



Cleaning Method and Determination of Residue Method Validation for Glassware and Sampling Equipment (By Ion Selective Electrode Meter)

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ABSTRACT

In the pharmaceutical industry, cleanliness and product quality are critical aspects that cannot be compromised. However, the company initiated a study to develop a new cleaning method by decreasing the concentration of detergent from 20% to 5%. The proposed cleaning method was then evaluated to ensure its effectiveness in removing residues of the detergent and sodium fluoride, an active ingredient commonly found in manufacturing facilities. Sodium fluoride is one of the least water-soluble substances, making it more likely to remain on equipment surfaces. Through the development of the residue method, it was found that detergent residues can be analyzed using UV-Visible spectrophotometry. The maximum wavelength of the detergent residue was identified at 224 nm, with a limit of detection (LOD) of 2 ppm. Sodium fluoride can be analyzed using an ion-selective electrode. By swabbing fluoride from stainless steel surfaces, the recovery rate was calculated to be 95%. The cleaning method was tested under two conditions: during cleaning and after the equipment had dried. A total of 50 equipment samples were randomly tested. The results showed that the residue levels were within acceptable limits, demonstrating that a 5% detergent concentration effectively removes residues. This experiment also achieved a 75% reduction in chemical costs.

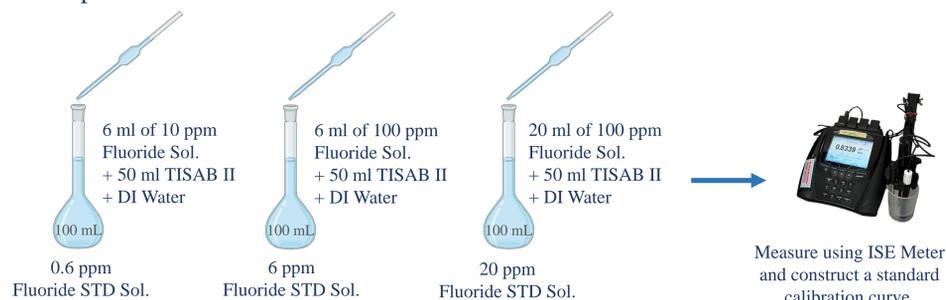
INTRODUCTION

In practice, the complete elimination of residues is challenging. Therefore, a standard was established with a maximum permissible residue limit of 10 ppm, ensuring product quality and mitigating cross-contamination. To reduce costs, the facility implemented a new cleaning method with a reduced detergent concentration, which was validated to effectively eliminate residues, particularly fluoride, the least water-soluble active ingredient.

