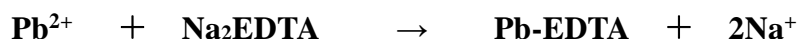


HIRANUMA APPLICATION DATA	Automatic Titrator	Data No.	G9	Apr. 5, 2019
Metals	Quantitative determination of lead ion			

1. Abstract

The chelatometric titration is generally used for the determination of lead ion (Pb²⁺). The pH region the lead ion can be directly titrated is pH 3.5 ~ 10 (stability constant = 17.88).¹ However, it generates Pb(OH)₂ precipitation at alkaline region. When performing titration under alkaline condition, the auxiliary complexing agent such as ethanolamine, tartaric acid, or citric acid should be added in advance to generate weak chelatometric complex and avoid the generation of lead hydroxide precipitation.

It is titrated at pH around 4 ~ 5 when performing under acidic condition. This report introduces an example that the lead ion in sample solution adjusted to pH 5 by hexamine solution is determined with using XO indicator (red purple →yellow).



2. Configuration of instruments and reagents

(1) Configuration

Main unit : Hiranuma Automatic Titrator COM series (Photometric titrator unit type M)
with 530 nm optical filter

Electrode : Glass - Reference electrode GR-501BZ (for pH confirmation)

(2) Reagents

Titrant : 0.01 mol/L EDTA standard solution

Buffer solution : 5 ~ 10 mL of 20 % hexamine solution

Indicator : 0.2 mL of XO indicator (0.1 % solution)

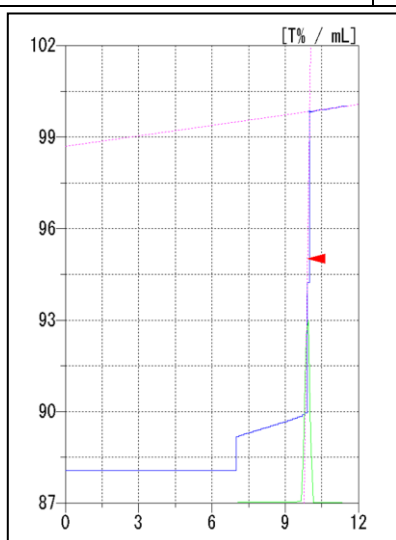
3. Measurement procedure

- (1) Dispense 1 mL of sample into a 100 mL beaker with volumetric pipette.
- (2) Add about 60 mL of DI water.
- (3) Add hexamine solution to adjust the pH to 5.
- (4) Add 0.2 mL of XO indicator.
- (5) Immerse photometric probe into sample solution and titrate with 0.01 mol/L EDTA standard solution.

4. Measurement conditions and results

Examples of titration conditions

Cnd. No.	1	Constant No.	1	Mode No.	24
Method	B cross	Size	1 mL	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	0
Amp No.	2	Molarity	0.01 mol/L	Del Sens	0 mV
D.Unit	T%	Factor	1.001	Int Time	5 sec
S- Timer	15 sec	K	207.2	Int Sens	3 mV
C.P. mL	7 mL	L	0	Br Speed	2
T.Timer	15 sec	Unit	g/L	Pulse	80
D.P. mL	0.1 mL	Formula	(D-B)*K*F*M/S		
End Sens	300	Decimal Places	4		
Over mL	1.5 mL	Auto input parameter	None		
Max Vol.	12 mL				



Example of titration curve

Measurement results

Number of Measurement	Size (mL)	Titrant Volume (mL)	Lead ion Concentration (g/L)
1	1	9.998	20.737
2	1	9.997	20.735
3	1	9.931	20.598
Statistic calculation		Avg.	20.69 g/L
		SD	0.080 g/L
		RSD	0.385 %

5. Note

(1) Measurement condition

The color of indicator reagent suddenly changes at the endpoint, therefore the minimum increment titration without “Del K” control allows to get good results. The function “CP mL” can reduce the measurement time by continuously adding slightly smaller volume of titrant than the titrant consumption required to the endpoint. The “Method” on condition parameter is set to “B Cross” because the endpoint is defined as the point where the color change of the indicator is completed.

(2) Interfering ions

There is no interference on the measurement even if alkaline-earth metal or Mg is contained in the sample. However, please note that Ni²⁺, Fe³⁺, Zn²⁺, Cd²⁺, and Co²⁺ ions are also titrated at the same time.

Keywords : Lead ion, Photometric titration, Chelatometric titration

References

(1) K. Ueno, “Chelatometry”, 1989, Nankodo, Tokyo.

*Some measurement would not be possible depending on optional configuration of system.