UV-VISIBLE HARDWARE
OPERATIONAL QUALIFICATION

The Measure of Confidence

Agilent Enterprise Edition Compliance Services

Standard OQ Test Specifications for UV-Visible Spectrophotometers

The following tables describe the core hardware testing specifications for UV-Visible systems. See the corresponding attachment for a description of the optional tests available for UV-Visible systems, not part of the core program.

#	Core Test Name	Relevant Model	Setpoints	Lower Limits	Upper Limits
1				Accuracy	
	Wavelength Accuracy Source Line	Cary 50 | Cary 60	541.92 nm	<= 0.50 nm	n/a
		8453	486.00 nm	<= 0.20 nm	
			656.10 nm		
		Cary 100 | Cary 300	0.00 nm	<= 0.70 nm	
			486.00 nm	<= 0.20 nm	
			656.10 nm		
		Cary 4000 | Cary 5000 | Cary 6000i	0.00 nm	<= 0.08 nm	
			486.00 nm		
			656.10 nm		
		Cary 5000 | Cary 6000i	1312.2 nm	<= 0.40 nm	
			1968.3 nm		
2				Accuracy	
	Wavelength Accuracy Holmium Oxide in Perchloric Acid	Cary 50 | Cary 60 | Cary 100 | Cary 300 | Cary 4000 | Cary 5000 | Cary 6000i	241.15 nm	<= 1 nm	n/a
			287.15 nm		
			361.5 nm		
			536.3 nm	<= 3 nm	
		8453	241.10 nm	<= 0.7 nm	
			249.92 nm		
			278.06 nm		
			287.33 nm		
			333.42 nm		
			345.48 nm		
			361.23 nm		
			385.76 nm		
			416.45 nm		
			451.30 nm		
			467.88 nm		
			485.31 nm		
			536.80 nm		
			640.68 nm		

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Standard OQ Tests Specifications for UV-Visible Spectrophotometers (continued)

#	Core Test Name	Relevant Model	Setpoints		Lower Limits	Upper Limits
3					Resolution (Acc.)	
	Toluene/Hexane Resolution	Cary 50 | Cary 60 | Cary 100 | Cary 300 | Cary 4000 | Cary 5000 | Cary 6000i			>= 1.5	n/a
		8453			> 1.6	n/a
4					%Transmittance	
	Stray Light – Potassium Chloride	All models	198 nm		>= 0.0 %T	< 1.0 %T
5			Wavelength	Absorbance	Accuracy (Abs)	
	Photometric Accuracy Potassium Dichromate	All models	235 nm	0.747 Abs	<= 0.01	n/a
			257 nm	0.867 Abs		
			313 nm	0.292 Abs		
			350 nm	0.644 Abs		
			430 nm	0.954 Abs	<= 0.012	

See the corresponding attachment for a description of the optional tests available for UV-Visible systems, not part of the core program.

End of Section - Standard OQ Test Specifications for Agilent UV-Visible Systems.

OQ Test Design and Rationale for UV-Visible Spectrophotometers

Many GMP/GLP enforcement agency inspectors now ask firms to provide a risk assessment of their equipment and computer systems plus a science-based rationale for subsequent validation and qualification testing.

GENERAL RISK STATEMENT: Any HPLC/LCMS, GC/GCMS or UV-Visible system used for raw material testing or final drug product / medical device testing in GMP or used in formal GLP studies will likely fall into a HIGH RISK category. This risk assessment will imply the need for IQ & OQ & on-going qualification. ANY USER SPECIFIC RISK ANALYSIS SUPERCEDES THIS GENERAL RISK STATEMENT.

The rest of this section outlines the science-based rationale for each test in the Agilent hardware OQ plus a brief test design and procedure description.

The recommended set of hardware OQ tests described in this EQP derives from Agilent's interpretation of FDA, USP, and GAMP4 guidelines and other authoritative expert literature.

OQ test design incorporates both modular and holistic testing, which is a proven and regulatory acceptable approach. Wavelength tests are used for optical assembly modules. Remaining tests are used for the evaluation of the whole spectrophotometer.

Certified reference standards and calibrated traceable thermometers and digital flowmeters are used.

Considering the number of setpoints, parameters, and conditions of each recommended OQ test, the proven concepts of worst case, range, and representative have been applied. If a property or characteristic is known to have its worst performance at one end of a range of use, this is the setpoint that should be tested and other setpoints are not required. If a property or characteristic has no known worst case, testing at the high and low points of the range of use is required. If there are too many possible use cases and conditions to realistically test (and none is a worst case), a representative sample for test is the best approach.

