

CLEANING LABORATORY EVALUATION SUMMARY

SCL #: 2014
DateRun: 06/24/2014
Experimenters: Jason Marshall
ClientType: Cleaner Manufacturer
ProjectNumber: Project #1
Substrates: Liquid
PartType: Coupon
Contaminants: None
Cleaning Methods:
Analytical Methods: Surfactant Titration
Purpose: Create SOP for quality control of disinfectant product

Experimental Procedure: Products:
MOLDEX Disinfectant Concentrate
• EPA Reg Number 1839-169-82480
• EPA Est. No. 67874-MA-1

MOLDEX Disinfectant (RTU)
• EPA Reg Number 1839-83-82480
• EPA Est. No. 67874-MA-1

Step 1. - Electrode Preparation

When you receive the Nitrate ISE you screw in one of the Nitrate tips into the body of the electrode...it has an O ring attached. After you assemble the Nitrate ISE electrode, soak the tip in the 0.02 N sodium lauryl sulfate solution to condition it. The tip starts out dry and the plastic membrane at the bottom needs to be conditioned at least one hour. Check reference electrode (yellow) fluid levels.

NOTE: don't use a KNO₃ solution as the outer filling solution that comes with the 900200 electrode - contains Nitrate that will interfere with readings

Inner solution - green

Unscrew top back piece from yellow body

Slide the cap and spring up the wire

Push the inner section down so the tip pops out of the bottom

Slide the inner section down until the small hole is exposed

There will be a off-white sleeve covering the filling hole, slide it down to open the hole

Using the green solution remove cap and inner red plug

Attach white cover with dispensing nozzle

Squeeze bottle until the inner tube is filled to just below the hole

Recover the hole

Slide inner tube back into electrode housing

Slide spring and cap back down and tighten

Remove the blue plug from the outer section of the electrode

Using the 3M (or 4M) KCl (clear solution), remove cover and red plug

Attach second white cover with dispensing nozzle

Squeeze bottle until the tube is filled to just below the hole

Leave the blue plug out of the probe during testing

Place both probes into holders on stand

Lower holder so that both probes are submersed into a small beaker containing the 0.02 N SDS solution (made up as outlined below)

Allow to soak for at least an hour

Step 2. - Preparation of sodium dodecyl sulfate (SDS) Titrant (which is also referred to as sodium lauryl sulfate) (Use VMR benzethonium chloride and VMR specially pure SDS)

Benzethonium Chloride

Dry >2.5 grams of benzethonium chloride (hyamine 1622) at 105 C for 30 minutes

Let cool in dry location

Weigh out 2.3-2.4 g of benzethonium chloride (+/-0.1 mg). Record weight

Add to a 250 ml volumetric flask

Dilute with DI water

Take a 10 ml sample of this stock solution and add 80 ml DI water

Check pH of this 100ml of solution that will be titrated below

If below 10.5, adjust using a 0.02N NaOH solution. (if over 10.5 add 1-3 drops 0.02N HCL)

Sodium dodecyl sulfate (SDS) Stock Solution

Weigh 5.7 - 5.8 grams of sodium dodecyl sulfate (+/-0.1 mg)

Add to 1000 ml volumetric flask

Dilute to volume with DI water

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Check approximate N (Normality) by dividing weight by molecular weight
 $\sim 5.7\text{g}/288.37\text{ g/mole} = \sim 0.01976\text{ moles per liter}$

Titration to determine exact N (Normality) of SDS Titration Solution

Titrate with the same procedure as the product batch testing

Place beaker on stirring platform (after adding the 100ml of benzethonium chloride solution from above)

Insert electrodes

Titrate with the $\sim 0.02\text{ N}$ SDS solution

Calculation of the actual N (Normality) of the SDS solution

$\text{N SDS} = (\text{Hyamine} \times 10^4) / (448 \times (\text{SDS volume titrated}))$

SDS Normality =

$(\text{Hyamine, g}) \times (10) \times (4)$

$448 \times (\text{SDS, mL})$

Input of exact SDS N into the Excel Data entry for Quaternary QC Testing

1. Put SDS N (Normality) into the appropriate Tab 1 Cells of both the RTU Quat QC Data Entry, and, CONCENTRATE Quat QC Data Entry

