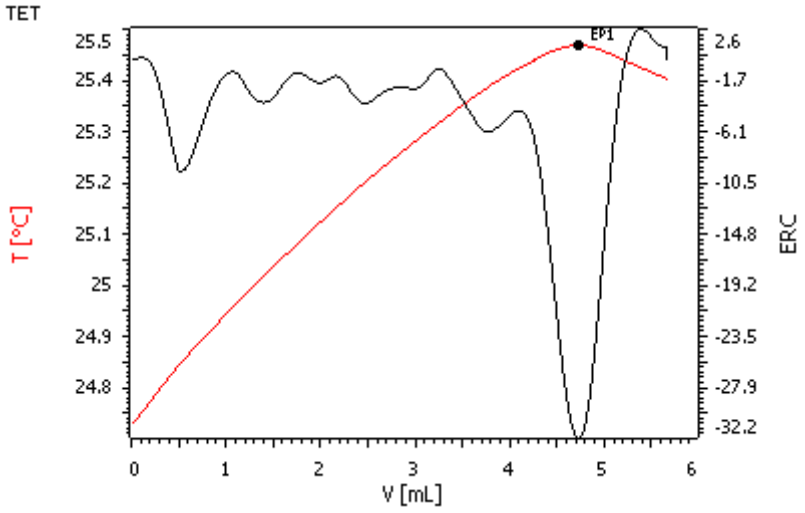
Titration Application Note H–130

Determination of nitrite by sulfamic acid



This Application Note describes the determination of nitrite by thermometric endpoint titration with sulfamic acid. The nitrite content of a solution may be analyzed down to approximately 0.2 mmol/L.

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Method description

Principle
Nitrite reacts in a strongly exothermic manner with sulfamic acid:

$H^+ + NO_2^- + H_3NSO_3 \rightarrow H_2SO_4 + N_2 + H_2O$

Sample
An approximately 0.02 mol/L solution of nitrite was prepared from reagent grade potassium nitrite.

Sample preparation

None.

Configuration

Basic equipment list for automated titration

814 USB Sample Processor	2.814.0030
859 Titrotherm	2.859.0010
Sample rack 24 x 75 mL	6.2041.340
Thermoprobe	6.9011.020
Sample beaker 75 mL	6.1459.400
802 Stirrer	2.802.0010
Stirring propeller 104 mm	6.1909.020
2 × 800 Dosino	2.800.0010
1 × Dosing unit 10 mL	6.3032.210
1 × Dosing unit 5 mL	6.3032.150
tiamo™	6.6056.222

Solutions

Titrant	c(H_3NSO_3H) = 0.1 mol/L sulfamic acid
Reagent	Concentrated HCl

Analysis of sample
A suitable aliquot of solution containing approximately 0.2–0.5 mmol nitrite is pipetted into a titration vessel, and deionized water added to bring the volume to approximately 30 mL. The *tiamo*™ titration program adds 1 mL concentrated HCl by Dosino immediately prior to the titration with H_3NSO_3H.

Determination of systematic error ("blank")
Prepare an approximately 0.02 mol/L nitrite solution, and pipette aliquots ranging from 10–25 mL into titration vessels. Make to approximately 25 mL with deionized water, and titrate as above. Determine the systematic error by regression analysis, plotting aliquot volume on the x axis, and mL H_3NSO_3H titrant on the y-axis. The y-intercept (in mL) is the systematic error of the determination.

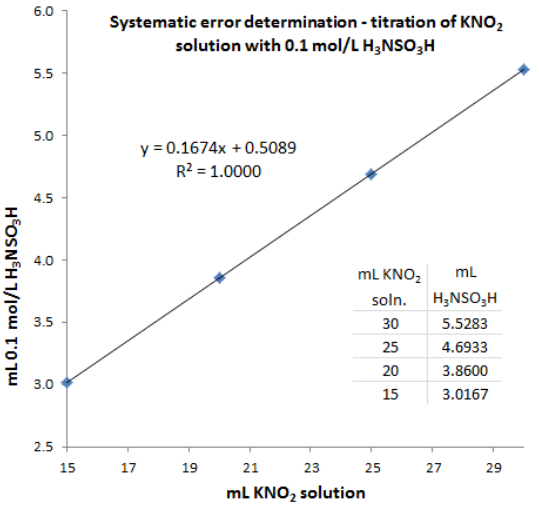
Standardization of titrant.
Sulfamic acid is a recognized primary standard for acidimetry, and is stable and non-hygroscopic in solid form. However, aqueous solutions hydrolyze slowly, and so it is prudent to make up small quantities and discard after a few days.

Parameters
Basic experimental parameters

Titrant dose rate (mL/min)	2
ERC EP1 (exothermic)	–25
Data smoothing ("filter factor")	70
Stirring speed (802 Stirrer)	15
Evaluation start (mL)	1
Damping until (mL)	1

Results:
Analysis of ≈0.02 mol/L KNO_2 solution:
NO_2^- = 0.795 ± 0.002 g/L

Blank determination



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