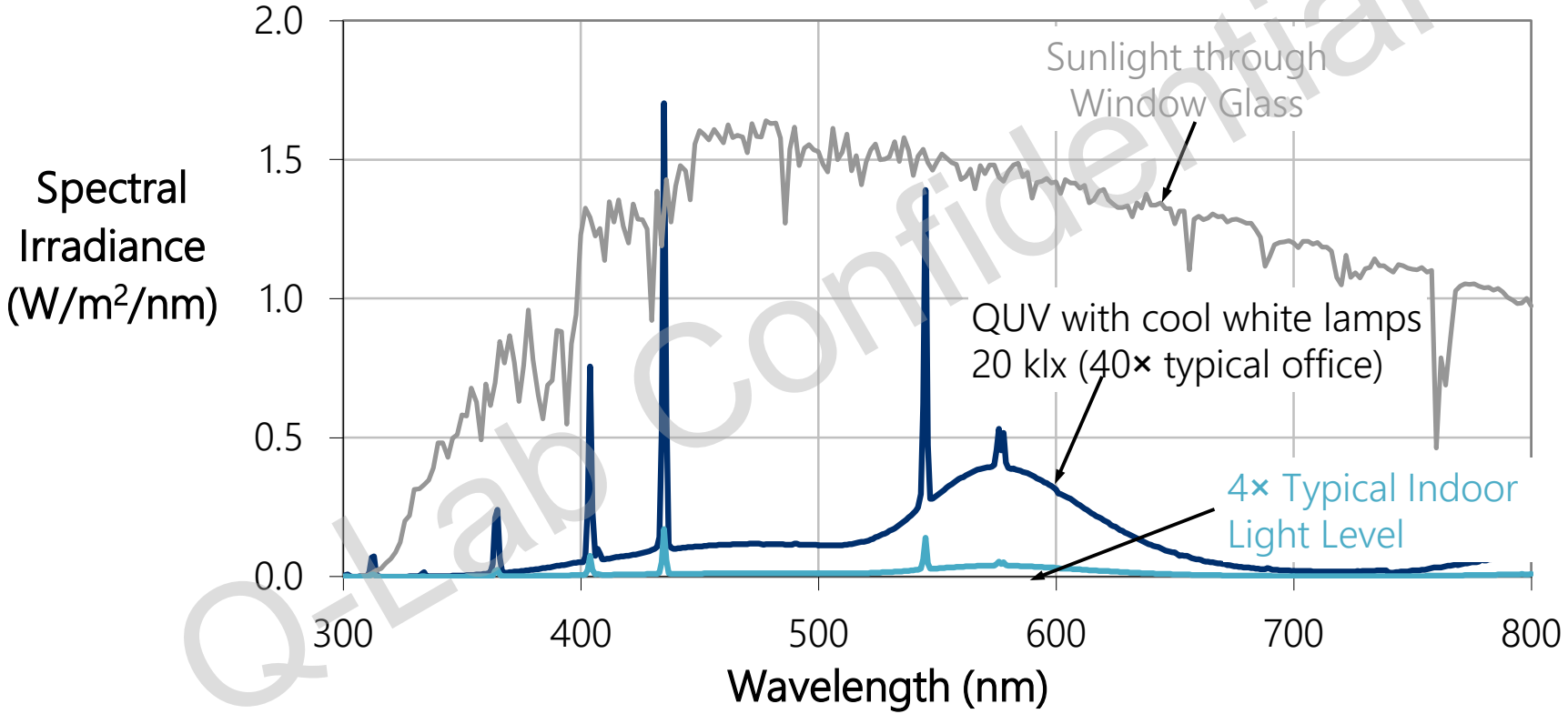
Sunlight through Window Glass



Interior Lighting



Commercial Indoor Lighting



Natural Exposures

Natural Exposures

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

为了了解你的材料在实际服役环境中的使用情况，

Put it in the service environment!

