Hanna Titration Procedure

NCO (Isocyanate) Method via ASTM D2572

Description

Method for the determination of isocyante (NCO) in plating samples to a mV endpoint. The results are expressed in **% NCO**.

Reference

Reference Method ASTM D2572.

Meter

- Automatic Potentiometric Titrator - HI932

Electrode

- pH Electrode - HI1049B
- Temperature Probe - HI7662-T

Reagents

- 1M Lithium Chloride (LiCl) in Ethanol (EtOH) Electrolyte
- pH 4.01 Calibration Solution (1L) - HI7004/1L
- pH 10.01 Calibration Solution (1L) - HI7010/1L
- 0.1M HCl in H_2O or in IPA
- THF Solvent (Trifluoromethylbenzene)
- 0.1N DBA in THF Reagent
- Deionized Water- HI70436
- Acidified Deionized Water

Accessories

- Scientific Scale/Balance
- Paper
- 150mL Beakers
- 100mL Beakers
- Transfer Pipettes
- PDVF Propellers
- Stopwatch or Timer
- Class A Volumetric Pipette or Graduated Cylinder

Device Preparation

- Connect the electrode and temperature probe to Analog Board 1 of the titrator.
- Press "Select Method" from the main screen. Use the arrow keys to highlight your desired NCO method and press "Select".
- Install a 25-mL burette with either 0.1M HCl on pump one and verify that no air bubbles are present in the burette or tubing. If the burette was pre-filled (and sitting for more than 12 hours), prime the burette until all the air has been removed completely.

Electrode Preparation

- Unscrew the fill cap and remove the storage cap.
- Turn the electrode upside down, and using the transfer pipette, remove the aqueous electrolyte.
- Transfer deionized water into the electrode to about halfway to the fill hole cover. Invert, and then remove the deionized water. Repeat this two more times.
- Add the non-aqueous electrolyte (1M LiCl in IPA) halfway to the fill hole cover, invert, and then remove the electrolyte from the electrode. Repeat two more times.
- Refill the electrode with the non-aqueous electrolyte up to the fill hole, and replace the fill hole cover. (Do not tighten the fill cap completely.)
- Attach the electrode to Analog Board 1.

Electrode Calibration

- **NOTE: These steps should be performed before the first titration of the day.**
- Check the electrolyte level of the electrode and add the LiCl in isopropyl alcohol to the fill hole.
- Replace the fill cap and tighten it loosely, or keep it removed.
- Rehydrate the electrode by soaking the bulb and junction in a beaker with acidified deionized water for at least 5 minutes.
- Press MODE and then select "pH".
- **NOTE: For calibration, you will be using the mV values in the bottom right-hand corner of the screen.**
- Fill a beaker with pH 4 buffer until the electrode can be properly submerged (~100 mL).

Hanna Titration Procedure

NCO (Isocyanate) Method via ASTM D2572

- Place the beaker below the stirrer assembly, ensuring that the electrode is properly submerged.
- Press the STIR button on the titrator to activate the stirrer.
- Record the mV readings at 30 seconds and 60 seconds.
- Determine the difference between the mV readings.
- The ideal difference between the two mV readings should be < 1 mV. For the difference to be acceptable it must be <2 mV difference.
- Press the STIR button again to deactivate the stirrer.
- Raise the stirrer assembly up and rinse the stirrer and probe with deionized water.
- Place the electrode into the acidified deionized water and allow it to soak for at least 1 minute.
- Fill a beaker with pH 10 buffer until the electrode can be properly submerged (~100 mL).
- Place the beaker below the stirrer assembly, ensuring that the electrode is properly submerged.
- Press the STIR button on the titrator to activate the stirrer.
- Record the mV readings at 30 seconds and 60 seconds.
- Determine the difference between the mV readings.
- The ideal difference between the two mV readings should be < 1 mV. For the difference to be acceptable it must be <2 mV difference.
- Press the STIR button again to deactivate the stirrer.
- Raise the stirrer assembly up and rinse the stirrer and probe with deionized water.
- Next, calculate the mV difference between pH buffer 4 and 10 readings which were taken at 60 seconds (i.e. pH 4 @ 60 seconds 126.3 / pH 10 @ 60 seconds -215.9)
- If the mV difference is >324 mV then the pH calibration is acceptable (i.e. 126.5 mV – (-215.9 mV) = 342.4 mV).
- If the test is unacceptable the first time, repeat the calibrations and calculations using fresh buffer solutions.
- If the test remains unacceptable after the second calibration attempt, replace the electrolyte in the electrode, rehydrate the electrode in acidified deionized water, and repeat the calibration..

Blank Preparation and Analysis

- Press the "Select Method" key.
- Scroll until you find your desired blank method for NCO.
- Select the method.
- Transfer 35 mL of THF solvent into a 100 mL beaker.
- Place the beaker under the stirrer assembly, and lower the assembly.
- Press STIR to activate the stirrer.
- Add 25 mL of 0.1N DBA in THF.
- Check that the sensing portion of the electrode is submerged.
- Press "Start". The titrator will start the analysis.
- At the end of titration, when the equivalence point is reached, 'titration complete' will appear with the concentration. The concentration will be displayed in Liters of titrant.
- Record the result, as well as the mL of titrant dispensed.
- Perform one additional blank titration, and then average the results.
- Select "Method Options".
- Scroll down and select "Blank Option".
- Select the option "V-Blank".
- Enter the blank volume in liters (you will need to convert the mL of titrant dispensed to L (liters), divide the mL dispensed by 1000).
- Select "Accept".
- Make sure to save the method before returning to the main titration screen.

Sample Preparation

- Press the "Select Method" key.
- Scroll until you find the % NCO method.
- Select the method.
- Mass approximately 1 g of sample* directly into the beaker.

***NOTE:** To calculate the appropriate sample size, use this calculation:

$$\frac{[(\text{Mole}) * (\text{Molecular Weigh})]}{(\text{Expected \%})} = \text{Sample Size}$$

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- Measure precisely 35 mL of THF and transfer it to your beaker.
- Place the beaker below the stirrer assembly and lower the stirrer assembly.
- Press the STIR button to activate the stirrer.
- Transfer 25 mL of the 0.1N DBA in THF and continue stirring for 15 minutes.

Analysis

- At the 15 minute mark, titrate the mixture immediately.
- Place the beaker under the stirrer assembly and lower it to immerse the electrode, temperature probe, and stirrer*. Ensure that the sensing portion of the electrode is 5-6 mm below the surface. **NOTE:** The dispensing tip should be in contact with the surface of the sample (slightly submerged).
- Press "Start". The titrator will start the analysis.
- At the end of titration, when the equivalence point is reached, 'titration complete' will appear with the NCO concentration. The result is expressed in **% NCO**.

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