

Mineral Ore

Calcium Oxide in Ash

Chelatometric titration by
Automatic Potentiometric Titrator

Standard

1. Abstract

Calcium oxide in ash is measured first by preparing test sample by adding nitric acid to dissolve and add pure water. Add Potassium hydroxide to the sample liquid, and titrate with 0.01mol/L EDTA. The endpoint is obtained by color change of the indicator on titration curve.

The Calcium oxide is calculated from titration volume of EDTA.

2. Reference

- 1) Chelatometry by Kagehira Ueno from Nankoudo Publication
- 2) Experiment and Calculation for Quantitative Analysis –Vol. by Seiji Takagi from Kyoritsu Publishing Company

3. Cautions in measurement

- 1) Handle with care when you work on chemicals. Work in a well ventilated room or use a draft.
- 2) Reactivity of sample and titrant may change in chelatometric titration. Adjust pH to the optimal level.

4. Post-measurement care

Clean the photo sensor with pure water thoroughly.

5. Test equipment

Main unit : Automatic potentiometric titrator
(Option Photometric preamplifier PTA-)
Electrode : Option Photo sensor
Interference filter (530nm)

6. Reagent

Titrant : 0.01mol/L EDTA
Additive : Pure water, Nitric acid, 8mol/L Potassium hydroxide
Indicator : NN indicator (mixed with 100-times mass of potassium sulfate)

7. Measurement procedure

—Preparation—

- 1) Sample 2g in a 100mL beaker.
- 2) Add nitric acid to dissolve sample and filter the solution.
- 3) Add pure water to make it 1000mL in total as for prepared sample.

—Measurement—

- 1) Deliver 5.0mL to be exact in a 100mL beaker, and add pure water.
- 2) Add 0.1g of NN indicator and 4mL of 8mol/L Potassium hydroxide.
- 3) Titrate with 0.01mol/L EDTA to obtain concentration of Calcium oxide.

8. Formula

Calcium oxide (%) = $(EP1 - BL1) \times FA1 \times C1 \times K1 / SIZE$

EP1 : Titration volume (mL)

BL1 : Blank level (0.00mL)

FA1 : Factor of titrant (1.00)

C1 : Concentration conversion coefficient (0.5608mg/mL)
(0.01mol/L EDTA 1mL \equiv 0.5608mg CaO)

K1 : Unit conversion coefficient (0.1)

SIZE : Sample size (g)
(Use 1/200 of prepared sample)

9. Example of measurement

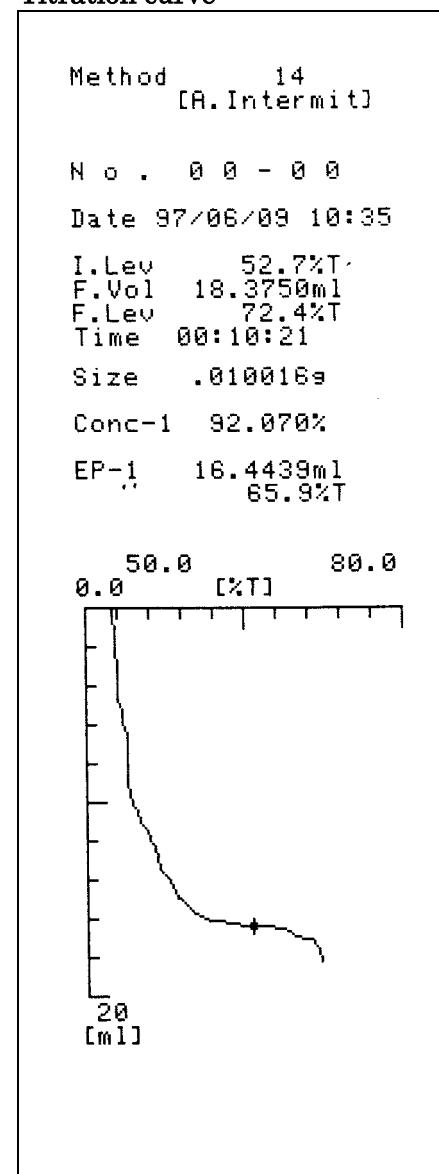
— Ambient condition —

Room temperature : 22 °C	Humidity : 57 %	Weather : Cloudy
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-Titration parameter-

Model : AT-400	
Method No. : 14	
Titr.mode : Auto	
Intermit	
Titr.form : EP Stop	
[TITR. PARA]	[CALCU. PARA]
Form : EP Stop	Sample Measurement
Buret No. : 1	Conc1 CalcuNo. 2
Preamp : PTA	Conc1 Dim. [%]
Detector No. : 3	Conc1 EP Position 1
Dimension : %T	Data [f(EP2-Blank)]
Max.Vol : 40.0mL	T.Type [Normal]
W.Time : 0s	Data [f(T.Vol)]
Direction : Auto	Local Blank
	Blank 0.0mL
	Common T.Factor
	K1 0.1
	C1 0.5608mg/mL
	Temp.Comp. [Off]
[CTRL. PARA]	
End Point No. : 1	
S(dE) : 50	
S(E/mL) : 100	
O.Titr : 0mL	
Gain : 1	
S.Pot : 4.0mV	
Stab. : 0.5mV/s	
Delay Time : 1s	
L.Time : 30s	
M.Unit : 0.5mL	
Separation : Off	
A.Simulation : Off	

-Titration curve-



(The above printout data were obtained from titration by AT-400)

«Titration parameter»

Form: of titration / APB No. the burette used in titration / Dimension: of potential unit
 Detector No.: the detector used in titration/ Unit of potential / Max Volume. of titration
 Wait Time: before titration starts/ Direction.: of titration

«CTRL. PARA: Control parameter»

End Point No. number of EPs detected / S(dE): EP potential (difference) / S(E/mL): EP potential (differential)
 Over Titr.Vol. over-titration volume / Gain: sensitivity of detection signal/ S.Pot.: potential changes of sampling signal
 Stab.: stability sense potential/ L.Time: time for stability check /M.Unit.: titration for sampling signal /Separation: of potential/ A.Simulation: redetection of EP

«CALCU. PARA: Result parameter»

Calc.No.: of formula / Conc.1: formula 1 / Conc1 Dim: unit of concentration/Conc1EP Position: EP at conc. 1
 K1: unit conversion coefficient/ Factor: of reagent /Blank : blank level /Data: titration form used in calculation
 T.Type: titration type (normal or back) /Common T.Factor: factor of titrant registered
 C1(mg/mL): conc. at EP1/Data: calculation of titration / Temp.Comp.: temperature compensation

—Measurement results—

n	Sample (g)	Titration (mL)	Calcium oxide (%)
1	0.010016	9.0033	50.410
2	0.010016	9.0003	50.393
3	0.010016	9.0094	50.444

Concentration of Calcium oxide	
Mean	50.416 %
SD	0.026 %
RSD	0.052 %

* The above results were obtained by 3 tests of the same sample.

* Red underline shows the data from page 3/4.

10. Summary

The ash is the residue after plants and animals are burned down. Main component elements of ash are Calcium and Potassium with trace amount of Aluminum·Ion·Zinc. These substances exist as oxide or carbonate, and when dissolved in water, they usually show strong alkalinity.

The test result shows a good repeatability with 0.05% relative standard deviation.

Precise and reliable measurement is assured by the automated potentiometric titration.