把材料放在实际环境中去！

Natural Exposures

- Benchmark Commercial Sites

标准商业曝晒场

- South Florida, Arizona Desert

南佛罗里达，亚利桑那沙漠

  - Inexpensive 不贵
  - Reliable 可靠
  - Extreme environments create acceleration 环境恶劣

- At your own facility

在你自家的厂区

  - “Scientific Window Sill Testing” 窗台测试
    - Convenient 方便
    - Easy to make frequent observations 便于查样
  - DIY Exposures

Natural and Accelerated 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

快消品在商业曝晒场的曝晒费用不贵，在很短的时间内就可以得到有用的数据

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

用氙灯和紫外老化测试机也可以很快测试快消品的光稳定性

Xenon Arc Testers

Q-SUN
Xe-3-HCE

Q-SUN
Xe-1-BCE

Q-SUN Xenon Test Chamber

Benefits of Xenon Arc Testing

- Realistic simulation of longwave UV and visible portion of sunlight

长波紫外和可见光真实模拟

- Optical filters can simulate different kinds of glass

光学滤镜可以模拟不用种类的玻璃

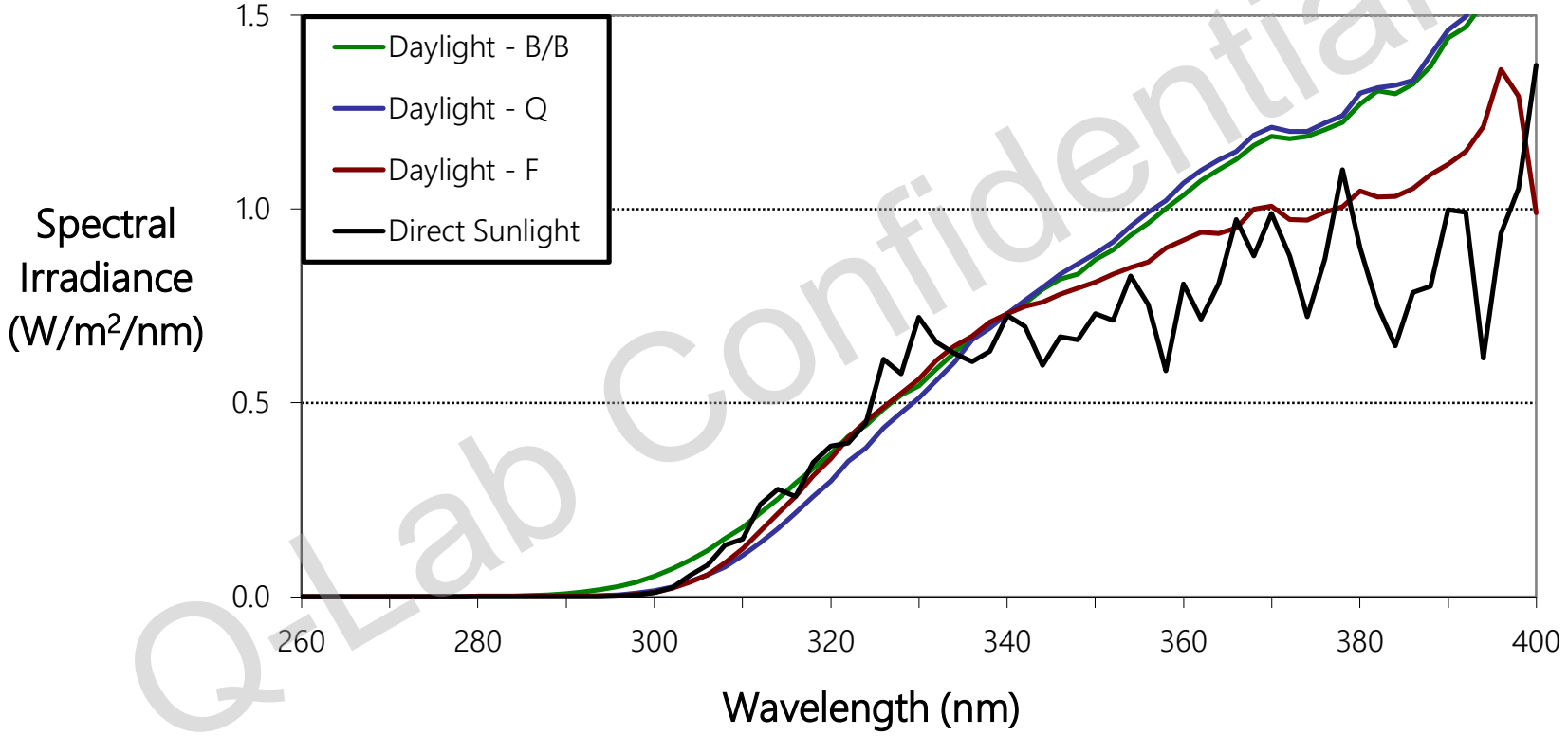
- Relative Humidity Control

相对湿度控制

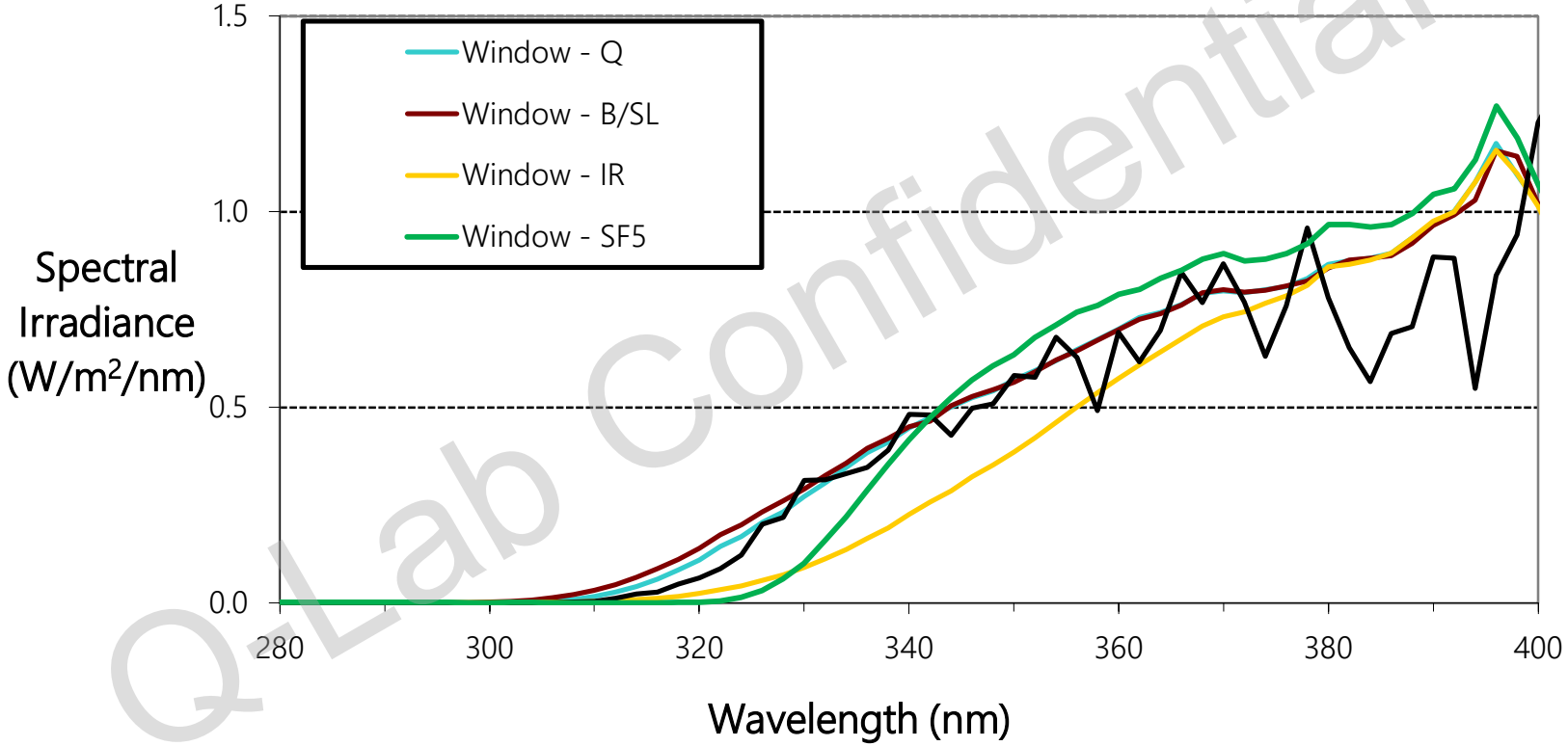
Optical Filters

- Daylight Filters 日光过滤器
  - Exterior exposures
- Window Glass 窗玻璃过滤器
  - Indoor exposures, textiles, inks
- Extended UV 紫外延展过滤器
  - Automotive, aerospace

Xenon Arc with Daylight Filters



Xenon Arc with Window Filters



Irradiance Control

- Narrow Band 窄带
  - 340 nm
  - 420 nm
- Total UV (300-400 nm) Wide Band 宽带
- Global (300-800 nm) – not recommended

300-800nm 控制不推荐

  - Shorter wavelengths cause more photodegradation

    短波造成主要光老化
  - Lamp aging can cause more than 50% reduction in critical UV wavelengths

