

Iron and Steel

Cobalt in Cemented Carbide

Redox titration by
Automatic Potentiometric Titrator

Standard

CIS 032B

1. Abstract

Cobalt in Cemented Carbide is measured according to CIS 032B-2007 Potentiometric titration of Cobalt in Cemented Carbide. Titrate with 1/30mol/L Potassium ferricyanide up to the endpoint, which is the maximum inflexion on titration curve.

Cobalt concentration is calculated from titration volume of Potassium ferricyanide.

The principle is quantification by oxidizing Cobalt (II) to Cobalt (III) in strong Ammonia solution using Potassium ferricyanide.

2. Reference

- 1) CIS 032B-2007 Potentiometric titration of Cobalt in Cemented Carbide
- 2) “Experiment and Calculation for Quantitative Analysis ” –Vol.2 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) Check reagent of Potassium ferricyanide on concentration as appropriate.

4. Post-measurement care

Rinse the electrode with pure water, and keep it dipped in a beaker filled with pure water to refrain from drying up.

5. Test equipment

Main unit: Automatic potentiometric titrator (Standard preamplifier STD-)

Electrode: Platinum electrode

Ceramic type reference electrode

6.Reagent

- Titrant : 1/30mol /L Potassium ferricyanide (Hexacyanoferrate(III)) solution
Additive : Pure water, Nitric acid (1+1), Hydrofluoride acid, Ammonia water, Ammonium citrate
Reagent : Standard cobalt solution

7.Measurement procedure

—Preparation—

- 1) Dissolve 300g Citric acid (Monohydrate) in 500mL pure water, and neutralize to pH7 by adding ammonia water while cooling. Then, add water to make 1000mL in total for preparing Ammonium citrate.
- 2) Dissolve 11g of Potassium ferricyanide in water making 1000mL in total for preparing 1/30mol/L Potassium ferricyanide solution.
- 3) Dissolve 1000g Cobalt (purity more than 99.9%) in 20mL Nitric acid (1 + 1), and cool. Transfer cooled Cobalt to a 1000mL measure flask, and dilute with water up to the mark as for standard cobalt solution.

—Check—

- 1) Transfer 50.0mL standard cobalt to a 300mL beaker.
- 2) Add 100mL of Ammonium citrate, and cool it below 15°C. After cooled, add 80mL of Ammonia water.
- 3) Titrate with 1/30mol/L Potassium ferricyanide to obtain the equivalence to 1mL Cobalt from the following equation:

$$f = G / V$$

f : Cobalt (g) equivalent to 1mL of Standard potassium ferricyanide

G : Cobalt (g)

V : Consumed volume of Potassium ferricyanide (mL)

—Measurement—

- 1) Prepare 0.2g sample in a 500mL Polyethylene beaker.
- 2) Add 20mL Nitric acid (1+1) and warm it over hot water. Add Hydrofluoric acid by one drop after another to dissolve slowly. After dissolved completely, cool it until liquid volume comes down to approximately 5mL.
- 3) After cooled, add 50mL water and 100mL ammonium citrate, and cool it in bath below 15°C.
- 4) Add 80mL Ammonia water, and immediately dip the electrode. Titrate with 1/30mol/L Potassium ferricyanide to obtain concentration of cobalt.

8.Formula

$$\text{Cobalt (\%)} = (\text{EP1} - \text{BL1}) \times \text{FA1} \times \text{C1} \times \text{K1} / \text{SIZE}$$

EP1 : Titration volume (mL)

BL1 : Blank level (0.00mL)

FA1 : Factor of tyrant (1.9857g/mL)

C1 : Concentration conversion coefficient (1)

K1 : Unit conversion coefficient (0.1)

SIZE : Sample size (g)

9. Example of measurement

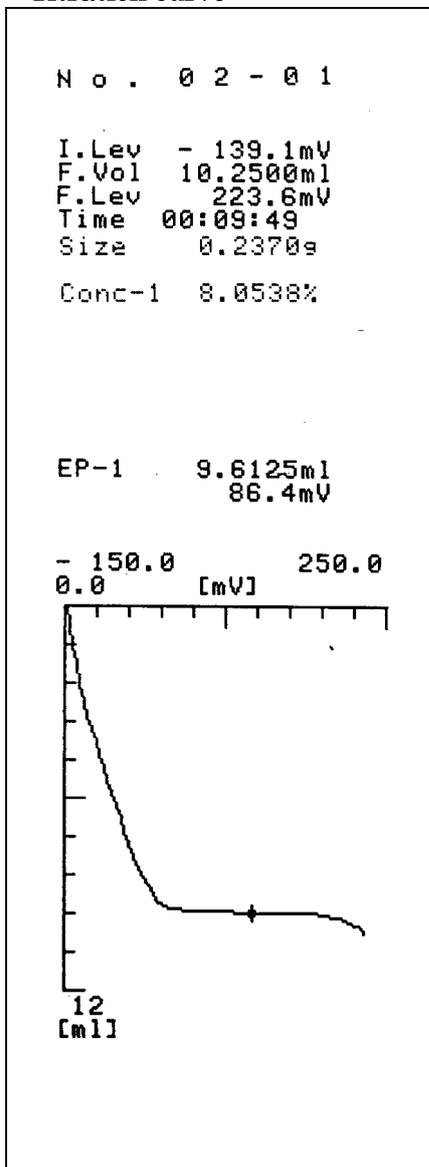
— Ambient condition —

Room temperature : 24 °C	Humidity : 85 %	Weather : Rainy
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- Titration parameter -

Model : AT-400	
Method No. : 18	
Titr.mode : Auto	
Intermit	
Titr.form : EP Stop	
[TITR. PARA]	[CALCU. PARA]
Form : EP Stop	Sample Measurement
Buret No. : 1	Conc1 CalcuNo. 2
Preamp : STD	Conc1 Dim. [%]
Detector No. : 2	Conc1 EP Position 1
Dimension : mV	Data [f(EP2-EP1)]
Max.Vol : 20.0mL	T.Type [Normal]
W.Time : 0s	Data [f(T.Vol)]
Direction : Auto	Local Blank
	Blank 0.00mL
	Local T.Factor
	Factor 1.9857
[CTRL. PARA]	K1 0.1
End Point No. : 1	C1 1.0mg/mL
S(dE) : 50	Temp.Comp. [Off]
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 are obtained from titration by AT-400)

« TITR. PARA: Titration parameter »

Form: of titration / Buret No.: the burette used in titration / Detector No.: the detector used in titration
 Dimension: potential unit / 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
 Separation: of potential / 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 / Conc1 EP 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)	Cobalt (%)
1	<u>0.2370</u>	<u>9.6125</u>	<u>8.0538</u>
2	0.2265	9.1946	8.0608
3	0.2410	9.7968	8.0720

Concentration of cobalt	
Mean	8.0622 %
SD	0.0092 %
RSD	0.1139 %

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

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

10. Summary

Cemented Carbide is an alloy made from sintered hard metallic carbide powder, and is simply called Hard metal, which is used in making hard metal tools. Generally, it means sintered mixture of Tungsten carbide (WC) and Cobalt (Co) as binder. The material is used in making abrasion resistance cutting tools and dies.

Cobalt is an atomic number 27 element with chemical symbol Co.

The sample test shows a good repeatability with 0.11% relative standard deviation.

Precise and reliable measurement is assured by the automated potentiometry.