Analytical Test Method Validation Report Template

1. Purpose

The purpose of this Validation Summary Report is to summarize the finding of the validation of test method "*Determination of ……",* following Validation Protocol "….".

2. Scope/Test Method Description

Optional: may be restated from the protocol.

3. Background

Optional: may be restated from the protocol.

4. Strategy

Optional: may be restated from the protocol.

5. Procedures

Include synopsis of procedure for execution of validation of each characteristic. Include acceptance criteria for each characteristic.

5.1. Accuracy

Accuracy should be reported as percent recovery of the known added amount or as the difference between the mean and the accepted true value together with the confidence intervals.

The concentration ranges used should be stated, as should the number of replicates tested.

In the table, the terms concentration 1, 2, and 3 should be replaced with the actual concentrations used in the experiments. Additional rows may be added if more concentrations were tested.

Table: % Recovery & Difference between Mean and Accepted Value

Analyte Level	Actual Concentration	Individual % Recovery	Mean % Recovery	P/F	% RSD	P/F
Level 1						
	Acceptance Criteria					
Level 2						
	Acceptance Criteria					
Level 3						
	Acceptance Criteria					

5.2. Precision

5.2.1. Method Precision

Describe the experiments to determine repeatability. Appropriate table shells for this section are provided below. This table assumes 3 replicate sample preparations at 3 concentrations and assumes

quantitation of the results. In cases where visual inspection is used, replicate chromatograms may be used in place of tables.

Analysis Repeatability Using Different Concentrations

Analyte Level	Amount of Analyte	Measured parameter	Mean	% RSD	P/F
Concentration Level 1	Replicate 1				
	Replicate 2				
	Replicate 3				
	Acceptance Criteria				
Concentration Level 2	Replicate 1				
	Replicate 2				
	Replicate 3				
	Acceptance Criteria				
Concentration Level 3	Replicate 1				
	Replicate 2				
	Replicate 3				
	Acceptance Criteria				

Repeatability using Multiple Determinations at the Test Concentration

Determination	Measured parameter
Replicate 1	
Replicate 2	
Replicate 3	
Replicate 4	
Replicate 5	
Replicate 6	
Mean	
% RSD	
Acceptance Criteria	
P/F	

5.2.2. Intermediate Precision

Depending on the nature of the analysis and results – visual inspection or quantitative data, either the appropriate figures or table should be included in this section. The table below shows a sample layout for reporting intermediate precision where quantitative data is obtained.

Determination	Analyst 1 /Lab 1/Day 1	Analyst 2 /Lab2/Day 2
Sample 1		
Sample 2		
Sample 3		
Sample 4		
Sample 5		
Sample 6		
Mean (N=6)		
% RSD (N=12)		
Acceptance Criteria (N=12)		
P/F		

5.3. Specificity

Provide representative figures of a placebo, representative sample, and a stressed sample.

Figure 1: Placebo Sample

Figure 2: Representative Sample

Figure 3: Stressed Sample

Discuss the results of the specificity experiments. If method specificity is not demonstrated, describe how additional test methods in combination with this test method are used to provide the appropriate specificity.

Stress Condition	Purity Angle	Purity Threshold	% Degradation
Heat			
Oxidation			
Humidity			
Light			
Acid			
Base			

5.4. Linearity and Range

Define the range of concentrations chosen in both concentration units and as % of the test concentration in the method. Briefly describe how the standard solutions were prepared.

State statistical analysis was used and specific tests that were performed. Discuss the scatter in the data points around the linear regression line, i.e. whether the scatter appears to be random or non-random, and the implications of the data scatter pattern.

Provide a graph of the linearity data showing the individual data points and the regression line. Display the regression equation on the graph or include as a sentence in the final report.

Figure: Linearity Graph (The axes on this graph should be labeled, including units)

State the range over which the assay was validated. In the table below, the terms concentration 1, 2, 3, 4, and 5 should be replaced with the actual concentrations used in the experiments. Additional rows may be added if more samples were tested.

Level			Area
Concentration Level relative nominal working standard in %	**Actual Concentration**		
Concentration 1			
Concentration 2			
Concentration 3			
Concentration 4			
Concentration 5			
Slope			
y-intercept			
Correlation Coefficient Acceptance Criteria r ≥ 0.XXX		**P/F**	

5.5. Limit of Detection

Provide a figure showing the detection limit for the analyte.

Figure: Representative Sample Showing <Name of Analyte> at the Limit of Detection

Provides a visual statement of this limit and may represent one of the calibration curves. The data used to calculate the actual level is captured in the table below.

Replicate	Anlyte Peak Area	Analyte S/N
1		
2		
3		
Acceptance Criteria		≥3 for each replicate
Pass/Fail		

5.6. Limit of Quanititation

State how the limit of quantitation is to be established – visual inspection, signal-to-noise ratio or standard deviation of the slope and response factor, and provide the relevant supporting data. If the visual detection method is used, provide the values associated with the terms “acceptable accuracy and precision.”

Figure: Representative Sample Showing <Name of Analyte> at the Limit of Quantitation

This figure should be clearly labeled to enable a reviewer to locate the peak(s) for the analyte(s) of interest.

Signal-to-Noise ratio applicable where the data is quantitatively assessed.

Replicate Determination	Analyte Peak Area	Analyte S/N
1		
2		
3		
4		
5		
6		
Mean		
% RSD		
Acceptance Criteria	% RSD ≤ XX.0%	≥10 for each replicate
Pass/Fail		

5.7. Robustness:

Standard Solution Stability

Time	Room Temperature			2 – 8 ºC		
	% of Analyte	% Difference	P/F	% of Analyte	% Difference	P/F
Day 0						
Day x						
Day Y						
Day Z						

Sample Solution Stability

Time	Room Temperature			2 – 8 ºC		
	% of Analyte	% Difference	P/F	% of Analyte	% Difference	P/F
Day 0						
Day x						
Day Y						
Day Z						

Test methods Parameter

Method Parameter	%RSD (area)	RRT
Injection Volume (Nominal)		
Injection Volume 1		
Injection Volume 2		
Mobil Phase (Nominal)		
Mobil Phase 1		
Mobil Phase 2		
Column Temp. (Nominal)		
Column Temp. 1		
Column Temp. 2		
Detector Wavelength (Nominal)		
Detector Wavelength 1		
Detector Wavelength 2		
Sample Extraction Time (Nominal)		
Sample Extraction Time 1		
Sample Extraction Time 2		
Sampling Time (Nominal)		
Sampling Time 1		
Sampling Time 2		
Flow Rate (Nominal)		
Flow Rate 1		
Flow Rate 2		
Column Lot 1		
Column Lot 2		
Column Lot 3		

6. Deviations from Protocol

Include in this section: "*No deviations from validation protocol……"* or list the deviations and reasons for those deviations. State the impact of the deviation or variance on the protocol. State the impact of the variation or deviation on the ability of the experiment to be suitable to validation.

7. Conclusion

Summarize the results of the Validation Study and conclude whether or not the Test Method is appropriate for its intended use base on the validation results given in this report and the acceptance criteria set forth in the Validation Protocol.

8. Recommendations

Indicate any changes that need to be made to the Test Method before it should be approved. These changes should be a result of the robustness testing outcome and may include modifying or supplementing the System Suitability section of the Test Method and/or adding caution statements about requirements for analyst control of experimental parameters.

9. Attachments

Calibrated equipment list, signature log of executors, copies of pertinent training records, data tables, chromatograms or printouts from equipment, figures as defined by results presentation and appropriate notebook references or pages.