Tech Note
SPECORD PLUS

Validating Instrument Performance According to USP Chapter <857> Guidelines with SPECORD PLUS UV/Vis Spectrophotometers

Introduction

UV/Vis spectroscopy is one of the most widely used analytical techniques in laboratories of the pharmaceutical industry. Quality control and assurance analysis of pharmaceutical products are operated under stringent quality guidelines established by international bodies like United States Pharmacopeia (USP) to ensure that Good Manufacturing Practices (GMP) are followed.

Periodical validation of analytical instrument performance on its operational range is an essential part to ensure the reproducibility and reliability of the results given. The USP chapter <857> (43rd edition) describes performance qualification parameters for a UV/Vis spectrophotometer.

Your Benefits

- Confirm device performance according to USP specifications
- ASpect UV software intuitively guides you through measurements
- Software features include comprehensive user management, secure analysis, and data processing

USP <857> Validation

Validation according to USP includes the following parameters:

- Control of wavelength
- Control of absorbance and photometric linearity (new)
- Limit of stray light
- Resolution

USP <857> involves validation of the spectrometer in the operating range, i.e. the range in which the expected measured values are found. In order to cover an extended measuring range, both in the wavelength and the absorbance, a larger number of methods and certified reference materials (CRM) are suggested. It is the responsibility of the user to select the CRMs and methods suited to the required operating range. The USP <857> also permits, in addition to the standards it recommends, alternative CRMs once their certification can be guaranteed.

Control of wavelengths

This qualification study ensures that the accuracy of wavelengths over intended operational range is within acceptable limits. Accuracy and precision of wavelengths are studied in the operational range of instrument and thereby different CRMs are required.

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CRM	Accuracy	Precision
Holmium in perchloric acid.	200–400 nm ± 1 nm 400–780 nm ± 2 nm	≤ 0.5 nm
Cerium oxide solutions	200–400 nm ± 1 nm	≤ 0.5 nm
Didymium solutions or glasses	400–900 nm ± 2 nm	≤ 0.5 nm
Holmium oxide glass filter	200–400 nm ± 1 nm 400–780 nm ± 2 nm	≤ 0.5 nm
D2 emission lines	486.0 and 656.1 nm ± 2 nm	≤ 0.5 nm

Control of absorbance and photometric linearity

This qualification study ensures that the transmittance accuracy, precision, and linearity of a given system are within acceptable limits. Different concentration ranges of CRMs are required to check the absorbance range and photometric linearity. $K_2Cr_2O_7$ solution (from 20 mg/L to 200 mg/L), neutral density glass filters, traceable to NIST, have to be used based on the operational analytical range. For photometric linearity, at least three concentration ranges in the operational range need to be analysed.

CRM	Accuracy	Precision
$K_2Cr_2O_7$ solution (235; 257; 313; 350 nm)	≤ 1 A: ± 0.010 A > 1 A: ± 1% A	≤ 1 A: ≤ ± 0.005 A > 1 A: ≤ ± 0.5% A
Neutral density glass filters (440; 465; 546,1; 590 and 635 nm)	≤ 1 A: ± 0.008 A > 1 A: ± 0.8% A	≤ 1 A: ≤ ± 0.005 Abs > 1 A: ≤ ± 0.5% A

Limit of Stray Light

Filters with a sharply defined spectrum (cut-off filters) are used for the stray light measurements, which block any light from passing through below a certain wavelength. Ideally, filters with a cut-off wavelength that lies as close as possible above the required wavelengths are used.

The USP <857> offers two options for determining the stray light. In the method *10 mm versus 5 mm*, a cell with 5 mm path length is first measured as a reference (filled with the filter solution) and then a 10 mm cell (filled with the same solution) as a sample. The resulting peak is used to determine the position and height of the maximum absorbance and the associated stray light value S_λ. In the second method *10 mm versus 10 mm* H_2O, the filter solution is measured against water as reference.

In both measurements, cells with a pathlength of 10 mm are used. The absorbance measured at the certificate wavelength (at the edge of an absorbance peak) must be greater than or equal to 2 A.

