Light Stability Testing of Home and Personal Care Products
个人护理品光稳定性测试

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Technical Marketing Specialist
Q-Lab Corporation

Q-Lab Corporation

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

Cleveland,Ohio
Headquarters &
Instrument Division

Bolton, England
Q-Lab Europe

Shanghai, China
Q-Lab China

Saarbrücken Germany

Q-Lab Outdoor Weathering Sites

Miami, Florida

Phoenix, Arizona

Cleveland, Ohio

Perfume is expensive. As a consumer, will you buy the bottle on the right?
香水是价格很贵的产品，作为消费者，您会购买最右边的那瓶吗？

Same Medicine，
Different Bottles
同样的药不同的瓶子

今年买的，
有色瓶

去年买的，
透明瓶

此药品的光稳定性不好？
换包装之后，是否提高了光稳定性?
换包装花了多少钱？

Question 1

- 您目前是否已开展相关的光稳定性测试？

a. 有
b. 客户有要求，准备开展
c. 暂无

Agenda

- What is Light Stability 什么是光稳定性
- Why test 为什么要做测试
- ICH Guidelines ICH 指南解读
- Xenon Arc Testing 氙灯测试
- Fluorescent UV & Cool White Testing 紫外，冷白荧光灯测试
- Natural Exposures 自然曝晒
- Points to Consider when Testing 测试中的重要点

Light Stability
光稳定性

Sunlight 阳光

Artificial Light
人工光源

Why test
为什么要做测试

- Meet Specifications 满足测试标准
- Avoid Catastrophes 避免风险
- Enhance your Reputation 提高声誉
- Verify Supplier Claims 供应商验证
- Big Saving on Material Cost 降低材料成本
- Less reworks in stability programs if degradation is understood at an early stage 利用前期的测试避免后期的重复劳动

Light Stability Testing Methods
几种常用的测试标准

- ICH Guideline for the Photostability Testing of New Drug Substances and Products
- ISO 24443 （in vitro） & ISO 24444 (in vivo)
- FDA 21 CFR Parts 201 and 310 (June, 2011)
- Measurement of UVA: UVB Ratios According to the Boots Star Rating System
- COLIPA Method for in vitro determination of UVA protection, 2011

ICH Guidelines

– International Conference on Harmonization: Guidelines for the Photostability Testing of New Drug Substances and Products
– Joint effort of U.S., European, Japanese regulatory agencies
– New products and drug substances should not exhibit “unacceptable change” when exposed to light

ICH Guidelines

1. Test the DRUG SUBSTANCE 原料
2. Test the exposed DRUG PRODUCT outside of the immediate pack and if necessary 包装前
3. Test the Drug Product in the immediate pack; and if necessary 初级包装
4. Test on the Drug Product in the marketing pack 上市销售包装

Expose to Light
光照


- 在仓储，运输，零售过程中会接触到光照
- 即使是不透明包装，在使用的时候也会见光

Note from EMEA(欧洲药品管理局): The European Agency for the Evaluation of Medicinal Products. February 2003.

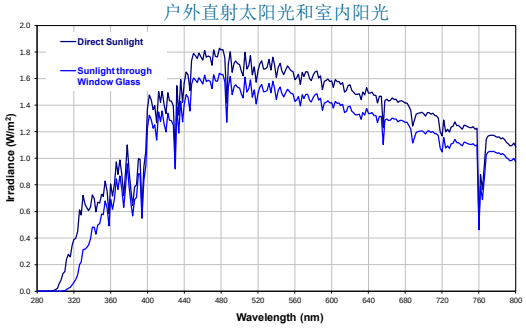
…To provide evidence on how the quality of a drug substance or drug product varies with time under the influence of a variety of environmental factors such as

