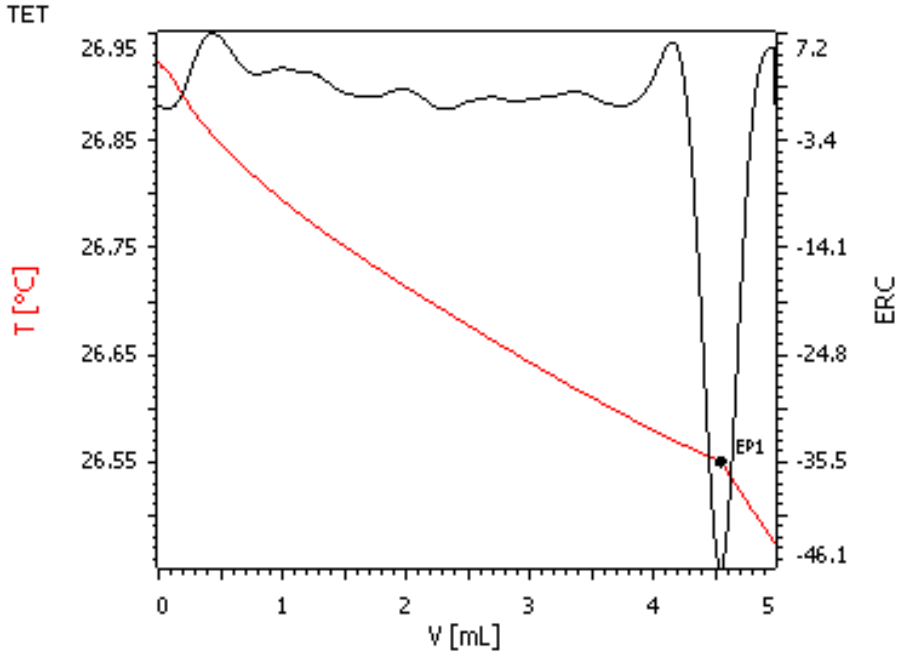
Titration Application Note H–132

Thermometric endpoint titration of hydrogen peroxide by iodometry



Hydrogen peroxide solutions may be determined by an iodometric thermometric endpoint titration (TET). Iodide is oxidized to iodine, which is reduced exothermically by standard sodium thiosulfate titrant.

Metrohm

Method description

Principle

A diluted sample of hydrogen peroxide is acidified with acetic acid, and potassium iodide is added. Because hydrogen peroxide oxidizes iodide fairly slowly, the reaction is catalyzed by the addition of a small amount of ammonium molybdate. The iodine generated is titrated with standard sodium thiosulfate solution.
(1 mol H_2O_2 ≙ 2 mol $S_2O_3^{2-}$)

Samples

Samples of disinfectant grade solutions of hydrogen peroxide

Sample preparation

For best accuracy, samples must be diluted before use. An appropriate amount of hydrogen peroxide solution is weighed in a 200 mL volumetric flask and make up to volume with deionized water. The following table may be used as a guide.

% H_2O_2	Approximate mass to be weighed / g
50	2.0
35	2.5
15	5.0
8	10.0

Configuration

859 Titrotherm	2.859.1010

Solutions

Titrant	c($Na_2S_2O_3$) = 1 mol/L sodium thiosulfate solution
Iodide solution	β(KI) = 500 g/L potassium iodide solution
	Glacial acetic acid
Catalyst solution	β($(NH_4)_2MoO_4$) = 40 g/L ammonium molybdate solution, stabilized with 1 mL 28% NH_3 solution/100 mL.

Analysis of samples

Pipette 20 mL of an aliquot into a titration vessel, and add immediately ***in this order*** 5 mL glacial acetic acid, 5 mL iodide solution and 0.1 mL catalyst solution. After 15 seconds, the titration is started.
The titration will proceed to an exothermic endpoint. *Note that additions of $Na_2S_2O_3$ solution generally cause a decrease in temperature. Nonetheless, the reaction with iodine is definitely exothermic.*

Parameters

Titrant dose rate	2 mL/min
ERC EP1 (exothermic)	–25
Filter factor	50
Stirring speed	8
Evaluation start	0.5 mL
Damping until	0.5 mL

Calculations

$$w_{H_2O_2} = \frac{(V_{EP1}\text{-Blank}) \times c_{Na_2S_2O_3} \times f \times M_{H_2O_2} \times 0.1}{m_s \times 2}$$

V_{EP1}: endpoint in mL
Blank: mL, determined by regression analysis
$c_{Na_2S_2O_3}$: concentration of titrant in mol/L
f: correction factor (“titer”)
$M_{H_2O_2}$: molar mass of H_2O_2 (34.01468 g/mol)
0.1: conversion factor
m_s: sample mass in g
2: stoichiometric factor

Blank determination

The blank value needs only to be determined during the initial setup, or when the titrant dose rate or filter factor is altered.

Prepare a test solution as above, such that a 25 mL aliquot will produce a titration volume of approximately 5 mL.
Prepare and titrate aliquots of 10, 15, 20,and 25 mL of this solution, making to 30 mL with deionized water where necessary.
In ***tiamo***™, the aliquot volume in mL (x-axis) is plotted against endpoint volume in mL (y-axis), and the y intercept value is determined. This value has to be saved as a common variable in ***tiamo***™.

Results

Content H_2O_2 / %	RSD / %
15.02 (n = 5)	0.33

Metrohm