CRM	Limits
12 g/L KCl (190 nm–210 mm)	Method A: 10 mm versus 5 mm
10 g/L NaI (210 nm–280 nm)	Requirement: A ≥ 0.7 A
Acetone (250 nm–350 nm)	Method B: 10 mm versus 10 mm
50 g/L $NaNO_2$ (300 nm–420 nm)	Requirement: A max ≥ 2.0 A

Resolution

With the CRM for resolution, the spectral bandwidth of the photometer is validated. A 0.020% solution of toluene in hexane against hexane as a reference is measured and the absorption maximum (at approx. 269 nm) and the absorption minimum (at approx. 266 nm) are determined. From the ratio A269/A266 the resolving capacity is calculated.

The USP states that a slit of 2 nm or smaller is sufficient for most quantitative applications. For the SPECORD PLUS devices this means that the spectral bandwidth of 0.5/1/2 nm (SPECORD 210 and SPECORD 250 PLUS) and 1.4 nm (SPECORD 50 and SPECORD 200 PLUS) can be tested and that they meet the USP requirements.

Aspect UV Software

Analytik Jena provides solutions for all USP <857> compliance requirements with its software interface ASpect UV. The intuitive software for the SPECORD PLUS spectrophotometer series provides powerful tools for flexible and compliant analysis in highly regulated environments. With the comprehensive user management and AJ file protect, data is protected against arbitrary and unintentional manipulation, thus guaranteeing data integrity. More details on standard and additional measurements according to USP <857> can be found in the attached overview.

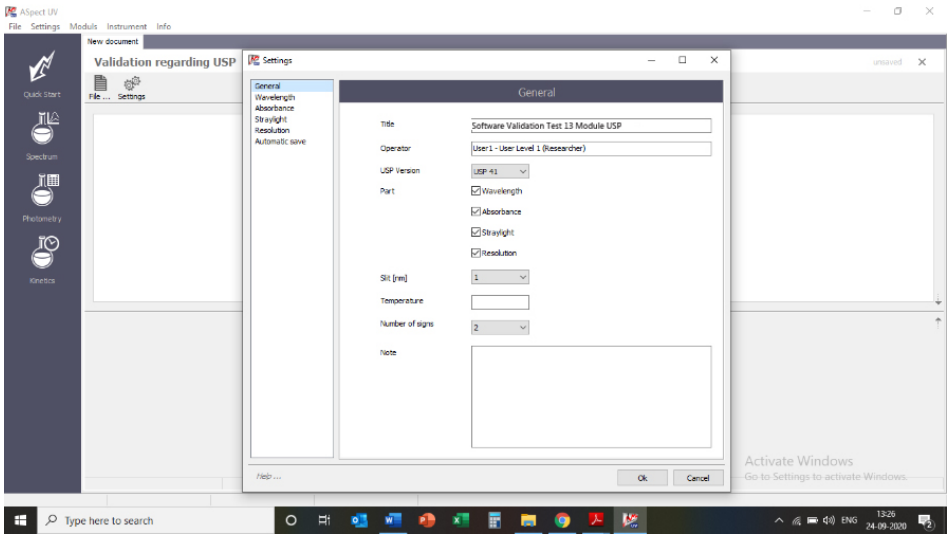

Figure 1 ASpect UV Validation Support Software Module acc. to USP <857>.

Standard measurements according to USP 857

Customer Operation Range	Up to version USP 41	From version USP 42
Wavelength range [nm]	279 nm to 864nm	279 nm to 875 nm
Photometry range [Abs]	UV range from 0.3 to 0.9 Abs VIS range 1.0 Abs	UV range from 0.1 to 1.45 Abs VIS range 0.25 to 1.0 Abs
Slit width [nm] (1 slit width per validation)	Slitting devices selectable between 0.5; 1 or 2 nm and with fixed slit device at 1.4 nm	

