LAB-135: Validation of Analytical Test Procedure

Table 1: For Non-Compendial Analytical and Biological Test Methods

Parameter	Identification	Impurity Quantitation	Impurity Limit Test	Assay	Dissolution	Biological Test Method	Physical Properties
Specificity	X	X	X	X	X	X	(a)
Linearity		X		X	X	X	(a)
Range		X		X	X	X	(a)
Accuracy		X		X	X	X	(a)
Precision Repeatability Intermediate Reproducibility		 X X (b) X (b)		 X X (b) X (b)	 X X (b) X (b)	 X X X	 X X (b) X (b)
Limit of Detection		X	X			(a)	(a)
Limit of Quantitation		X				X	(a)
Robustness	(a)	X	(a)	X	X	(a)	(a)

Table 2: For Compendial Analytical and Biological Test Methods

Parameter	Identification	Impurity Quantitation	Impurity Limit Test	Assay	Dissolution	Biological Test Method	Physical Properties
Specificity	X (a)	X (a)	X (a)	X (a)	X (a)	X (a)	
Linearity		X (a)		X (a)	X (a)		(c)
Accuracy		X (a)		X (a)	X (a)	X	(c)
Precision Repeatability Intermediate Reproducibility		 X (a) X (a&b)		 X (a) X (a) X (a&b)	 X (a) X (a&b)	 X (a) X (a) X (a)	 X (a) X (b)
Limit of Detection		X (a)	X (a)				(c)
Limit of Quantitation		X (a)					(c)

Table 3: Accuracy Limits for Given Release Limits of Active

Release Limit	% Recovery
99 – 101	99.5 – 100.5
98 – 102	99.0 – 101.0
97 – 103	98.5 – 101.5
96 – 104	98.0 – 102.0
95 – 105	97.5 – 102.5
90 – 110	95.0 – 105.0

Table 4: Accuracy Limits for Given Release Limits of Impurities

Degradant / Impurity Level (%)	% Difference
≤0.10	±0.03 absolute
>0.10 and ≤0.50	±0.05 absolute
>0.50 and ≤5.00	±10% relative
>5.00	±0.50 absolute

Table 5: Method Precision Limits for Actives

Limit Range (assay)	%RSD
99 – 101%	0.3
98 – 102%	0.7
97 – 103%	1.0
96 – 104%	1.3
95 – 105%	1.7
90 – 110%	3.3

Table 6: Method Precision Limits for Degradants and Impurities

Degradant / Impurity Level (%)	% Difference from the target concentration or mean
≤0.10%	±0.03% absolute
>0.10 and ≤0.50%	±0.05% absolute
>0.50 and ≤5.00%	±10% relative
>5.00%	±0.50% absolute