Temperature, 温度
Humidity,湿度
and Light…光照

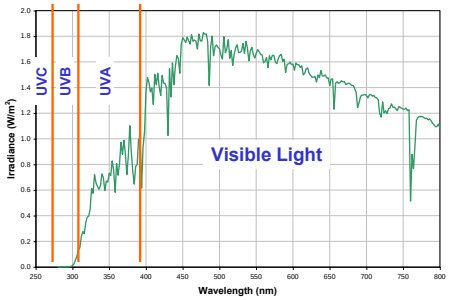
Sunlight

- Direct 户外直射太阳光
- Filtered through window glass 透过窗玻璃的太阳光

Sunlight--Direct and Through Window Glass
户外直射太阳光和室内阳光

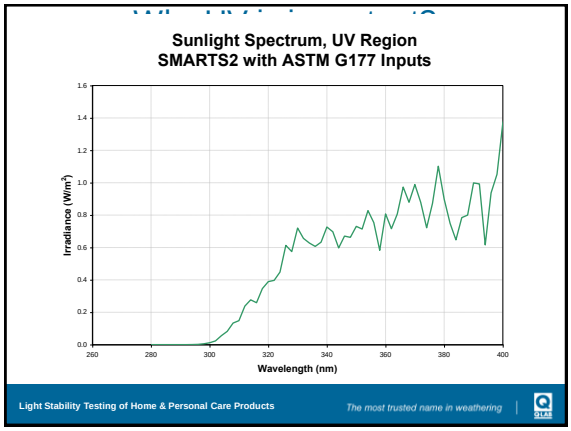


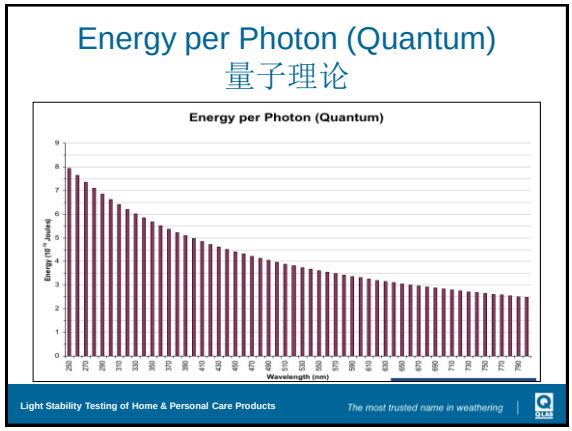
Sunlight Spectrum, SMARTS2 with ASTM G177 Inputs

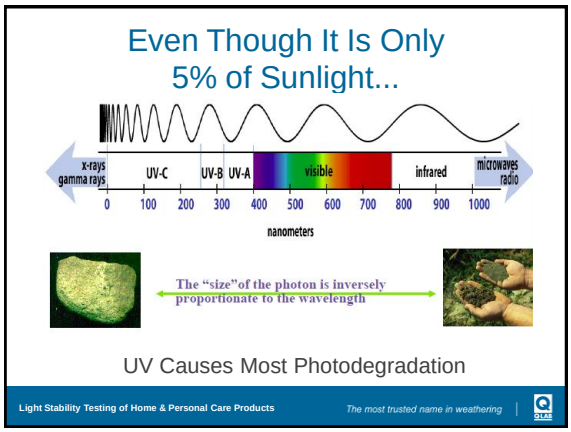


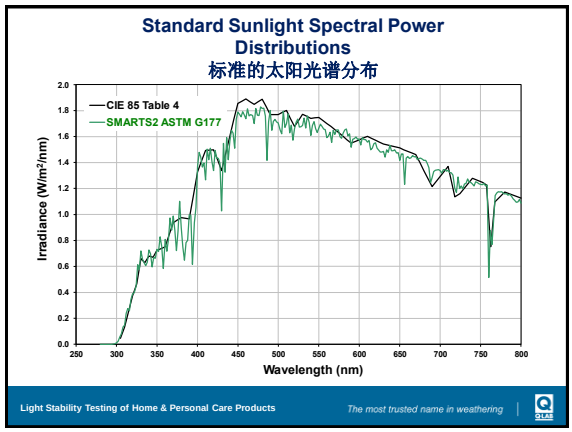
Wavelength Regions of UV
紫外线的波长范围

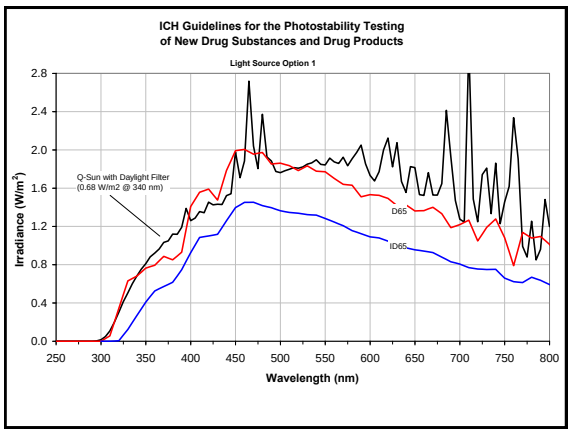
UV - C **100 – 280 nm**	**Found in outer space**
UV – B **280 – 315 nm**	**Includes shortest wavelengths at earth's surface: severe polymer damage; absorbed by window glass**
UV – A **315 – 400 nm**	**Causes polymer damage**