Certified Reference Material – Included, certified

	Selection	Parameter	Test equipment designation	Wavelength / Absorbance	
1	Included	Wavelength	Holmium oxide [F1]	279 nm	
				360 nm	
				453 nm	
				536 nm	
				637 nm	
2	Included from version USP 42	Wavelength	Didymium oxide [F7W]	329 nm	
				472 nm	
				512 nm	
				681 nm	
				875 nm	
3	Included up to version USP 41	Wavelength	Didymium oxide solution [UV25]	731 nm	
				740 nm	
				794 nm	
				801 nm	
				864 nm	
4	Included	Wavelength	D2E lines	486.0 nm	
				656.1 nm	
5	Included from version USP 42	Absorbance	Potassium dichromate 20 mg/l [UV14+UV20]	0.25 Abs (@ 235 nm)	
				0.29 Abs (@ 257 nm)	
				0.10 Abs (@ 313 nm)	
				0.22 Abs (@ 350 nm)	
6	Included	Absorbance	Potassium dichromate 60 mg/L [UV14+UV60]	0.75 Abs (@ 235 nm)	
				0.86 Abs (@ 257 nm)	
				0.30 Abs (@ 313 nm)	
				0.65 Abs (@ 350 nm)	

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	Selection	Parameter	Test equipment designation	Wavelength / Absorbance	
7	Included from version USP 42	Absorbance	Potassium dichromate 100 mg/L [UV14+UV100]	1.25 Abs (@ 235 nm)	
				1.45 Abs (@ 257 nm)	
				0.50 Abs (@ 313 nm)	
				1.07 Abs (@ 350 nm)	
8	Included from version USP 42	Absorbance	Neutral glass filter 0.25 Abs [F2]	0.25 Abs (@ 440 nm)	
				0.25 Abs (@ 465 nm)	
				0.25 (@ 546,1 nm)	
				0.25 Abs (@ 590 nm)	
				0.25 Abs (@ 635 nm)	
9	Included from version USP 42	Absorbance	Neutral glass filter 0.5 Abs [F3]	0.5 Abs (@ 440 nm)	
				0.5 Abs (@ 465 nm)	
				0.5 Abs (@ 546,1 nm)	
				0.5 Abs (@ 590 nm)	
				0.5 Abs (@ 635 nm)	
10	Included	Absorbance	Neutral glass filter 1.0 Abs [F4]	1.0 Abs (@ 440 nm)	
				1.0 Abs (@ 465 nm)	
				1.0 Abs (@ 546.1 nm)	
				1.0 Abs (@ 590 nm)	
				1.0 Abs (@ 635 nm)	
11	Included	Straylight	KCl (12 g/L) [UV1+UV12] or [UV1+UV1H]	Liquide filter vs. water or Liquide filter 10mm vs. 5mm	
12	Included	Straylight	NaI (10 g/L) [UV10+UV12] or [UV10+UV10H]	Liquide filter vs. water or Liquide filter 10mm vs. 5mm	
13	Included	Straylight	Aceton [UV19] or [UV19+UV19H]	Liquide filter vs. air or Liquide filter 10mm vs. 5mm	
14	Included	Straylight	$NaNO_2$ (50 g/L) [UV11+UV12] or [UV11+UV11H]	Liquide filter vs. water or Liquide filter 10mm vs. 5mm	
15	Included	Resolution	Toluol/Hexan [UV9+UV6]		

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Agreement on additional measurements according to USP 857

Certified Reference Material – optional, regularly certified

	Selection	Parameter	Test equipment designation	Wavelength / Absorbance	
16	#	Wavelength	lower UV [UV35]	201 nm	
				211 nm	
				222 nm	
				239 nm	
				252 nm	
17	Included from version USP 42 #	Wavelength	Didymium oxide [F7W]	329 nm	
				472 nm	
				512 nm	
				681 nm	
				875 nm	
18	#	Wavelength	Holmium oxide solution [UV5]	241 nm	
				249 nm	
				278 nm	
				287 nm	
				333 nm	
				345 nm	
				361 nm	
				385 nm	
				416 nm	
				451 nm	
				467 nm	
				485 nm	
				536 nm	
				640 nm	
19	Included up to version USP 41 #	Wavelength	Didymium oxide solution [UV25]	731 nm	
				740 nm	
				794 nm	
				801 nm	
				864 nm	

