National Forage Testing Association Non-Reference Method

NFTA Method 3.1.1 - Standardization of Hydrochloric Acid Standard Solution

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Introduction

This procedure can be used to standardize hydrochloric and several other acids. This method is used for the reference method for ADF.

Scope

This method is applicable for the preparation and standardization of hydrochloric acid solution.

Basic Principle

An acidic solution is titrated with a standardized base solution to determine normality.

Equipment

Buret, 50 mL, graduated to 0.1 mL
Illuminated magnetic stirrer
Volumetric pipet, 40 mL, class A

Reagents

Hydrochloric acid, concentrated, 36.5 to 38%, reagent grade
Sodium hydroxide, standard solution (see method 3.1.2)
Distilled water, carbon dioxide (CO_2) free prepared either 1) by boiling for 20 min and cooling with soda-lime protection or 2) by bubbling air, freed from CO_2 by passing through tower of soda lime, through water for 12 hr.
Indicator, same as used in Kjeldahl method (3.1)

Safety Precautions

- Handle acid safely: Use acid-resistant fume hood; always add acid to water unless otherwise directed in method; wear face shield and heavy rubber gloves to protect against splashes; if acids are spilled on skin, immediately wash with large amounts of water.

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Procedure

Preparation

1. Dilute appropriate volume of 36.5 to 38% HCl to 10 L with carbonate-free distilled water as indicated below and mix well:

Desired normality	mL HCl to dilute to 10 L
0.01	8.6
0.02	17.2
0.10	86.0
0.20	172.0
0.50	430.1
1.00	860.1

Standardization

1. Fill a 40 mL volumetric pipet with the acid to be standardized and discard to rinse the pipet.
2. Withdraw a 40 mL aliquot of the HCl into the volumetric pipet. Wipe the tip of the pipet before transferring the acid into a 250 or 300 mL Erlenmeyer that has been rinsed with CO_2-free H_2O. Do this in triplicate.
3. Rinse a Schellbach buret with standardized NaOH of approximately same concentration as acid to be standardized. Fill the burette and wait for at least a minute (to allow the solution to drain down the inside walls) before taking an initial reading. Always read the burette at eye level and at the tip of the meniscus.
4. Add indicator used in Kjeldahl method to the acid solution and titrate, a drop at a time, with NaOH while stirring.
5. Titrate to orange endpoint (color change from red to orange to yellow). Use of an illuminated background is helpful. When the endpoint is reached, remove any drops from the burette tip (by touching the flask to the tip) and wash down the sides of the Erlenmeyer with CO_2-free H_2O to see if the color persists. If it doesn't, add another partial drop of NaOH and check the color before reading the burette.
6. Calculate normality and adjust as necessary with HCl or water. Mix thoroughly and recheck standardization as described above.
7. Record standardization in log book.

Comments

- The titration values of replicates should be within 0.05 mL of each other.
- Normality will be exact only if the same indicator is used in determination as in standardization.
- Several other standardization procedures are available. See AOAC. 1990. Official Methods of Analysis. 15th Ed. 936.15.
- Tris-hydroxymethyl-aminomethane (THAM), primary base standard, may be

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substituted for standardized NaOH in steps 3 and 4.

Calculations

$$Normality\ of\ \ HCl = \frac{mL\ of\ \ Base\ Std\ \ X\ \ Normality\ of\ \ Base\ Std}{mL\ of\ \ HCl\ in\ \ flask}$$

Base Std = NaOH or THAM primary base standard

If the normality is too high, dilute solution to required normality by following formula:

$$V\ = \frac{V_2\ X\ N_2}{N_1}$$

N_2 = normality of stock
V_2 = volume of stock solution
V_1 = volume to which stock solution should be diluted to obtain desired normality (N_1).

Reference:

Standard Solution of Hydrochloric Acid. (936.15) Official Methods of Analysis. 1990. Association of Official Analytical Chemists. 15th Edition.