    灯管老化造成紫外波段辐照度大幅下降

Irradiance Control Point Conversion

Example: Window-B/SL filter

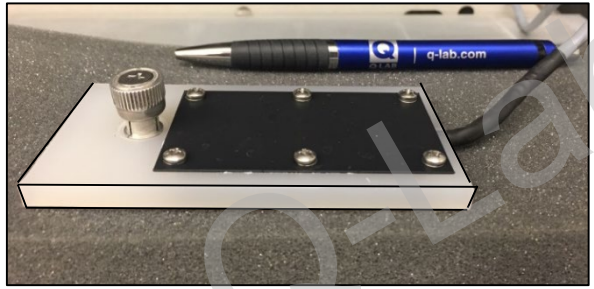
Control Point	Irradiance
340 nm	0.35 W/m^2/nm
420 nm	0.79 W/m^2/nm
TUV (300-400 nm)	40 W/m^2

These conversion factors only apply for this particular filter

Temperature Control

- Black panel 黑板
  - Hotter than ambient in sunlight
    在光照下高于空气温度
  - Not necessarily same as specimen temperature
    和样品温度不一样
  - Exists for test repeatability and reproducibility
    提高了测试的可重复性和可在现性
- Chamber air 箱体空气
  - Controlled somewhat independently
    独立控制
  - More relevant for some applications
    在一些应用中更有用
- Chiller System 制冷系统
  - Removes heat to allow normal indoor temperatures inside xenon arc test chamber
    箱内温度控制更低，接近室内环境温度

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

更便宜的方案

- Highly repeatable and reproducible spectrum

更稳定的光谱

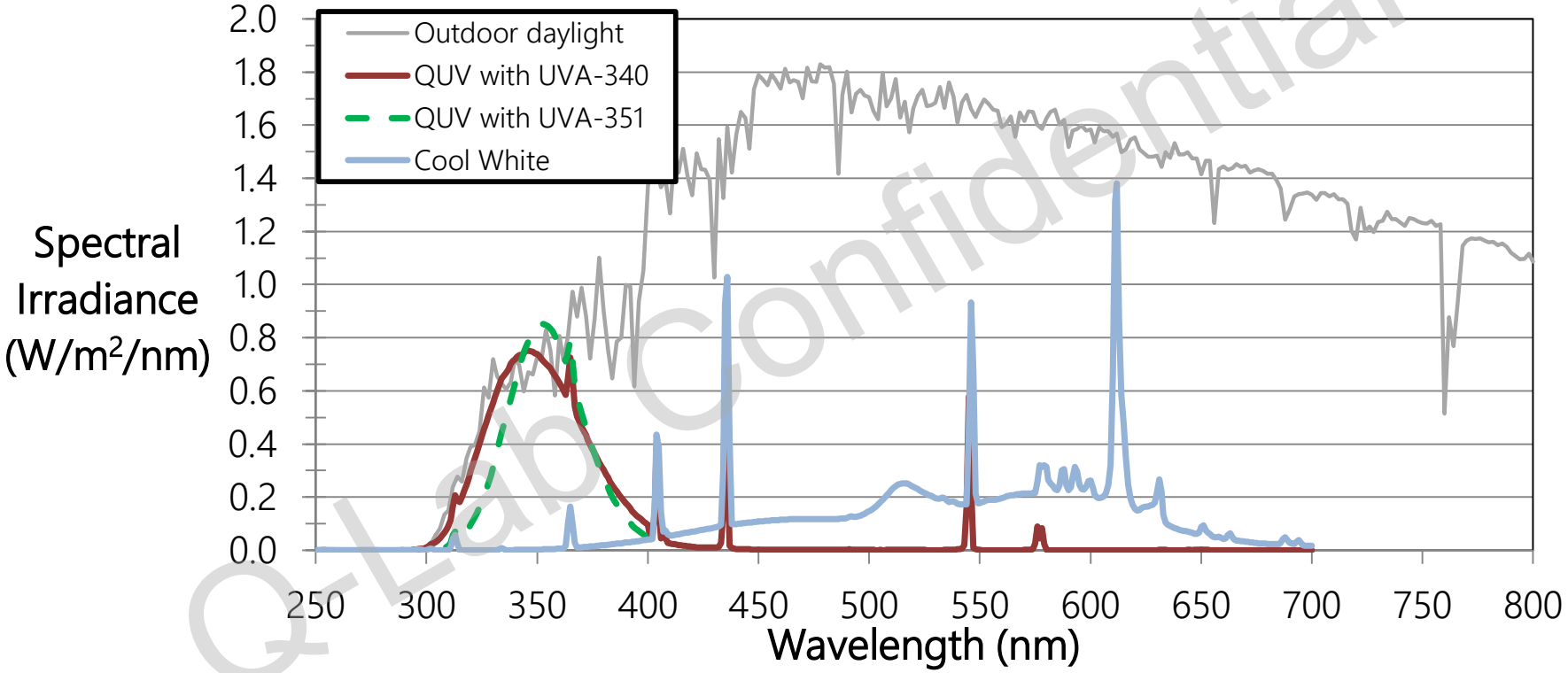
- Cool White lamps are an excellent reproduction of commercial lighting