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	Selection	Parameter	Test equipment designation	Wavelength / Absorbance	
20	#	Wavelength	Hg emission lines	194.2 nm	
				237.9 nm	
				248.2 nm	
				(only slit is smaler than 2 nm) 253.7 nm	
				265.4 nm	
				289.4 nm	
				296.7 nm	
				302.15 nm	
				334.15 nm	
				365.0 nm	
				404.7 nm	
				407.8 nm	
				435.8 nm	
				546.1 nm	
				577.0 nm	
				579.1 nm	
				871.7 nm	
				1014.1 nm	
				1092.1 nm	
21	#	Absorbance	Niacin liquide filter 6 mg/L [UV59+UV51]	0.25 Abs (@ 213 nm)	
				0.25 Abs (@ 261 nm)	
22	#	Absorbance	Niacin liquide filter 12 mg/L [UV59+UV52]	0.51 Abs (@ 213 nm)	
				0.50 Abs (@ 216 nm)	
23	#	Absorbance	Niacin liquide filter 18 mg/L [UV59+UV53]	0.77 Abs (@ 213 nm)	
				0.75 Abs (@ 261 nm)	
24	#	Absorbance	Niacin liquide filter 24 mg/L [UV59+UV54]	1.04 Abs (@ 213 nm)	
				1.01 Abs (@ 261 nm)	
25	Included from version USP 42 #	Absorbance	Potassium dichromate 20 mg/L [UV14+UV20]	0.25 Abs (@ 235 nm)	
				0.29 Abs (@ 257 nm)	
				0.10 Abs (@ 313 nm)	
				0.22 Abs (@ 350 nm)	

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	Selection	Parameter	Test equipment designation	Wavelength / Absorbance	
26	Included from version USP 42 #	Absorbance	Potassium dichromate 100 mg/L [UV14+UV100]	1.25 Abs (@ 235 nm) 1.45 Abs (@ 257 nm) 0.50 Abs (@ 313 nm) 1.07 Abs (@ 350 nm)	
27	#	Absorbance	Potassium dichromate 160 mg/L [UV14+UV160]	2.00 Abs (@ 235 nm) 2.35 Abs (@ 257 nm) 0.80 Abs (@ 313 nm) 1.70 Abs (@ 350 nm)	
28	#	Absorbance	Potassium dichromate 200 mg/L [UV14+UV200]	2.50 Abs (@ 235 nm) 2.95 Abs (@ 257 nm) 1.00 Abs (@ 313 nm) 2.10 Abs (@ 350 nm)	
29	#	Absorbance	Neutral glass filter 0.04 Abs [F390]	0.04 Abs (@ 440 nm) 0.04 Abs (@ 465 nm) 0.04 Abs (@ 546.1 nm) 0.04 Abs (@ 590 nm) 0.04 Abs (@ 635 nm)	
30	Included from version USP 42 #	Absorbance	Neutral glass filter 0.25 Abs [F2]	0.25 Abs (@ 440 nm) 0.25 Abs (@ 465 nm) 0.25 (@ 546,1 nm) 0.25 Abs (@ 590 nm) 0.25 Abs (@ 635 nm)	
31	Included from version USP 42 #	Absorbance	Neutral glass filter 0.5 Abs [F3]	0.5 Abs (@ 440 nm) 0.5 Abs (@ 465 nm) 0.5 Abs (@ 546.1 nm) 0.5 Abs (@ 590 nm) 0.5 Abs (@ 635 nm)	
32	#	Absorbance	Neutral glass filter 2.0 Abs [F203]	2.0 Abs (@ 440 nm) 2.0 Abs (@ 465 nm) 2.0 Abs (@ 546.1 nm) 2.0 Abs (@ 590 nm) 2.0 Abs (@ 635 nm)	
33	#	Resolution	D2E lines	486.0 nm 656.1 nm	

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	Selection	Parameter	Test equipment designation	Wavelength / Absorbance	
34	#	Resolution	Hg lines	194.2 nm	
				237.9 nm	
				248.2 nm	
				(only slit is smaller than 2 nm) 253.7 nm	
				265.4 nm	
				289.4 nm	
				296.7 nm	
				302.15 nm	
				334.15 nm	
				365.0 nm	
				404.7 nm	
				407.8 nm	
				435.8 nm	
				546.1 nm	
				(only slit is smaler than 2 nm) 577.0 nm	
				(only slit is smaler than 2 nm) 579.1 nm	

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