Sunlight Spectrum, UV Region
SMARTS2 with ASTM G177 Inputs
Irradiance (W/m²)
Wavelength (nm)
Light Stability Testing of Home & Personal Care Products
The most trusted name in weathering


Energy per Photon (Quantum)
量子理论
Energy per Photon (Quantum)
Wavelength (nm)
Light Stability Testing of Home & Personal Care Products
The most trusted name in weathering

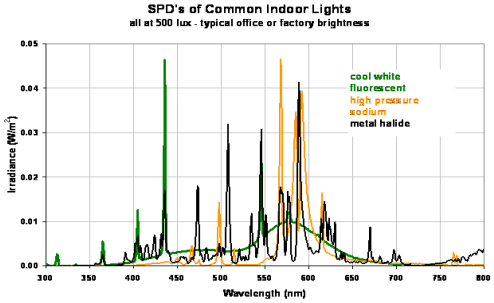

Even Though It Is Only
5% of Sunlight...
x-rays
gamma rays
UV-C
UV-B
UV-A
visible
infrared
microwaves
radio
0 100 200 300 400 500 600 700 800 900 1000
nanometers
The "size" of the photon is inversely proportionate to the wavelength
UV Causes Most Photodegradation
Light Stability Testing of Home & Personal Care Products
The most trusted name in weathering


Standard Sunlight Spectral Power
Distributions
标准的太阳光谱分布
CIE 85 Table 4
SMARTS2 ASTM G177
Irradiance (W/m²/nm)
Wavelength (nm)
Light Stability Testing of Home & Personal Care Products
The most trusted name in weathering

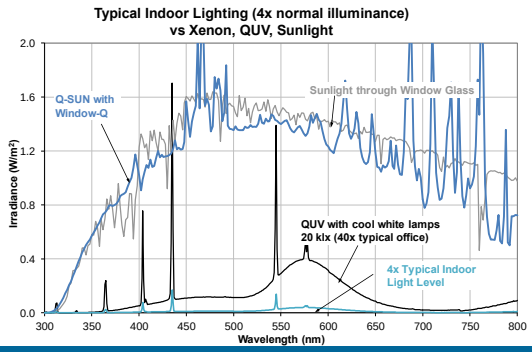

ICH Guidelines for the Photostability Testing
of New Drug Substances and Drug Products
Light Source Option 1
Q-Sun with Daylight Filter
(0.68 W/m2 @ 340 nm)
D65
ID65
Irradiance (W/m²)
Wavelength (nm)


Interior Lighting室内光源
Light Stability Testing of Home & Personal Care Products
The most trusted name in weathering

Indoor Lighting in Store
商超中的光源



太阳光和商超冷白荧光灯的比较



How to Define Visible Light
如何定义可见光

Lux is a unit of illumination (visible region)

Luminance is how ***Bright*** a light source looks. It is **not** a direct measure of the energy of light, but rather of the illumination it provides as perceived by the human eye.

流明是通过人眼对于光的敏感程度而定义的

Irradiance is the rate at which light energy falls on a surface, per unit area. W/m2 (UV region)

辐照度是光的能量落在单位面积上的功率

The relation between illuminance and Irradiance
流明和辐照度的联系

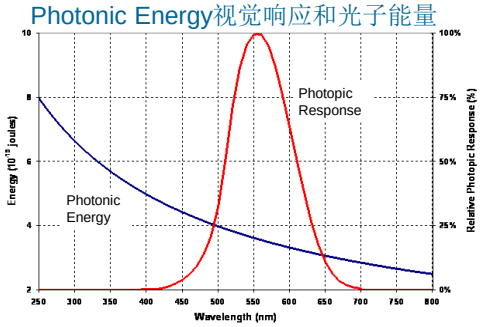
Example:

Wavelength (nm)	Photopic Response (lumens/W)		Irradiance (W/m^2)		Illuminance ($lumens/m^2$)(lux)
555	683.00	x	0.33	=	227.24

Now, sum up the value at each wavelength.

