Hanna Titration Procedure

ASTM D974 Low Range Acid Number, Photometric Method

Description

Method for the determination of acid number low range, in petro-chemicals, following the photometric titration method of D974 to a color change endpoint with the HI932 Automatic Titrator. The result is expressed in **(mg/g) of KOH**.

Reference

ASTM D974 – 04 Standard Test Method for Acid and Base Number by Color-Indicator Titration.

Meter

- Automatic Potentiometric Titrator - HI932

Electrodes

- 625 nm Photometric Electrode - HI900602

Reagents

- Potassium Hydroxide (KOH) in IPA, 0.1M or 0.01M (see Figure1 Note)
- Solvent (Toluene-DI-IPA at 100:1:99)
- 1-Naphtholbenzein Indicator Solution, 10g/L in Solvent
- IPA Rinse

Accessories

- Analytical Balance
- Volumetric Glassware (Class A)
- 150 mL Glass Beakers
- Automatic Pipette and Pipette Tips

Device Preparation

- Connect the photometric electrode to the titrator.
- Press "Select Method" from the main screen. Use the arrow keys to highlight the 'D974 Acid Number' method and press "Select".
- Install a 5-mL burette with potassium hydroxide (KOH) titrant on pump one and verify that no air bubbles are present in the burette or tubing. If necessary, prime the burette until all the air has been removed completely.

Electrode Preparation

- Put the titrator into mV mode by pressing "Mode".
- Then, select analog board 1, and press "mV1".
- Fill a 120 mL beaker with 100mL deionized water.
- Submerge the electrode into the deionized water.
- Press "STIR" to gently stir the solution, ensuring the stir speed does not exceed 800 RPM.
- Remove the green protective cap from the electrode (it looks like a traditional electrode fill cap, and is located beneath the word "CAL").
- Use the provided calibration screwdriver to turn the calibration screw until the mV reading on the titrator reads 1000 ± 5 mV.
- Press "Mode" and then "Titrator" to return to titration mode.
- **NOTE**: This calibration should be performed upon initial installation, and once per week thereafter.

Blank Preparation

- Press "Select Method" from the main screen. Use the arrow keys to highlight the 'D974 Acid Number-Blank' method and press "Select".
- Use a Class A glass graduated cylinder to transfer exactly 100.00 mL of TAN SOL to a clean beaker.
- Using an automatic pipette, transfer 0.5 mL of 1-Naphtholbenzein indicator solution to the beaker.

Blank Analysis

- Place the beaker under the stirrer assembly and lower it to immerse the photometric electrode and stirrer. Ensure that the optical cell 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 result. The result is expressed in **L**. (This is L of titrant consumed by the blank).
- Remove the photometric electrode and stirrer from the sample and rinse them thoroughly with IPA.
- Record the result.

Hanna Titration Procedure

ASTM D974 Low Range Acid Number, Photometric Method

- Repeat the blank preparation and analysis 2 more times.
- Take an average of the 3 results that are expressed in L.

Sample Preparation:

- Press "Select Method" from the main screen. Use the arrow keys to highlight the 'D974 Acid Number' method and press "Select".
- Go to "Method Options". Use the arrow key to scroll to Blank Value and "Select".
- Enter the average L taken from the blank analyses.
- Select "Accept".
- Then make sure to save the method before exiting the method and returning to the main screen.
- Using the analytical balance, mass out the sample in a 150 mL glass beaker. ***NOTE: Use Table 1 for suggested sample sizes per ASTM D974.***
- Use a Class A glass graduated cylinder to transfer exactly 100.00 mL of TAN SOL to the beaker.
- Using an automatic pipette, transfer 0.5 mL of 1-Naphtholbenzein indicator solution to the beaker.

Analysis

- Place the beaker under the stirrer assembly and lower it to immerse the photometric electrode and stirrer. Ensure that the optical cell 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 Acid Number. The result is expressed as KOH (mg/g).
- Remove the photometric electrode and stirrer from the sample and rinse them thoroughly.
- Record the result.

Figure 1. Sample size table per ASTM D974.

NOTE: These sample sizes are based on a titrant strength of 0.1M KOH in IPA; it is recommended to use 0.01M KOH in IPA when acid numbers are less than or equal to 1.0 mg/g and to divide sample sizes above by 10.

Acid Number or Base Number	Size of Sample, g	Sensitivity of Weighing, g
New or Light Oils		
0.0 to 3.0	20.0 ± 2.0	0.05
Over 3.0 to 25.0	2.0 ± 0.2	0.01
Over 25.0 to 250.0	0.2 ± 0.02	0.001
Used or Dark-Colored Oils		
0.0 to 25.0	2.0 ± 0.2	0.01
Over 25 to 250.0	0.2 ± 0.02	0.001

[A] Light-colored samples of low acid number permit the use of 20-g samples to obtain more precise results. The sample size for dark-colored oils is limited to the quantity specified to minimize possible interference by the dark color.

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