冷白荧光灯重现室内照明环境

- Very easy to use

使用简单

Fluorescent UV Light Spectra



ICH Guidelines

International Conference on Harmonization: Guidelines

国际药品注册协调会议：指导原则

for the Photostability Testing of New Drug Substances and Products

新药的光稳定性测试

ICH Guidelines

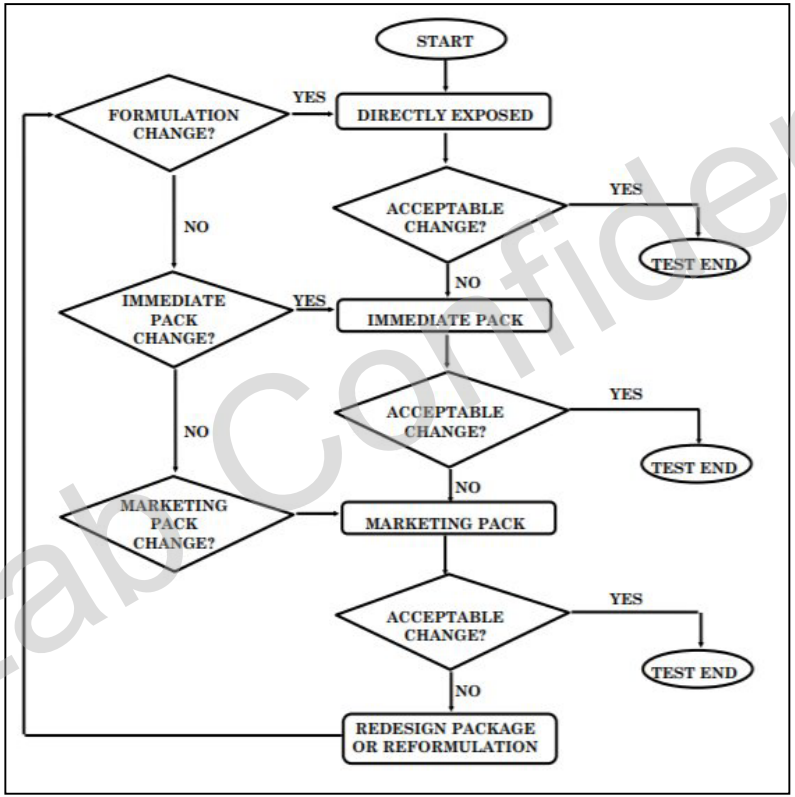
- Joint effort of U.S., European, Japanese regulatory agencies
- New products and drug substances should not exhibit “unacceptable change” when exposed to light

新药不应该出现“不可接受的变化”当曝露在光照下

- Two exposure options are available

两种曝晒方式

ICH Guidelines Flowchart



ICH Guidelines

Two Exposure Options 两种曝晒方式

- D65/ID65 light source*

D65/ID65光源

  - "artificial daylight fluorescent lamp combining visible and ultraviolet outputs, xenon, or metal halide lamp"

    含紫外和可见光的人造光源，比如氙灯，卤素灯等

  - Wavelengths below 320 nm may be filtered

    320nm以下的波段被滤掉

- Cool white fluorescent and "near ultraviolet lamp"

冷白荧光灯和近紫外灯

** 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. CIE85 / CIE241 Solar Spectral Irradiance would have been a better choice for lightstability tests.*

ICH Guidelines

Radiant Exposure 曝晒剂量

- Exposures are based on UV radiant and illuminance dosages

曝晒基于紫外辐照和可见光剂量

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

照度是对可见光的测量，它采用辐照度剂量并应用人眼的视觉响应曲线

ICH Guidelines

Radiant Exposure Criteria 曝晒标准

- Two exposure values must be reached:

两种曝晒方法必须满足：

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

    可见光至少 1.2 m lux *hour

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

    紫外光至少 200W *hour

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

D65或ID65光源没法同时满足以上最低曝晒要求

- No single light source can meet the visible light exposure conditions without significant “over-exposure” of the UV portion