OQ Test Design and Rationale for UV-Visible Spectrophotometers (continued)

1. Wavelength Accuracy – Source Lamp Emission Lines

Rationale: Wavelength Accuracy is performed as a verification of the xenon lamp performance in the Cary 50 and Cary 60 models or the deuterium lamp performance in all other models. The xenon lamp produces a photometric peak maximum at 541.92 nm. The deuterium lamp produces photometric peak maxima at 486.0 nm and 656.1 nm. In this test, Wavelength Accuracy is verified by scanning a spectrum a number of nanometers above and below the theoretical absorbance maxima for the deuterium lamp. The test passes if the reported wavelength maxima do not differ from the expected values by more than the accuracy limit. This test is applied to all UV/Vis spectrophotometers and meets all major pharmacopoeia and ASTM requirements.

Procedure: Power on and warm up the spectrophotometer for the appropriate time prior to testing. If a cell changer is present disable the cell changer for testing. Verify there are no samples in the reference and sample cell holders. Using the spectrophotometer's controlling data software (CDS) setup the Wavelength Accuracy Test for the Source Lamp Emission Line. Perform the test by following the software prompts. When the test is complete record the values to the test record.

2. Wavelength Accuracy - Holmium Oxide in Perchloric Acid Solution

Rationale: Wavelength Accuracy is calculated by comparing the reported lambda maxima values against the expected lambda maxima values of a Holmium Oxide in Perchloric Acid test solution (Holmium Perchlorate). The test passes if the reported wavelength maxima do not differ from the expected values by more than the accuracy limit. This test is applied to all UV/Vis spectrophotometers and meets all major pharmacopoeia and ASTM requirements.

Procedure: Power on and warm up the spectrophotometer for the appropriate time prior to testing. If a cell changer is present disable the cell changer for testing. Using the spectrophotometer's CDS setup the Wavelength Accuracy Test for the Holmium Oxide in Perchloric Acid (Holmium Perchlorate). Perform the test by following the software prompts. When the test is complete record the values to the test record.

3. Spectral Resolution

Rationale: Decreased spectral resolution can impact the photometric sensitivity and selectivity. Spectral Resolution is determined by calculating the peak-to-trough ratio between the reported absorbance maximum and minimum from a scan of 0.02% Toluene in Hexane. The absorbance maximum should occur at approximately 269 and the minimum at approximately 266 nm. The test passes if the calculated spectral resolution ratio is greater than or equal to the test limit. This test is applied to all UV/Vis spectrophotometers and meets European Pharmacopoeia requirements.

Procedure: Power on and warm up the spectrophotometer for the appropriate time prior to testing. If a cell changer is present disable the cell changer for testing. Using the spectrophotometer's CDS setup the Resolution Toluene/Hexane Test. Perform the test by following the software prompts. When the test is complete record the values to the test record.

4. Stray Light - Potassium Chloride at 198 nm

Rationale: The Stray Light Test measures the presence of light outside the bandwidth of a selected wavelength that reaches the detector. The presence of stray light can decrease the photometric selectivity, increase the photometric response and create non-linear response of the instrument causing problems with quantitative analysis. Potassium Chloride will completely absorb light below 200 nm, however is transparent above 200nm. The test will pass if the Percent Transmittance is less than 1.0% at 198 nm. This test is applied to all UV/Vis spectrophotometers and meets all major pharmacopoeia and ASTM requirements.

Procedure: Power on and warm up the spectrophotometer for the appropriate time prior to testing. If a cell changer is present disable the cell changer for testing. Using the spectrophotometer's CDS setup the Stray Light – KCl (Potassium Chloride) at 198 nm Test. Perform the test by following the software prompts. When the test is complete record the values to the test record.

OQ Test Design and Rationale for UV-Visible Spectrophotometers (continued)

5. Photometric Accuracy - Potassium Dichromate

Rationale: The Photometric Accuracy - Potassium Dichromate Test uses two strengths of the Potassium Dichromate standard Firstly the 60 mg/L Potassium Dichromate standard is used to measure the difference between the expected absorbance values (listed on the Certificate of Analysis) and the actual absorbance values reported by the instrument at four wavelengths: 235 nm, 257 nm, 313 nm, and 350 nm. The Test passes if the expected absorbance values are within +/- 0.01 AU of the actual absorbance values at the four wavelengths.
Then the 600 mg/L Potassium Dichromate standard is used to measure the difference between the expected absorbance value (listed on the Certificate of Analysis) and the actual absorbance value reported by the instrument at 430 nm. The Test passes if the expected absorbance value is within ± 0.012 AU of the actual absorbance value at 430nm. This test is applied to all UV/Vis spectrophotometers and meets all major pharmacopoeia, ASTM, and NIST requirements.

Procedure: Power on and warm up the spectrophotometer for the appropriate time prior to testing. If a cell changer is present disable the cell changer for testing. Using the spectrophotometer's CDS setup the Photometric Accuracy – Potassium Dichromate Test for both the 60 mg/L and 600 mg/L standard concentrations. Perform the test by following the software prompts. When the test is complete record the values to the test record.

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