Case Study

Design Qualification
Operational Qualification
HPLC Equipment

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Case study

Scenario: A Food laboratory plans to purchase 10 new HPLC systems for multiple applications.

A new food lab will be set up with 10 HPLC systems. All are controlled from computers. All instruments are expected to be used for standard and narrow bore columns for isocratic and for gradient runs. Standard detection is UV wavelength variable wavelength. About 30% of all applications will require spectral confirmation with a diode-array detector. Injection and data evaluation, including spectral evaluation, will be done automatically. Some samples are unstable at room temperature. Some of the analyses done on the HPLC's will be part of GLP studies and the lab plans to get accreditation for ISO 17025.

To simplify DQ, IQ and OQ, and to allow highest flexibility for the lab, all HPLC systems should have the same configuration.

A few remarks:

1. We recommend setting the limit for the baseline noise of the UV/Visible DAD to 6 x 10-5 AU. This is 3 times higher than the instrument specification. This provides enough tolerance in case the condition of the systems is not in ideally matched with new maintenance parts.

2. The OQ procedure should be scheduled once a year. Because the detector's baseline noise is critical for the success of the application, this test should be scheduled every month.

3. The instrument is always calibrated with a chemical standard before and during a series of sample analyses. Therefore the accuracy of the injection volume does not need to be tested.

Important Notice

The case study shows examples that can go into a DQ and OQ. It is not complete and has to be customized for individual situations

Design Qualification

Criterion	Qualification
Intended use	General food HPLC analysis
User requirement specification for the HPLC analysis	• 40 samples / day • Automated over-night analysis • Confirmation of peak identity and purity with diode-array detection • Automated compound quantitation and printing of report
Functional	
Pump	Gradient, min binary flow range: 0.2 to 10 ml/min
Detector	UV/Vis Diode-array, wl 200 to >400
Autosampler	100 samples variable volume injection from 0.2 to 100 ul without hardware change temperature control 4 to 40 deg C
Column compartment	25 to 40 deg C, peltier controlled
Computer	System control, data acquisition for signals and spectra, peak integration, external and internal standard calibration, at least 2 level calibration, spectral evaluation for peak purity and compound confirmation. Multi-method sequencing. bracketing electronically save all chromatograms, methods, log-books generated by the system.
Operational	Detector: Baseline noise: <5 x 10-5 AU Sampler: Precision inj. volume: <1% RSD Pump: precision of retent.time: <1% RSD Column oven: accuracy +- 2 deg C
User instructions	Operational manual on paper On-line help Computer based on-line tutorial
Validation/qualification	Vendor must provide IQ and OQ documents and services

Maintenance	Vendor must deliver maintenance procedure and recommend schedule
	Instrument must include early maintenance feedback for timely exchange of most important maintenance parts
	Maintenance procedures must be supplied on Multimedia CD ROM
Training	Vendor must provide familiarization and training
Vendor qualification	
Quality system	Vendor must have a quality system in place, e.g., ISO 9000
Software development	Vendor must develop SW according to documented procedure. Vendor must supply validation certificate or declaration of validation.
User feedback	Vendor must have a user feedback and response system and follow up procedures for problems reported by the user.
Support	Vendor must provide local on-site support through qualified people.

Operational Qualification

Scenario: Food Analysis lab with 10 new HPLC systems (for details see case study: DQ)

Parameter	Procedure (*)	User Limit
Flow/Leak testing	Flow test by volume or weight/time	± 5 %
Baseline drift	ASTM Method E19.09, 20 min	2 x 10-3 AU
Baseline noise	ASTM Method E19.09, 20 x 1 min	1 x 10-4 AU
Precision of injection volume	6 x injection of caffeine standard, RSD of peak areas	2 % RSD
Precision of flow rate	6 x injection of caffeine standard, RSD of retention times	1 % RSD
Detector linearity	inject 5 standards	1.5 AU, 5%
Wavelength accuracy	use built in holmium oxide filter at 361 nm	± 2 nm
Temperature accuracy	comparison with external measuring device	± 1 °C
Temperature precision	monitoring temperature over 20 min	± 0.5 °C
Autosampler carry over	injection of blank solvent after large amount	< 0.3 %
Mobile phase composition accuracy	step gradients from 4 to 7 % B, step heights relative to 100%, with acetone tracer	± 1 %
Mobile phase composition precision	precision of step gradients from multiple runs	0.3 %
Mobile phase composition ripple	peak to peak noise at 4, 5, 6 and 7% B	0.2 %
Computer system	run OQ tests automatically All specified tests should be completed and results printed according to the SOP	
	run ChemStation Verification Test with a test method, that includes peak purity check and spectral library search	