没有一种单一人造光源可以在满足可见光剂量的同时紫外不超标的

- “Over-exposure” is perfectly acceptable

过量曝晒是可接受的

Value 1: Calculating Lux-hours

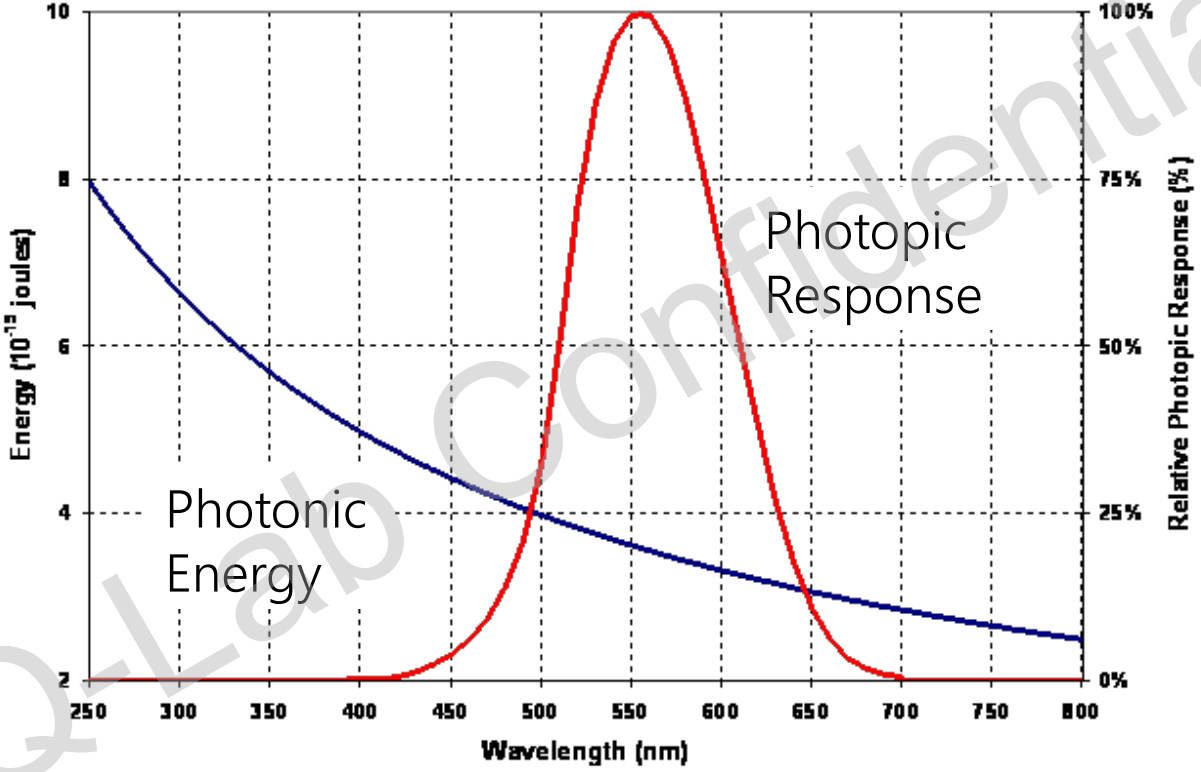
Irradiance (W/m^2) at each wavelength
×
Photopic Response (lumens/W) at wavelength
=
Illuminance ($lumens/m^2$) or lux

Example:

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

Sum the values at each wavelength, multiply 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

光谱给出了每个波长下的辐照度（W/m2）

- Sum irradiance at wavelengths 300-400 nm (Total UltraViolet or "TUV")