2. The updated Tab 1 Master is then subsequently copied into each additional Tab used for each Quat batch to be tested with this specific SDS titration solution

3. A new batch of SDS titration solution should be made every 1-2 weeks

Step 3. - Burette Preparation

Fill burette with 0.02 N SDS solution (previously made and checked for concentration levels)

Place a waste collection beaker under the burette and open the stopcock

Allow fluid to flow out of the tip for 1-2 seconds to ensure air pockets are removed

If no flow happens, gently tap the burette just below the stopcock to help induce flow

Once flow is obtained close stopcock

Record starting volume on the graduated burette

Step 4A - Sample Preparation for CONCENTRATE

Using an electronic balance, tare 250 volumetric flask

Add accurately weighed sample of the batch (+/- 0.1 mg)

$\sim 20\text{-}30$ grams for the CONCENTRATE product

Dilute sample to 250 ml using deionized, distilled or bottled water (order of water source preference)

(Bottled water has been used for the QC titrations)

Cover and invert bottle a couple of times to help mix the solution

Take a 10 ml sample of dilution and place in a 250 ml beaker

Add 80 ml of water (or buffer solution if available)

Add stir bar

Check pH

pH should be 10.5

If lower than 10.5 add 1-3 drops 0.02N NaOH, mix and measure again

If higher than 10.5 add 1-3 drops 0.02N HCL, mix and measure again

Place beaker on stir plate, add stir bar and turn on stirring action

Want a steady non-vortex rate of mixing, adjust rate if necessary

Lower electrodes into solution

Tips should be just under the solution

The black electrode should not be submersed beyond grey tip

Turn on meter

Make sure reading mV (not pH)

To change to mV press mode button

Allow mV reading to stabilize

When ready, record mV reading

Titrate by opening the stopcock slowly, dispensing about 3-4 drops at a time (0.1 mL)

Record mL added to beaker

Record the mV level after addition

Titrate until the mV has a significant drop (70-80 mV drop)

Conduct 7-8 more additions beyond this drop

Enter results into Excel sheet

SDS Normality for Concentrate =

$(\text{Hyamine, g}) \times (10) \times (4)$

$384 \times (\text{SDS, mL})$

* The MW for the active Quat in the Concentrate is 343

Step 4B - Sample Preparation for RTU

Using an electronic balance, tare 250 volumetric flask

Add accurately weighed sample of the batch (+/- 0.1 mg)

~ 35 grams for the RTU product

Add 80 ml of water (or buffer solution if available)

Add stir bar

Check pH

pH should be 10.5

If lower than 10.5 add 1-3 drops 0.02N NaOH, mix and measure again

If higher than 10.5 add 1-3 drops 0.02N HCL, mix and measure again

Place beaker on stir plate, add stir bar and turn on stirring action

Want a steady non-vortex rate of mixing, adjust rate if necessary

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Lower electrodes into solution
Tips should be just under the solution
The black electrode should not be submersed beyond grey tip
Turn on meter
Make sure reading mV (not pH)
To change to mV press mode button
Allow mV reading to stabilize
When ready, record mV reading
Titrate by opening the stopcock slowly, dispensing about 3-4 drops at a time (0.1 mL)
Record mL added to beaker
Record the mV level after addition
Titrate until the mV has a significant drop (70-80 mV drop)
Conduct 7-8 more additions beyond this drop
Enter results into Excel sheet

SDS Normality for RTU undiluted sample (does not need to be multiplied by 4 like the Benzethonium Chloride or the SDS Normality for the Concentrate) =
(Hyamine, g)*(10)
 $384 * (\text{SDS, mL})$

* The MW for the active Quat in the RTU is 384

Step 5 - At the end of testing
Remove electrodes from solution in beaker and rinse with water
Blot dry and close the blue plug on the yellow reference electrode
Turn off meter
Turn and unplug stir plate
Store the probes in 0.02 N SDS solution for up to a week
If not using probe within a week, drain fluids from both chambers and store empty probes in supplied box
Empty the burette and discard the remaining SDS burette solution
Close the stopcock and fill 1/3 full with water
Empty stopcock and allow water to flow out of the burette into a waste beaker

Results:

Summary:

Conclusion: