

CLEANING VALIDATION TECHNIQUES

	Initial Cost	Set-Up Time	Detection Limits	Qualitative/ Quantitative	ID specific Ingredient?	Check for which residue	Analysis of what?	Speed Of Analysis	Drawbacks	Typical Customers	Customer Popularity
Visual	None	None	Varies	Qualitative	No	Soil	Part	Immediate	Subjective	Non-FDA	Yes
Titration	Minimal	Minimal	~ 0.1%	Quantitative	No	Cleaner	Cleaner Bath / Rinse water	~ 5 minutes	Solvents – use & disposal	Non-FDA	No
Refractive Index	Minimal	Minimal	~0.25%	Quantitative	No	Cleaner	Cleaner Bath / Rinse water	< 5 minute	Hazy/cloudy solutions are difficult to read.	Non-FDA	No
Foam Test*	Minimal	Minimal	~0.25%	Qualitative	No	Cleaner	Cleaner Bath / Rinse water	< 5 minutes	Hard water.	Non-FDA	Yes
Conductivity*	Low	Minimal	~0.05%	Quantitative	No	Cleaner	Cleaner Bath / Rinse water	Immediate	Hard Water.	FDA & Non-FDA	Yes
Methylene Blue*	Minimal	Minimal	~0.005%	Semi-Quantitative	Yes	Cleaner	Cleaner Bath / Rinse water	~ 15 minutes	Solvent – use & disposal.	Non-FDA	Yes
Methylene Blue w/ UV-vis Spec	Moderate	Moderate	ppm	Quantitative	Yes	Cleaner	Cleaner Bath / Rinse water	~ Hour	Solvent – use & disposal. \$ Upkeep.	FDA	Yes
Black Light*	Low to Moderate	Low	ppm (with UV detector)	Qualitative	No	Both	Part	Immediate	Skin & Eye exposure. May need UV dye.	Non-FDA	No
TOC	High	Moderate	Ppm	Quantitative	No	Both	Rinse Water Part Wash Swab	Several Minutes	Solvents interfere.	FDA	Yes
GC/LC/IC	High	High	ppm	Quantitative	Yes	Both	Rinse Water Part Wash Swab	Several to many minutes	Solvents – use & disposal. \$\$ Upkeep.	FDA	Yes

*Validation methods attached.



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International Products

C O R P O R A T I O N

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Cleaning Validation Methods

The following qualitative procedures describe how to establish a cleaning validation method for vessels, parts, and surfaces that are otherwise difficult to visually inspect.

I. Conductivity

For use with: Micro-90[®], LF2100[®], Micro[®] A07, Zymit[®], Zymit[®] Pro

This method exploits the sodium content of the following cleaners to determine concentrations using conductivity.

II. Methylene Blue

For use with: Micro-90[®], LF2100[®], Micro A07[®], Zymit[®] Pro

This method exploits the formation of a sulfonate-methylene blue complex to determine trace quantities of cleaner residue.

III. Optical Brightener

For use with: Micro-90[®], LF2100[®], Micro[®] A07, Surface-Cleanse/930[®], Zymit[®], Zymit[®] Pro

This technique exploits a water-soluble optical brightener that fluoresces as low as <1ppm in a solution.

IV. Foam Test

For use with: Micro-90[®], Surface Cleanse/930[®]

This technique utilizes the foaming properties of surfactants to determine the presence of cleaner residue. (Method not included here. Contact International Products Corporation for further details.)

If your cleaning process requires a quantitative test method, e.g. to meet FDA criteria, we can offer guidance with the following:

I. UV Visible Spectrophotometer

II. Total Organic Carbon (TOC)

Quantitative validation methods are available upon request. Contact International Products Corporation for information.

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Cleaning Validation Methods

I. Conductivity

For use with: Micro-90[®], LF2100[®], Micro[®]A07, Zymit[®], Zymit[®] Pro

This method exploits the sodium content of the following cleaners to determine concentrations using conductivity.

Equipment/Reagents

- Oakton Acorn Con 6 Conductivity Meter
- Oakton 1413 uS @25°C Conductivity Standard Solution
- Oakton 12.88 mS @25°C Conductivity Standard Solution

Procedure

1. Ensure the ATC factor is set to the default value of 2.1.
2. Calibrate the conductivity meter using both standard solutions.
3. Rinse conductivity probe in distilled water
4. Immerse conductivity probe in cleaning solution to be tested.
5. Record conductivity value in micro Siemens (uS). Use this figure for the calculations below.

Calculations

Divide the micro Siemens the RI reading by the factor that corresponds to the cleaner you are testing.

	Micro-90	LF2100	Micro A07	Zymit Low-foam	Zymit Pro
Concentration of cleaner (%v/v) = micro Siemens /	1217.3	1308.6	691.7	70.2	378.0

II. Methylene Blue

For use with: Micro-90 and LF2100

This method exploits the formation of a sulfonate-methylene blue complex to determine trace quantities of cleaner residue.

Equipment/Reagents

- Methylene Blue indicator solution
- 25 ml test tube or larger, with stopper
- Chloroform or 1-Bromopropane*

Procedure

1. Fill the test tube with 10cc of rinse water.
2. Add a drop of a 0.1% solution of methylene blue to the test tube.
3. Add 5cc of chloroform (or 1-Bromopropane) to the test tube.
4. Plug the tube and shake well.
5. If the chloroform layer turns blue, cleaner is present and additional rinsing is indicated.

Cleaning Validation Methods

III. Optical Brightener

For use with: Micro-90[®], LF2100[®], Micro[®] A07, Surface-Cleanse/930[®], Zymit[®], Zymit[®] Pro

This technique exploits a water-soluble optical brightener that fluoresces as low as <1ppm in a solution.

Equipment/Reagents

UV lamp, shortwave 254nm or longwave 366nm

D-282 UV-Blue dye, Water Soluble (manufactured by DayGlo Color Corporation, Cleveland, OH)

Purpose

This method determines the cleaner solution coverage and pattern, and the optimal rinse procedures to effectively remove cleaner residue.

Procedure

1. Prepare a 2% cleaner solution (or another concentration).
2. Dissolve 0.1% of the D-282 dye (or 5% of cleaner concentration) in the cleaning solution.
3. Clean tank interior with cleaning solution.
4. While still wet, inspect interior walls, corners, agitators, etc. using UV lamp. The UV lamp should be held at a distance of 12" from the wet surface to check for fluorescence.
5. If necessary, modify cleaning procedure until an acceptable cleaning coverage/pattern is observed.
6. Rinse tank interiors.
7. Repeat inspection of tank as described in step 4.
8. If necessary, modify rinse procedure until no more fluorescence is observed.
9. Document cleaning and rinse procedures as part of the tank validation method.

Limit of Sensitivity

In-house lab tests found that <1ppm of the D-282 dye was detected using the UV lamp.

Users should develop their own limits of sensitivity using their actual manufacturing tanks.