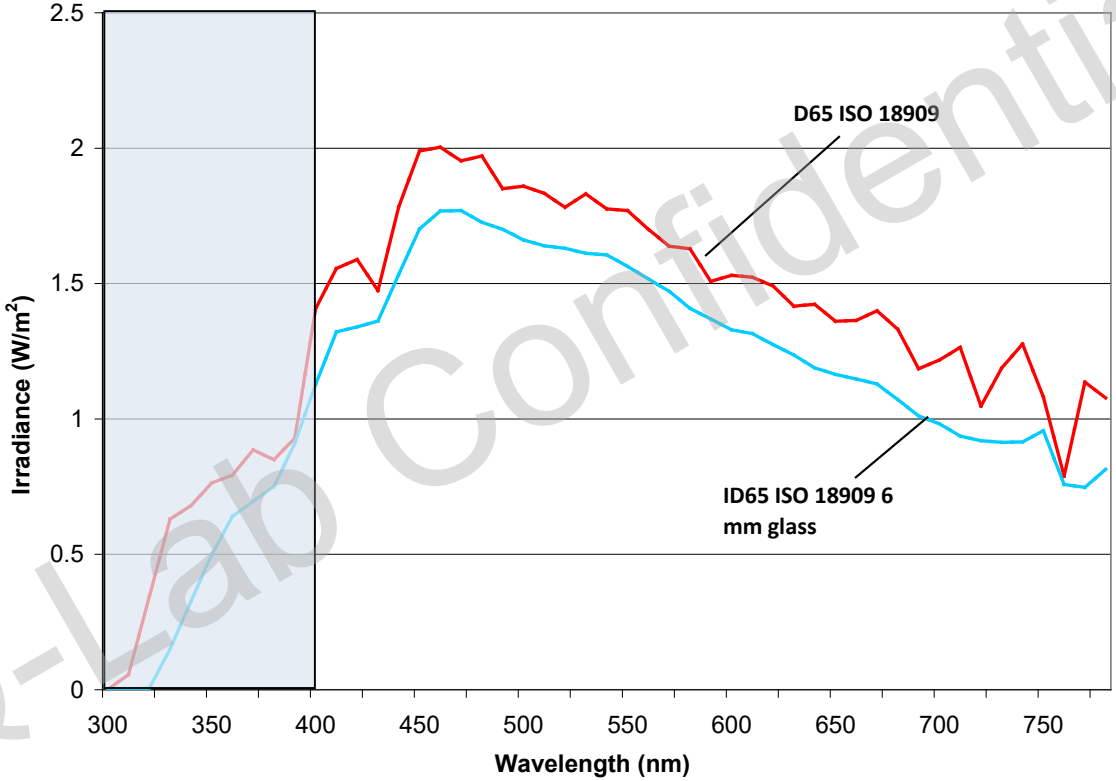
在紫外波段（300-400nm）加总辐照度

- Multiply this number by exposure time measured in hrs

  40 W/m^2 × 10 hours = 400 W-hours/m^2

辐照度和时间的乘积

Total UV Exposure (TUV, 300-400 nm)



ICH Guidelines

Temperature

Temperature is not specified, however …

温度没有规定，然而。。

- Thermal degradation should be evaluated separately in heat aging tests, not during lightfastness testing. Therefore, testing at normal room temperature ranges is desirable

热老化应该在烘箱中评估，而不是在光老化测试中。在一般室温环境下测试是理想的。

- Room temperature testing requires chilling the air circulated through the chamber

控制在室温范围需要制冷机

ICH Guidelines

Performing Option 1

- Q-SUN Xe-1-BCE
- Window – Q Filter
  (ID65 3 mm glass spectrum
- 420 nm irradiance control point, 1.10 W/m2/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

为了减少紫外曝晒，分成两部分执行

- Part 1: Run until 200 W-hr/m^2 TUV exposure, using Window-Q Filters

第一步，使用窗过滤器，累计到紫外剂量200 W-hr/m^2

- Part 2: Add a UV Blocking filter, recalibrate, and run to achieve 1.2 million Lux-hours (no additional TUV)

