Cleaning Validation ï Rinsing Test

(Ref.VAL-010)

Project number:

Protocol Number:

Product/Active:

Process Line:

RINSING RECOVERY STUDIES

1. Test Description

This test is to be conducted to document the validation of Total Organic Carbon (TOC) analysis method for use in measuring samples for cleaning validation. A parallel analysis of rinse samples will be carried out and compared using TOC and High Performance Liquid Chromatography (HPLC) analysis. Standard solutions will be applied to stainless steel plates, dried and the residue removed by rinsing using an appropriate solvent (usually water). Assessment of Linearity, Accuracy (recovery), LOQ, LOD and precision of the rinsing method will be determined.

2. Test Objective

1. Determine the linearity and precision of a series of standards rinsed from a stainless steel plate or container and measured by TOC and HPLC over a known concentration range.

2. Determine the accuracy (recovery) of a series of standards rinsed from a stainless steel plate or container and measured by TOC and HPLC over a known concentration range.

3. Determine the Limit of quantitation and limit of detection of a series of standards measured by TOC and HPLC over a known concentration range.

4. Determine correlation between HPLC and TOC analysis.

5. Determine the final HPLC and TOC rinsing limits based on recovery studies.

3. Acceptance Criteria

Test Objective	Measured Response	Acceptance Criteria
Linearity	The correlation coefficient (r^2) for the linear concentration range.	HPLC: equal to or greater than 0.997 TOC: equal to or greater than 0.980
Precision	% RSD	HPLC: The recovery %RSD less than or equal to 10.0%. TOC: The recovery %RSD less than or equal to

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(ppm)	
(50%)	
(100%)	
(400%)	

Plot Actual TOC Concentration (ppm) V's Measured TOC Response

Coefficient of Determination (R^2) =

Accuracy - Rinsing active residue

For each measured response over the concentration range examined in the Linearity test calculate **% recovery** and **% RSD** for each of the triplicate samples.

where % recovery = $\frac{\text{result found}}{\text{result expected}} \times 100$

Actual Active Concentration	Measured responses				Average result	Mean % recovery	% RSD
	1	2	3	4			
(50%)							
(100%)							
(400%)							

1. HPLC Analysis

Compare and summarise results according to acceptance criteria

2. TOC Analysis

Actual Total Organic Carbon Concentration (ppm)	Measured TOC (ppm)	Average TOC (ppm)	Mean % recovery	% RSD

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Lowest TOC rinse recovery value = _____

The final limits for the active residue for rinse solutions are:

Equipment	**Equipment Surface Area (cm^2)**	**Acceptable Carbon Residue Based on product formulation (mg of Carbon)**	**Rinse Volume used to rinse equipment Surface**	**Final TOC Limits for Carbon incorporating recovery results**
Mixing Vessel	eg. 26,664 cm^2	eg. 11.1325	eg.2500 mL	eg. 11.1325mg/2500mL x 0.8966 x 1000ug/mg = **3.993 ug/mL or ppm**
Holding Vessel				
Sparge Tube				
Transfer Line				
Glass Flask				
Other equipment				
Total				

6. Comments

7. Conclusion

8. Attachments

Validation Discrepancy Forms - nil