

Application Note Standardization of 0.5mol/L sodium nitrite

Industry Instrument Measurement method Standards Chemicals Automatic potentiometric titrator Polarization titration at constant voltage

1. Scope

Sodium nitrite is employed as a titrant in diazotization titration to measure primary aromatic amines, which are raw materials for products such as azo dyes. This Application Note introduces a method for the standardization of 0.5 mol/L sodium nitrite using amidosulfuric acid (also known as Sufamic acid) as a standard (Note1).

2. Precautions

To prevent decomposition and volatilization of nitrous acid, cool the solution to 15 $^{\circ}$ C or less during measurement.

3.-Post-measurement procedure

The platinum part of the twin platinum electrode should be wiped with a lens cleaning tissue to keep it clean.

4. Apparatus

Main unit	Automatic potentiometric titrator (Polarization titration preamplifier POT)
Electrode	Twin platinum electrode
	Temperature compensation electrode

5. Reagents

Titrant	0.5 mol/L Sodium nitrite solution
Additive reagents	Concentrated hydrochloric acid, Potassium bromide
Others	Pure water, Ice chips of frozen pure water

6. Procedure

-Calibration-

- 1) Prepare sample solution for titration.
- 2) Set the constant voltage value to 500 mV on the calibration setting screen.
- 3) The twin platinum electrode is immersed in the sample solution and the calibration is performed.

-Drying of amidosulfuric acid-

Dry amidosulfuric acid standard for volumetric analysis at 50 °C for 2 hours.

-Measurement-

- 1) Collect 0.3 g of dried amidosulfuric acid in a beaker and measure the mass.
- 2) Add 50 mL of pure water, 5 mL of conc. hydrochloric acid, 5 g of potassium bromide, and 30 g of ice chips.
- 3) Titrate with 0.5 mol/L sodium nitrite while keeping the liquid temperature below 15 $^{\circ}$ C.

7. Calculation

S

0.5 mol/L Sodium nitrite factor = S / $(0.048548 \times EP1) \times R/100$

Sample size (g)

- EP1 Titration volume (mL)
- R Purity of amidosulfuric acid (%)

8. Example

— Parameter —

<titr. mode=""></titr.>	Intermit titration mode	<u><ctrl. para.=""></ctrl.></u>	
<u><titr. form=""></titr.></u>	Automatic intersection detection	Number of EP	1
		End Sense	Auto
<u><titr. para.=""></titr.></u>		Angle	40 Deg.
Max Volume	20 mL	Gain	1
Channel/Unit(Ctrl.)	Ch3, µA	Data Sampling	ON
Wait Time	0 s	Data Samp.Pot.	999 mV
Titr. Type Check	No Check	Data Samp.Vol.	0.1 mL
		Ctrl. Speed	ON
		Cut-off time	15 s
		Unit volume	0.1 mL
		Dispense speed	1 s/mL
		Stirrer Speed	2

(Listed above are example settings. Availability of settings may vary by instrument model.)

- Example of titration curve -





- Measurement results -

Table 1 Measurement result

n	Sample (g)	Titration (mL)	factor
1	0.3481	7.1394	1.0043
2	0.3073	6.3183	1.0018
3	0.2913	5.9631	1.0062
Average	-	-	1.0041
SD	-	-	0.0022
RSD (%)	-	-	0.22

9. Notes

Note 1) The chemical reaction between sodium nitrite $(NaNO_2)$ and amidosulfuric acid (HSO_3NH_2) is shown below.

 $NaNO_2 + HSO_3NH_2 \rightarrow NaHSO_4 + H_2O + N_2$

The measurement method used was polarization titration at a constant voltage. In this titration, the endpoint is the intersection point of the titration curve.