第二步，加紫外阻隔滤片，重现校准，累计到可见光剂量1.2 million Lux-hours

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

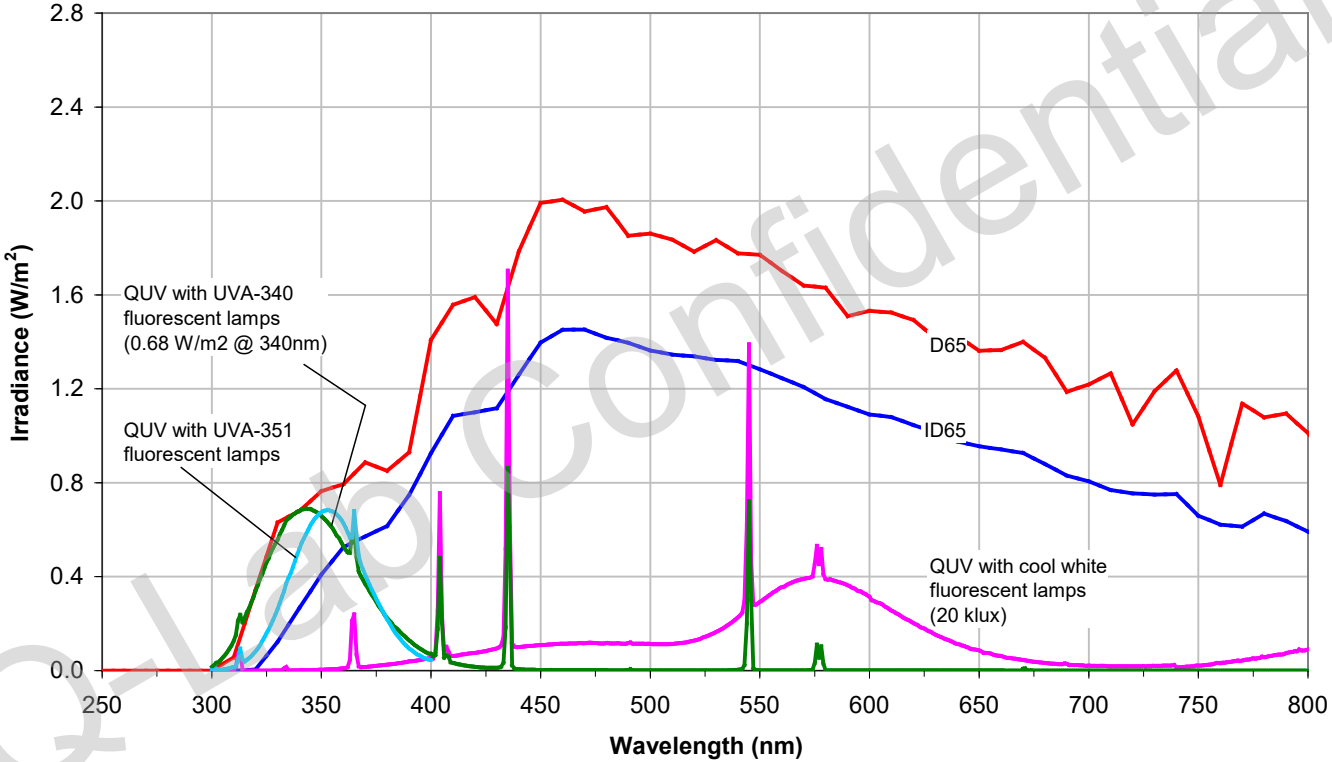
ICH Guidelines

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
光稳定性测试最佳实践

Best Practices And Practical Considerations

1. Perform natural exposures

自然曝晒

- Necessary for understanding accelerated results

理解加速老化测试结果

- Does lab test correctly rank material performance?

和实验室数据是否有好的相关性？

Miami outdoor Exposures

迈阿密的户外曝晒

Best Practices And Practical Considerations

2. **Test until failure** (forced degradation)
晒到完全失效

- Required for drug products
  - Identify impurities resulting from photodegradation
    识别光降解产生的杂质
  - Determine degradation pathways
    确定降解的路径
- Necessary for developing rank order performance
  建立排序相关性

Best Practices And Practical Considerations

3. Expose a control with your test specimen
使用参比材料

- Use Control Material of Known Durability
使用的参比材料是已经熟知其实验室和户外表现
  - Outdoor performance
  - Lab performance
- Similar Composition to Test Material
类似配方的测试材料
- Similar Degradation Mode to Test Material
类似的老化模式

Benefits of a Control

- Compare performance of control to a known material

和已经性能的参照样做比对

- Allows confidence in lab exposure

增加实验室的测试可行度

- Assure that laboratory tester is operating properly

确保实验室机器正常工作

Best Practices And Practical Considerations

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

最终成品件的测试

Whole Product Testing

Q-SUN Xe-3
3200 cm^2

Q-SUN Xe-1
1100 cm^2

Best Practices And Practical Considerations

5. Use realistic temperatures to prevent unrealistic failures

测试温度充分考虑实际情况

Testing with a chiller system (Xe-1 or Xe-3) allows for higher irradiance while maintaining cool temperatures

使用配置制冷机的氙灯设备，可以在较高辐照水平下保证比较低的测试温度

Thank you for your attention!

Questions?

Send your inquiry to:
kqu@q-lab.com

Q-Lab中国微信公众账号:
耐候腐蚀设备及测试专家

- ✓ 技术研讨会、网络研讨会信息
- ✓ 老化及腐蚀技术文章、最新测试标准解读等
- ✓ 相关技术问题，也可通过平台留言，我们会在24小时内和您联系

扫一扫，关注我们