（对于人眼最敏感的555nm的黄绿光，1W = 683 lm，也就是说，1W的功率全部转换成波长为555nm的光，为683流明。）

Photopic Response &
Photonic Energy视觉响应和光子能量



Question 2

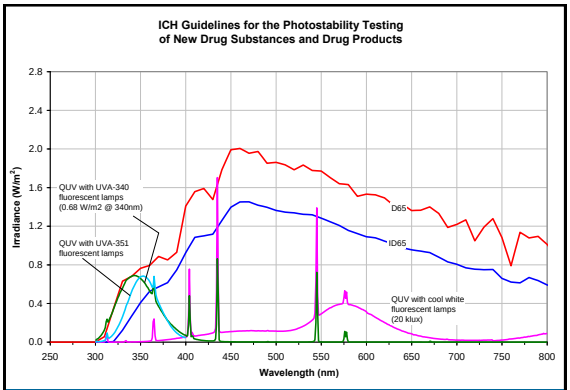
- ID65 主要是模拟以下哪种光源？

a. 户外直射太阳光
b. 透过窗玻璃的室内太阳光
c. 冷白荧光
d. 近紫外灯

ICH Guidelines

Two Exposure Options

1. D65/ID65 light source 近似D65/ID65光源
   - "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" 近似冷白荧光灯和近紫外灯



ICH Guidelines

Exposure Criteria曝晒要求

Expose samples to light providing illumination of not less than

1.2 million lux hours [400-800nm]

And

Integrated near UV of not less than 200 Watt hour/m2 [300-400nm]

ICH Guidelines

- Temperature not specified温度控制没有特别要求, but
  - Thermal degradation is a separate test, therefore…
  - Testing at normal room temperature ranges is desirable
    - Room temperature testing requires chilling the air circulated through the chamber使用制冷系统是测试温度与室温接近

Temperature Control
温度控制

- Black panel 黑板温度计
  - Hotter than ambient in sunlight
  - Not necessarily same as specimen temperature
  - Exists for test repeatability and reproducibility
- Chamber air 箱体空气温度
  - Controlled somewhat independently
  - More relevant for some applications
- Chiller System 制冷系统
  - Removes heat to allow normal indoor temperatures inside xenon arc test chamber

Black Panel Temperature
黑板温度计

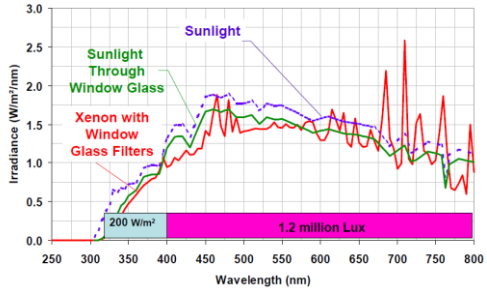
Two types of black panels!

ICH Guidelines

One possible way to run Option 1 方法一的实现

- Xenon arc test chamber equipped with a chiller氙灯老化箱
- Window glass filter (3 mm spectrum)窗过滤片
- 420nm irradiance control point 420nm 辐照度控制点

Xenon lamp with Window Glass Filter
经窗玻璃过滤片的氙灯



Optical Filters
过滤片种类

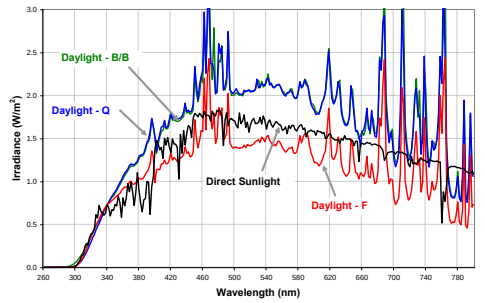
Daylight Filters 户外直射
(exterior exposures)

Window Glass 透过窗玻璃
(indoor exposures, textiles, inks, etc.)

Extended UV 紫外加强
(automotive, aerospace, etc.)