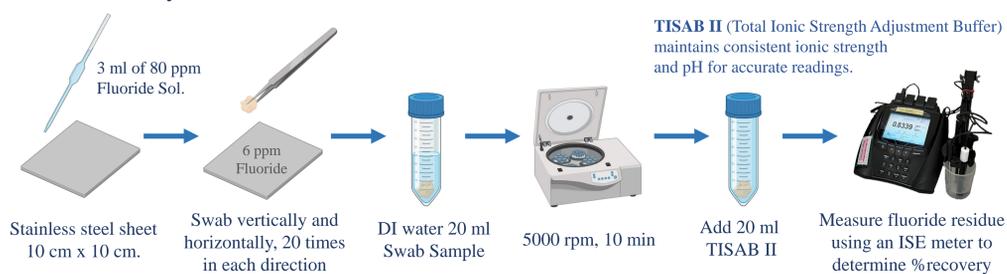
EXPERIMENTAL

Residue Method : Fluoride

- Preparation of Fluoride STD Solution for Standard Calibration Curve



- Recovery from Swab Method



Cleaning Method

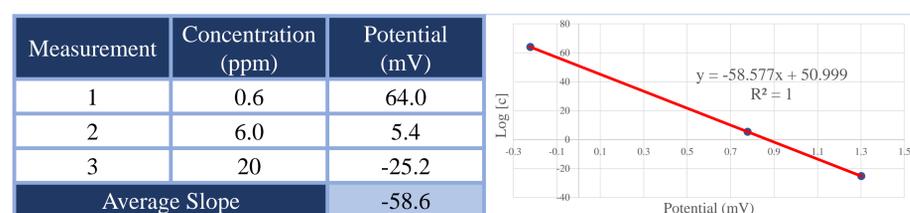
- Preparation of Rinse Sample



RESULTS AND DISCUSSION

Residue Method : Fluoride

- Standard Calibration Curve for Determine Fluoride ion



- Swab Recovery

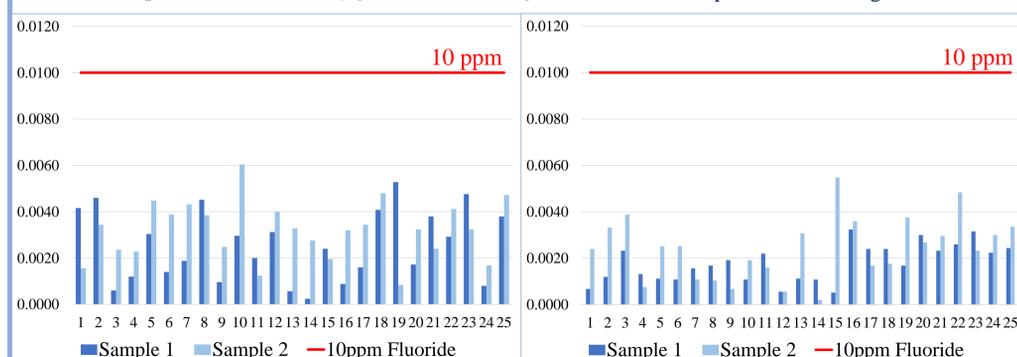
Measurement	Sample (ppm)	Sample - Blank (ppm)	% Recovery
Blank	0.0319	-	-
1	5.657	5.625	94
2	5.715	5.683	95
3	5.669	5.637	94
4	5.786	5.754	96
Mean	5.787	5.675	95

The swabbing method for residue is effective and can be efficiently used to detect fluoride residues, which can then be analyzed using the ISE meter.

Cleaning Method

- Rinse water by Ion Selective Electrode (ISE) meter

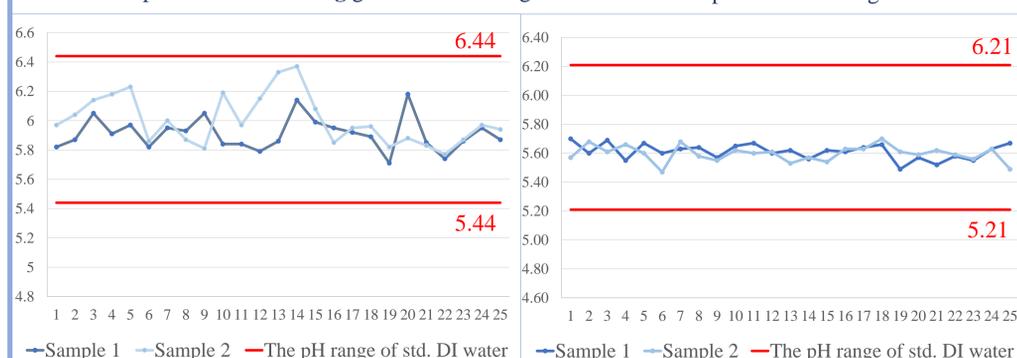
- Rinse samples collected during glassware cleaning.
- Rinse samples from dried glassware



The fluoride residue is below 10 ppm, preventing cross-contamination.

- Rinse water by pH meter

- Rinse samples collected during glassware cleaning.
- Rinse samples from dried glassware



The rinse water has a pH equivalent to that of water, effectively removing all residues.

CONCLUSION

Fluoride residues can be effectively measured using an ISE meter, ensuring accurate detection and analysis. In this study, a cleaning method utilizing a 5% detergent solution was found to efficiently eliminate fluoride residues from the sample equipment, maintaining cleanliness and preventing contamination. The successful implementation of this method offers a significant advantage to the company, as it can reduce the cost of detergent imports by up to 75%, leading to substantial cost savings while maintaining high standards of equipment hygiene and operational efficiency.

ACKNOWLEDGMENT

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