HIRANUMA APPLICATION DATA	Automatic Titrator	Data No.	O10	Feb. 03,2022

Factor standardization	Standardization of EDTA titrant

1.Abstract

Disodium dihydrogen ethylenediamine tetraacetate (hereinafter referred to as EDTA) is a chelating agent and has the property of forming chelate compounds with many metal ions. It is used as a titrant for a chelatometric titration method to quantify metal ions. The chelatometric titration method has a wide range of applications, various metal ions could be measured with the use of appropriate indicator reagent and pH.

Factors are indicated on the commercially available standard solution for volumetric analysis. The factor determination is required when the standard solutions are prepared in the laboratory. Also it is effective to check the repeatability by the factor measurement using a standard material to check the performance of titrator system. *Japanese Industrial Standard* JIS K 8001 and the *Japanese Pharmacopoeia* describe that zinc, which is a standard material for a volumetric analysis, should be used for the factor determination of EDTA standard solution.

In this report, zinc standard solution, which is prepared from standard material for volumetric analysis, is used for standardization of EDTA titrant. 1 mol of zinc and 1 mol of EDTA react quantitatively according to formula (1).

$$Zn2+ + EDTA \rightarrow Zn\cdot EDTA + 2Na+ \cdots(1)$$

In this method, an indicator reagent is used to determine the end point based on the color change of the sample solution. The indicator reagent has the property of binding to metal ions unreacted with EDTA, and the color changes depending on whether or not they bind with metal ions. The indicator reagent used in this report, Eriochrome Black T (hereinafter, EBT), changes the color of the solution from red to blue before and after the end point. By detecting this color change with a photometric probe, the end point of zinc titration with EDTA titrant is determined.

1) Japanese Pharmacopoeia Eighteenth Edition
2) Japanese Industrial Standard JIS K8001 General rules for test methods of reagents

2. Configuration of instruments and Reagents

(1) Configuration of instruments

Main unit	:	Automatic Titrator COM Series (with photometric unit, M-type)
Electrodes	:	Photometric probe (with 650 nm interference filter)
		Glass-reference combination electrode GR-501BZ

(2)Reagents

Titrant	:	0.01 mol/L (0.1 N) EDTA standard solution (Buret No. 1)
Standard material	:	0.01 mol/L (0.1 N) zinc standard solution (f = 1.028)
		0.6720 g of metallic zinc (purity 100.00%) was dissolved in 20 mL of 20 % hydrochloric acid, dilute to 1 L

/ [prepared volume (L)×nominal molar concentration(mol/ L)]

= [0.6720 / 65.38]×[100.00 / 100] / [1×0.01] = 1.028

pH adjuster (1) : 100 g/L sodium hydroxide solution

pH adjuster (2) : pH 10 ammonia buffer
Dissolve 7 g of ammonium chloride in 30 mL of DI water, add 57 mL of 30 % ammonia solution and dilute to 100 mL with DI water.

Indicator reagent : EBT indicator reagent
Prepared by dissolving 0.5 g of EBT and 4.5 g of hydroxylammonium chloride in 100 mL ethanol.

3. Measurement procedure

(1) Take 25 mL of 0.01 mol/L zinc standard solution into a 200 mL tall beaker with a volumetric pipette.
(2) Add 75 mL of DI water and a stirrer bar to the beaker.
(3) Immerse the glass-reference combination electrode to check pH of the solution. Add 100 g/L sodium hydroxide solution to adjust the pH to about 9.5-10.
(4) Add 2 mL of pH 10 ammonia buffer.
(5) Add 3 drops of EBT indicator reagent.
(6) Immerse the photometric probe and start titration with 0.01 mol/L EDTA titrant. The inflection point appearing on the titration curve is detected, and the end point is determined by B-Cross detection method.

4. Measurement conditions and results

Examples of titration conditions

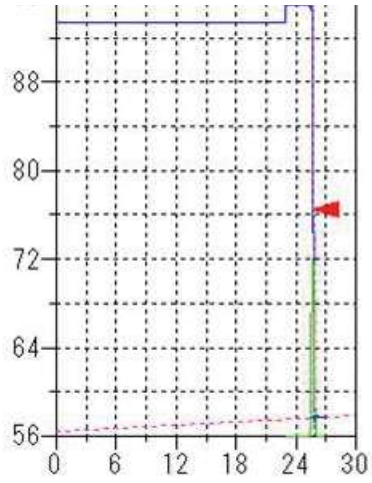
Cndt No.	1							
Method	B-Cross		Constant No.	1		Mode No.	21	
Buret No.	1		Size	25	mL	Pre Int	0	sec
Amp No.	2		Blank	0	mL	Del K	0	
D. Unit	T%		Molarity	0.01	mol/L	Del Sens	0	mV
S-Timer	5	sec	Factor	1.028	*1	Int Time	5	sec
C.P. mL	23	mL	K	0		Int Sens	5	mV
T Timer	10	sec	L	0		Brt Speed	2	
D.P. mL	1.0	mL				Pulse	80	
End Sens	200		Unit	Fact1				
Over mL	1.0	mL	Formula	S/(D-B)*F				
Max Vol.	40	mL	Digits	4				

*1: The factor of 0.01 mol/L zinc standard solution

Measurement results

Measurement No.	Sample Size(mL)	Titrant volume (mL)	Factor	Statistical results	
1	25	25.803	0.9960	Avg.	0.997
2	25	25.767	0.9974	SD	0.001

3966 RSD 0.07 %



Factor standardization measurement

Examples of measurement curves

5.Note

(1) About the parameter setting

In this titration, the color change of indicator occurs suddenly and sharply at the end point. To prevent the end point from being passed due to excessive increment volume of the titrant, the volume of each drop should be constant at 0.1 mL, which is set in the parameter “Pulse”. Setting the parameter “Del K” to zero allows the titration without dynamic control of the titrant increment volume. For the titrations where the electrode signal changes rapidly at the end point, this setting will improve the repeatability. The measurement time can be shortened by using the parameter “CP mL”, which set titrant volume 0.5-1.0 mL before the end point.

In addition, since end point of this titration is defined at the point where the color change of the indicator is completed, the end point detection is set to “B-Cross” on Method in parameter. If the beginning of color change should be defined as the end point, such as back-titration method, set the detection method to “F-Cross”.

(2) About selection of interference filter for photometric probe

The photometric probe comes with two types of interference filters, 530 nm and 650 nm. In this report, the EBT indicator reagent is used in the alkaline pH range, and the solution color changes from red to blue at the end point, thus 650 nm was selected for the filter. For chelatometric titration of zinc, there are other titration method using other indicator reagent and pH range. As shown in Application Data No. G1, when the indicator xylenol orange is used in the acidic pH, the color change at the end point is from red-purple to yellow and the 530 nm filter is used.

In general, when the color of the solution is (orange, red, and red-purple), the transmittance (%T) decreases relatively large with using a filter at 530 nm, and when the color of the solution is (green, and blue), the transmittance decreases at 650 nm. If multiple colors are mixed in the solution, it would not be as described above. In that case, select the wavelength of filter that can detect the larger change in transmittance.

Keywords： Factor standardization, Chelatometric titration, Photometric titration, EDTA, Disodium dihydrogen ethylenediamine tetraacetate, Zinc, Eriochrome black T, EBT

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