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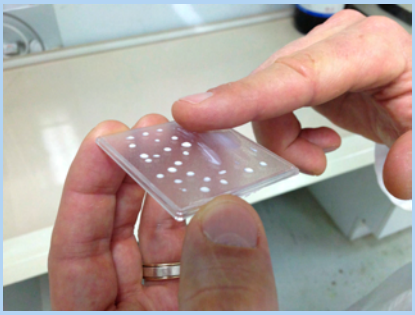
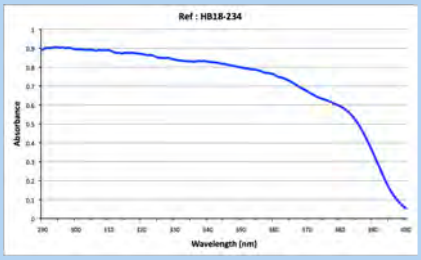
FDA Broad Spectrum Testing

STEPS



1. Product Sample Application

2. Spectrophotometric measurement



3. Absorbance Spectra

This Test qualifies Sunscreens for sale in the USA and is an FDA requirement.

Supportable Claims

- Broad Spectrum - FDA
- Critical Wavelength - FDA

FDA Requirements

This test fits the requirements of the FDA Rule for Labeling and Effectiveness Testing; Sunscreen Drug Products for Over-the-Counter Human Use. Based on the product showing a Critical Wavelength above 370 nm, "Broad Spectrum" is a permitted claim for sunscreen products with an SPF of at least 15.

Method Outline

The method is baed on that described in ISO 24443 ,but will differing film thickness and pre-irradiation requirements. Pre-irradiation light challenge of the sample film is to a fixed dose of 4 MED's independent of the claimed SPF of the product.
4 replicate measurements on 4 separate PMMA plates are made post irradiation, on non-overlaying areas of each of the plates.
Transmittance of a dried down film is measured between 290 nm and 400 nm. Requirements can be extrapolated by conversion of the ratios determined.

The spectral curve is measured, using Labsphere 2000 or Shimadzu spectrophotometer which has been fitted with an integrating sphere device.
The substrate for measurement is moulded Polymethylmethacrylate (PMMA) Plates.
A thin film of the test product, at a thickness of 0.75 mg/sq cm, is applied, by a standard application technique. This involves applying a series of around 30 dots over the area of the plate and then rubbing out evenly with the finger which has been pre-impregnated to saturation with the same product.

Measurements

Measurements are taken against a matching blank glycerin loaded PMMA plate, at increments of 1 nm between the range of 290 and 400 nm. Calculations are completed by input of the data onto a standardised spreadsheet.

Reporting

An FDA compliant report is provided in standard spreadsheet format. Critical Wavelength is reported. Spectral and calibration data is provided.

References

1.FDA Final Rule 2011 - see page 2

2.ISO 24443 - Determination of Sunscreen UVA Protection In vitro

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Extract from FDA Monograph

Federal Register / Vol. 76, No. 117 / Friday, June 17, 2011 / Rules and Regulations

(j) Broad spectrum test procedure. (1) UV Spectrometry. (i) Plate. Use opticalgrade polymethylmethacrylate (PMMA) plates suitable for UV transmittance measurements. The plate should be roughened on one side to a three dimensional surface topography measure (Sa) between 2 and 7 micrometers and must have a rectangular application area of at least 16 square centimeters (with no side shorter than 4 cm).

(ii) Sample holder. The sample holder should hold the PMMA plate in a horizontal position to avoid flowing of the sunscreen drug product from one edge of the PMMA plate to the other. It should be mounted as close as possible to the input optics of the spectrometer to maximize capture of forward scattered radiation. The sample holder should be a thin, flat plate with a suitable aperture through which UV radiation can pass. The PMMA plate should be placed on the upper surface of the sample holder with the roughened side facing up.

(iii) Light source. The light source should produce a continuous spectral distribution of UV radiation from 290 to 400 nanometers.

(iv) Input optics. Unless the spectrometer is equipped with an integrating sphere, an ultraviolet radiation diffuser should be placed between the sample and the input optics of the spectrometer. The diffuser will be constructed from any UV radiation transparent material (e.g., Teflon® or quartz). The diffuser ensures that the radiation received by the spectrometer is not collimated. The spectrometer input slits should be set to provide a bandwidth that is less than or equal to 1 nanometer.

(v) Dynamic range of the spectrometer. The dynamic range of the spectrometer should be sufficient to measure transmittance accurately through a highly absorbing sunscreen product at all terrestrial solar UV wavelengths (290 to 400 nm).

(2) Sunscreen product application to PMMA plate. The accuracy of the test depends upon the application of a precisely controlled amount of sunscreen product with a uniform distribution over the PMMA plate. The product is applied at 0.75 mg per square centimeter to the roughened side of the PMMA plate. The sunscreen product should be applied in a series of small dots over the entire PMMA plate and then spread evenly using a gloved finger. Spreading should be done with a very light spreading action for approximately 30 seconds followed by spreading with greater pressure for approximately 30 seconds. The plate should then be allowed to equilibrate for 15 minutes in the dark before the pre-irradiation described in paragraph (c) of this section.

(3) Sunscreen product pre-irradiation. To account for lack of photostability, apply the sunscreen product to the PMMA plate as described in paragraph (b) of this section and then irradiate with a solar simulator described in section 352.70(b) of this chapter. The irradiation dose should be 4 MEDs which is equivalent to an erythemal effective dose of 800 J/m2 (i.e., 800 J/m2-eff).

(4) Calculation of mean transmittance values. After pre-irradiation described in paragraph (c) of this section, mean transmittance values should be

determined for each wavelength λ over the full UV spectrum (290 to 400 nanometers). The transmittance values should be measured at 1 nanometer intervals. Measurements of spectral irradiance transmitted for each wavelength λ through control PMMA plates coated with 15 microliters of glycerin (no sunscreen product) should be obtained from at least 5 different locations on the PMMA plate [C1(λ), C2(λ), C3(λ), C4(λ), and C5(λ)]. In addition, a minimum of 5 measurements

of spectral irradiance transmitted for each wavelength λ through the PMMA plate covered with the sunscreen product will be similarly obtained after pre-irradiation of the sunscreen product [P1(λ), P2(λ), P3(λ), P4(λ), and P5(λ)]. The mean transmittance for each wavelength,
is the ratio of the mean of the C(λ) values to the mean of the P(λ) values, as follows:

$$\overline{T(\lambda)} = \frac{\sum_1^n P(\lambda)/n}{\sum_1^n C(\lambda)/n}$$

Where n ≥ 5
(5) Calculation of mean absorbance values. (i) Mean transmittance values, are converted into mean absorbance values,
at each wavelength by taking the negative logarithm of the mean transmittance value as follows:
(ii) The calculation yields 111 monochromatic absorbance values in 1 nanometer increments from 290 to 400 nanometers.
(6) Number of plates. For each sunscreen product, mean absorbance values should be determined from at least three individual PMMA plates. Because paragraph (d) of this section requires at least 5 measurements per plate, there should be a total of at least 15 measurements.
(7) Calculation of the critical wavelength. The critical wavelength is identified as the wavelength at which the integral of the spectral absorbance curve reaches 90 percent of the integral over the UV spectrum from 290 to 400 nm. The following equation defines the critical wavelength:
Where λc = critical wavelength
A(λ) = mean absorbance at each wavelength
dλ = wavelength interval between measurements
A mean critical wavelength of 370 nm or greater is classified as broad spectrum protection.