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Analytical and Bioanalytical Method Validation

Practical 10 - Protocol and data-analysis practical

Aim

To prepare analytical and bioanalytical validation protocols with the controls appropriate to each intended use.

Principle

Validation provides evidence that a procedure meets its intended analytical purpose over a defined reportable range. Relevant characteristics depend on whether the method measures identity, assay, impurities or another attribute.

A predefined protocol connects the analytical target and acceptance criteria to specificity, response behaviour, accuracy, precision and other required performance characteristics. Method development and risk assessment inform the validation design.

Materials and Requirements

- Written analytical procedure and target
- Suitable reference, placebo, impurity or challenge materials
- Predefined protocol and qualified measurement system
- Data-analysis worksheet

Procedure

1. Define the analyte, matrix, purpose, reportable range and performance requirements before generating validation results.
2. Identify the characteristics required for that use and explain the study design, levels, replicates and independent preparations.
3. Evaluate selectivity with appropriate blanks, placebo, relevant impurities or other justified challenges.
4. Assess response behaviour, accuracy and precision across the intended range. Include quantitation capability when required by the purpose.
5. Use development knowledge and deliberate small changes to understand relevant robustness factors; define operational controls.
6. Assess solution and preparation stability as needed and document handling conditions.
7. Review every result against the protocol and issue a report explaining deviations, limitations and the supported method use.

Observations and Record

Enter actual measurements or the supplied teaching data. Record the method, material identity, conditions and units.

Characteristic	Study design	Result	Predefined criterion and conclusion
Selectivity	Record challenges	Record	Evaluate
Accuracy	Levels and independent preparations	Record	Evaluate
Precision	Replicates / conditions	Record	Evaluate
Range and response	Model and residuals	Record	Evaluate

Characteristic	Study design	Result	Predefined criterion and conclusion
Other relevant characteristics	Justify	Record	Evaluate

Calculations

- Recovery (%) = measured added amount / known added amount x 100
- RSD (%) = standard deviation / mean x 100
- Bias (%) = (mean measured - nominal) / nominal x 100

Worked Example

Illustrative replicate assay results are 99.2, 100.0 and 100.8%. Mean = 100.0%, sample standard deviation = 0.8 percentage point and RSD = 0.80%.

For an independently added 10.0 mg amount recovered as 9.85 mg after valid native-content correction, recovery is 98.5%.

Interpretation uses the prespecified method-purpose criteria; there is no one universal recovery or RSD limit for every analytical use.

Bioanalytical additions

- For regulated concentration measurements, address the relevant ICH M10 characteristics, including selectivity, matrix effects, carryover, dilution integrity, stability and required run controls.
- A chromatographic teaching example may assess calibration and quality controls using the common M10 concentration-bias and precision criteria, with the distinct LLOQ allowance. Confirm applicability to the actual assay type.
- Keep extraction recovery, matrix response and study-preparation stability separate from routine pharmaceutical-product assay validation.

Result and Interpretation

- Conclude on the defined method purpose and range, with specific limitations and required controls.
- Do not substitute a high calibration R-squared value for validation.

Precautions

- Set criteria before viewing study results.
- Use independent preparations where the characteristic requires them.
- Keep failed and repeated results with justified investigations.
- Separate reference-material uncertainty from test-preparation variability.

Viva Questions and Answers

Why define the intended purpose first?

The necessary performance depends on what the method must measure.

Is linearity alone validation?

No; it does not establish selectivity, accuracy or precision.

Where is robustness knowledge useful?

It helps identify critical method parameters and practical operational controls.

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