
 UNIVERSITY OF KWAZULU-NATAL	<p style="text-align: center;"><i>Standard Operating Procedure</i></p>  PRG pollution research group	Effective Date: 20 June 2013	Version: 002
		Reviewed: 1 Nov 2017	
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Standard Operation Procedure – Chloride Analysis

1. Scope and Application

- The M926 Chloride analyzer is used for the determination of chloride ions. It is an instrumental analogue of 'Argentimetry', the titrimetric methods using Silver Nitrate reagent. Like these classical methods it relies on the chemical formation of the very insoluble salt, silver chloride. The importance of chloride determination has been realized for well over a century, with many variations and changes being made to the techniques in order to improve the detectability and selectivity.
- The M926 Chloride analyzer is a direct reading, digital chloride meter. It is designed for fast and accurate determinations of chloride levels in industrial samples.
- Sample volume is 0.5ml and results are displayed on a digital readout in mg/L(milligrams per liter chloride) or mg%(milligram percent)salt as sodium chloride.

2. Summary

- An accurately measured volume of sample (0.5ml sample is added to an acid buffer. The analyzer automatically titrates chloride ions by passing a known constant current between two silver electrodes, which provides a constant generation of silver ions.
- These silver ions combine with the chloride in the sample to form silver chloride, which is held in suspension by the colloid stabilizer.
- During the titration period the digital readout is updated every 0.3seconds. During these periods, the number of silver ions introduced into the sample combine with one unit measurement of chloride.
- Free silver ions begin to appear and the solution conductivity changes, when all the chloride has been precipitated as silver chloride,
- This change is detected by the detector electrodes and the readout is stopped, displaying the results directly readout in mg/L (milligrams per liter chloride) or mg%(milligram percent)salt as sodium chloride.
- Another sample may now be added to the same buffer and the cycle repeated.
- The digital display is held until starting another cycle, when it is automatically reset to zero.
- **Sample range: 10-999mg/l chloride or 2-165mg%salt.**

3. Apparatus and Glassware

- Sherwood Chloride Analyser Model 926
- 50 ml glass beakers
- Testing vials

4. Interferences

- Never leave bottles of Standard solution uncapped, as prolonged exposure to the atmosphere will affect the solution's concentration.

5. Collection, Preservation and Storage

- Collect faecal samples in 1L plastic buckets.
- Preferably, analyse samples immediately after sampling.
- Store samples at 4 °C or freeze dry samples.
- Preserve wastewater samples by acidifying with concentrated sulphuric acid to pH 2 and faecal samples by freeze drying or freezing.
- Reclose the reagent bottles immediately after use.

6. Safety Precautions

- Handle concentrated acid with care.
- Always use safety goggles, gloves and laboratory coat while working in laboratory.
- Wear face shield and protect hands from heat produced when contents of the vessels are mixed. After the analysis, clean bottles and beakers with clear water keep it for drying.
- Dispose the used gloves after completion of analysis.
- Clean the hands using antiseptic soap.
- Disinfect hands after washing with soap.
- Avoid spillage and contact with skin. In the latter case use copious washings with cold water and call for medical attention.

7. Sample Preparation –Faecal Sludge

1. Weigh out 2.0000g of well-mixed faecal sludge sample.
2. Blend the weighed sample with 500ml of distilled water in a 1L blender for 30 seconds on the highest speed.
3. Add 250ml distilled water and blend on highest speed until the sample is homogenised (this could range from 30 to 60 seconds).
4. Transfer the blended mixture into a 1L volumetric flask.

5. Add 200ml of blender washings into the flask and top up to 1L with distilled water.
6. Transfer the 1L solution to a plastic bottle and store at 4 °C.

8. Reagents

- Acid Buffer

9. Calibration

- Prepare a series of at least three standards, covering the desired range, and a blank by diluting suitable volumes of standard solutions. Prepare a calibration curve by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by appropriate dilution factor. Report only those values that fall between the lowest and the highest calibration standards. Dilute samples exceeding the highest standard should be diluted and re-analysed. Report results in mg/L.

10. Procedure

1. Connect power, switch on and allow 5 minutes for the machine to warm up.
2. Fill the plastic beaker, supplied, to the mark with the combined acid buffer, place the beaker on the platform and raise the platform until it locates in the up position.
3. Pipette 0.5ml of 200mg/l standard solution into the beaker.
4. Press the 'condition' button and wait for the condition cycle to complete.
5. Pipette 0.5ml of sample into beaker and press titrate button.
6. When the stirrer stops, note the reading on the display. Select button to mg % salt for required reading unit.
7. Repeat steps 4 and 5 for further samples.
8. The message 'condition in 2' will appear on the screen at the end of the 5th titration. Return to number 4 if only two further samples are to be run. Continue with number 9 if more than two samples are to run. 'Condition 1' will be shown after the 6th titration.
9. If " change buffer and condition / condition required" is displayed, continue with number 9.
10. Lower beaker and empty out contents. Rinse with deionized water and dry with clean tissue.
11. If necessary, adjust the vertical position of the anode, Item 2, so that it is the same length as the other electrodes.
12. If more samples are to be titrated return to number 2.
13. When determinations are complete, remove the beaker and dry the electrodes and stirrer by blotting with a clean tissue.

11. Operating Precautions

- The electrodes may go black during use; clean electrodes only if there are measurement errors.
- The analyzer requires a warm up period of 5 minutes to meet the stated specification.

- Reproducibly accurate results are dependent on the reproducible pipetting from sample and from aqueous standard to sample. Rinse out the pipette if the calibration is checked with an aqueous standard and reproducibly low results are obtained.
- Samples should have low ionic strength, neutral pH and free of sulphide, sulfhydryl silver halides, silver reactive substances (other than chloride), solid matter and high levels of dissolved solids.
- DO NOT LEAVE ELECTRODES IMMERSSED IN REAGENTS WHEN THE INSTRUMENT IS NOT IN USE.

12. Waste Disposal

- Flush down the sink with excess water.

13. Data Quality

14. References

- https://pdfs.wolfllabs.co.uk/.../Sherwood_Scientific_Chloride_Meter_926_Manual.pdf

APPROVAL OF STANDARD OPERATING PROCEDURE

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