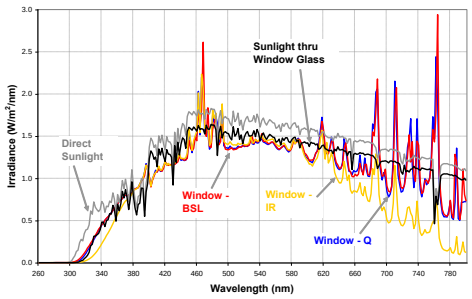
Xenon with Daylight Filters

Spectral Power Distribution of Q-Sun with Daylight Filters



Xenon with Window Filters

Spectral Power Distribution of Q-Sun with Window Filters



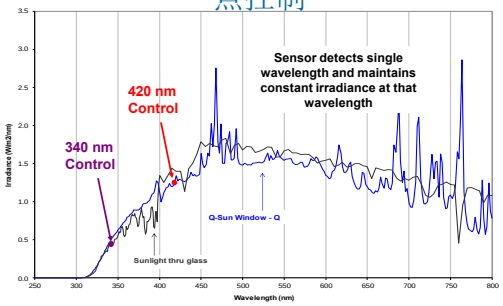
Irradiance Control辐照度控制

- Narrow Band点控制
  - 340 nm
  - 420 nm
- Total UV (300-400 nm) Wide Band波段控制
- Global (300-800 nm) – not recommended

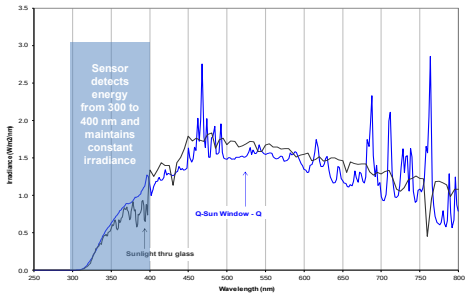
不推荐300-800nm的控制方式

  - The xenon lamps aging will shorter the UV energy 由于灯管的老化会导致UV辐照能量的衰减

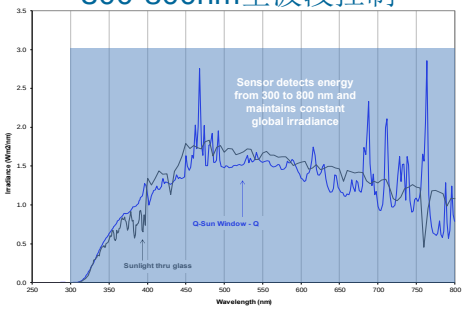
Narrow Band Control
点控制



Wide Band Control – TUV
TUV短波段控制



Global Control (300-800 nm)
300-800nm全波段控制



Example: Window B/SL Filter
下面代表的是相同的辐照度:

Control Point	Irradiance
340 nm	0.35 W/m^2
420 nm	0.95 W/m^2
TUV (300-400 nm)	43 W/m^2
Global (300-800 nm)	442 W/m^2

Question 3

以下哪种辐照度控制方法会因为灯管的老化而导致紫外辐照度的极大衰减?

a. 340nm 控制
b. 420nm 控制
c. 300-400 TUV控制
d. 300-800nm 全波段控制

One possible way to run Option 1
ICH Guidelines
利用氙灯试验箱来实现方法一

- Q-SUN Xe-1BC
- Window – Q Filter窗过滤片
- 420 nm irradiance control point 420nm点控制
- Chamber Air temperature control 箱体空气温度控制

Q-Sun Xenon Test Chamber
氙灯试验箱内部曝晒区域

ICH Guidelines

Test Cycle – Option 1
方法一的设置条件

- 1.10 W/m^2 set point (at 420 nm)
- 25°C Chamber Air temperature
- Run Test for 13.1 hours

- 650 Watt-hours UV (225% more UV than required)
- 1.2 million lux-hours

Irradiance & Test Time
辐照度和测试时间
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	1.2 million	647
0.70 W/m^2	20.7	1.2 million	647
0.80 W/m^2	18.1	1.2 million	647
0.90 W/m^2	16.1	1.2 million	647
1.00 W/m^2	14.5	1.2 million	647
1.10 W/m^2	13.1	1.2 million	647

Irradiance & Test Time
辐照度和测试时间
Option 1, Q-SUN with Window-Q

– *No single light source can meet the visible light exposure conditions without significant "over-exposure" of the UV portion*

所有光源在满足可见光曝晒的同时，已远远超过了UV曝晒的要求

– *"Over-exposure" is perfectly acceptable*

过度曝晒是可接受的

Option 1 Benefits/Drawbacks
option 1 的优点和缺点

- Benefits 优点

– All are single lamp methods 只用了一种灯管：氙灯
– Test both for UV or Lux requirement 氙灯满足了UV和Lux的要求
– Quick Test 测试快

- Drawbacks 缺点

– different filter, different SPD
– UV rich exposures 紫外 过量

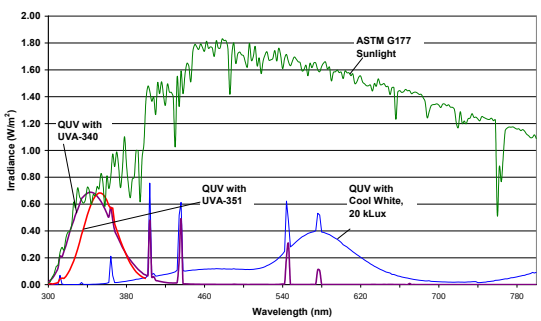
ICH Guidelines

Test Cycle – Option 2

Step 1: QUV with cool white lamps冷白荧光灯
Set Point: 20,000 lux
Time: 60 hours

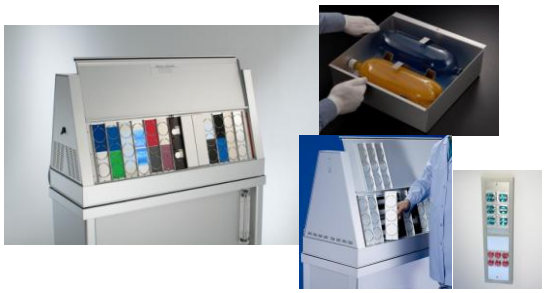
Step 2: QUV with UVA-351 lamps UVA-351灯管
Set Point: 0.55 W/m^2 @ 340 nm
Time: 4 hours

QUV Spectra vs. ASTM G177 Sunlight



QUV Fluorescent UV Weathering Tester and Cool White Light Stability Tester
紫外老化箱，既可以装紫外灯管，也可以装冷白荧光灯管

QUV Specimen Holders样品架



Option 2 Benefits/Drawbacks

- **Benefits**

– Inexpensive lamps 运行成本低，灯管便宜

- **Drawbacks**

– Use of two lamps makes sequence of exposure an issue 需要用两种灯管完成试验

Natural Exposures
自然曝晒

Natural Exposures

- Benchmark Commercial Sites标准商业户外曝晒场
  - South Florida
  - Arizona Desert
  - Inexpensive
  - Reliable
  - Extreme environments create acceleration
- At your own facility 在自家工厂，阳台
  - “Scientific Window Sill Testing”
  - Convenient
  - Projects sometimes forgotten

DIY Natural Exposures
自建的玻璃房

Points to Consider when Testing- Control
测试中需要利用“参照样品”

Expose a control with your test specimen
利用参照样品

- Use Control Material of Known Durability
  - Outdoor performance 户外的表现
  - Lab performance 实验室的表现
- Similar Composition to Test Material 近似成分
- Similar Degradation Mode to Test Material近似老化模式
- Competitor products 竞争对手产品

Benefits of a Control
利用参照样品的好处

- Compare performance of control to a known material与参照样品比较
- Allows confidence in lab exposure增加测试可信
- Assure that lab tester is operating properly确保设备运行正常

How to use Control Material
如何利用参照样品

- Selected a control product 选择参照样品
- Included control with all new product testing 和新产品一起测试
- Test 36 hours 测试一定的时间
- If new product fails, add lightfastness ingredients 添加或减少添加剂
- Retest 重新测试

Best Practices for Light Stability Testing

4. Test your product “In the package” in order to simulate the actual service environment.
最终上市销售的成品测试

Chamber Size and Capacity

Q-SUN Xe-3HC

3200 cm²

Q-SUN Xe-1BC

1100 cm2

Whole Product Testing

Q-SUN Xe-3

Q-SUN Xe-1

3D Parts Holders

Inter-Laboratory Comparisons

What is important 什么是关键参数

- Light Spectrum 光谱
  - Not all Window or Daylight filters are the same
- Irradiance Control & Lamp Aging 辐照度控制和灯管老化
  - Choose narrow band or TUV set points辐照度选择点控制或TUV控制

Questions?

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