Silver Content Determination by Volhard Titration

The silver content is determined by precipitation titration with potassium thiocyanate KSCN as a titrant. The titration is monitored by a combined silver ring electrode.

Sample	Approx. 0.1 M silver nitrate sol., 3 mL $AgNO_3$
Compound	Silver, Ag^+ M = 107.88 g/mol, z = 1
Chemicals	10 mL 0.1 M Nitric acid, HNO_3 50 mL Deionized water
Titrant	Potassium thiocyanate, KSCN c(KSCN) = 0.1 mol/L
Standard	5 mL 0.1 M $AgNO_3$
Indication	DMi148-SC combined silver ring electrode
Chemistry	$Ag^+ + SCN^- \rightarrow AgSCN$
Calculation	R = Q*C/m, mol/L C = 1/z
Waste disposal	Filtrate the solution and dispose the precipitate as special waste.
Author, Version	Thomas Hitz, MSG AnaChem, Version 1.0

Preparation and Procedures

CAUTION

- Use safety goggles, a lab coat and wear gloves. If possible, work in a fume hood.
- Ensure accurate cleaning of sensor is sufficient after each titration.

Preparation of 0.1 mol/L KSCN:

- Weigh 2.430 g of solid KSCN and dissolve it in 100 mL deionized water in a 250 mL volumetric flask. After complete dissolution, fill up to the mark with deionized water.

Sample titration:

- Pipette 5 mL 0.1 mol/L into the titration beaker
- Add 10 mL 0.1 HNO_3
- Add 50 mL deionized water
- Connect the beaker to the titration stand, and start the method.
- After titration, rinse accurately with deionized water the electrode, the stirrer and the dispensing tube with water, and if necessary clean them with e.g. a paper tissue before starting next sample.

Remarks

- The application method has been developed for the mentioned sample. It may be necessary to optimize the method for your sample.
- The AgSCN-precipitate may be sticking onto the electrode, tube and stirrer surfaces. To improve the efficacy of the cleaning procedure, the precipitate can be dissolved with diluted ammonia solution.

Instruments	- Titration Excellence T50/T70/T90 (Other Titrators: depending on instrument type, manual operation and method changes are necessary) - XS205 Balance
Accessories	- 1 x 10 mL DV1010 glass burette - PP Titration beakers ME-101974 - LabX® pro titration - Spatula

Results

0.1 M $AgNO_3$

All results

Method-ID	AgDM148	
Sample	Silver	(1/3)
R1 (Content)	0.089	mol/L
Sample	Silver	(2/3)
R1 (Content)	0.090	mol/L
Sample	Silver	(3/3)
R1 (Content)	0.090	mol/L

Statistics

Method-ID	AgDM148	
R1	Content	
Samples	3	
Mean	0.090	mol/L
s	0.001	mol/l
srel	0.644	%

Titration curve

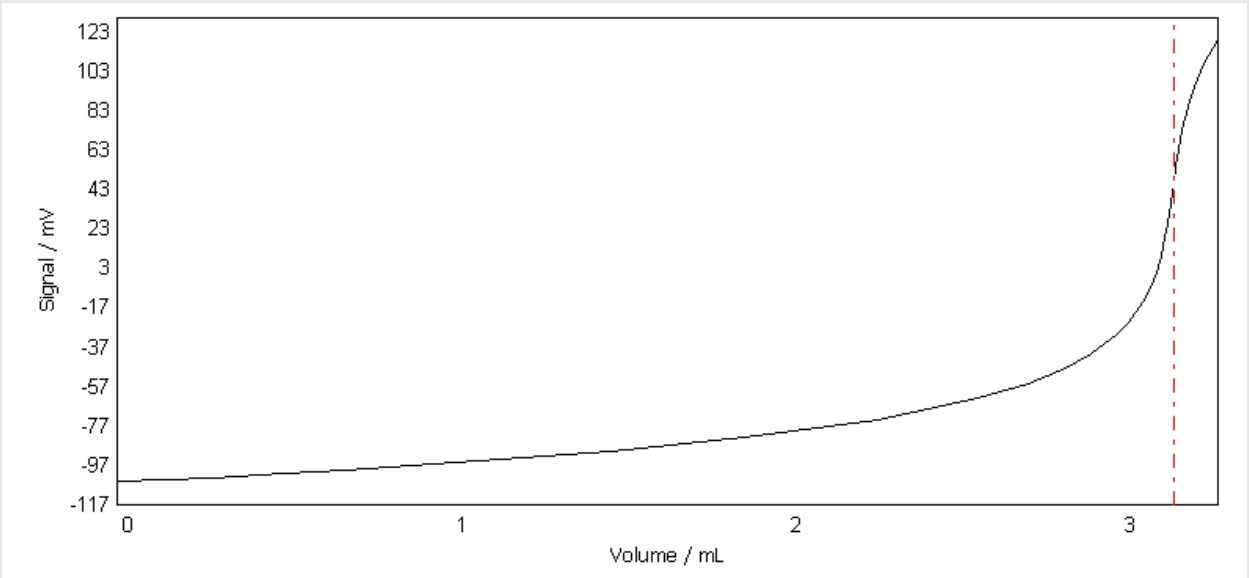

Table of measured values

	Volume mL	Increment mL	Signal mV	Change mV	1st deriv. mV/mL	Time s
	0.0000	NaN	105.3	NaN	NaN	0
	0.2855	0.2855	103.7	1.6	NaN	2
	0.4285	0.1430	101.6	2.1	NaN	5
	0.5000	0.0715	100.8	0.8	NaN	8
	0.6790	0.1790	99.1	1.7	NaN	16
	1.0790	0.4000	95.0	4.1	10.62	20
	1.4790	0.4000	89.9	5.1	12.66	22
	1.8790	0.4000	82.9	7.0	19.38	26
	2.2790	0.4000	73.7	9.2	34.35	29
	2.5780	0.2990	63.1	10.6	55.50	33
	2.7265	0.1485	55.7	7.4	76.38	36
	2.8390	0.1125	47.6	8.1	102.90	39
	2.9175	0.0785	40.6	7.0	135.97	42
	2.9885	0.0710	30.9	9.7	189.18	46
	3.0275	0.0390	24.8	6.1	245.61	49
	3.0715	0.0440	14.0	10.8	353.37	53
	3.0920	0.0205	7.4	6.6	450.07	56
	3.1115	0.0195	0.6	8.0	583.66	60
	3.1265	0.0150	8.8	8.2	715.51	63
	3.1375	0.0110	18.5	9.7	823.83	67
	3.1455	0.0080	25.5	7.0	950.42	70
	3.1550	0.0095	36.1	10.6	1077.87	74
EQP1	3.162279	NaN	44.8	NaN	1132.68	NaN
	3.1630	0.0080	45.7	9.6	1132.59	78
	3.1710	0.0080	55.1	9.4	1084.79	82
	3.1790	0.0080	63.1	8.0	980.99	85
	3.1885	0.0095	72.4	9.3	850.62	89
	3.1970	0.0085	79.2	6.8	751.00	92
	3.2095	0.0125	87.8	8.6	NaN	95
	3.2230	0.0135	95.0	7.2	NaN	99
	3.2420	0.0190	103.0	8.0	NaN	102
	3.2660	0.0240	111.1	8.1	NaN	105
	3.2955	0.0295	119.0	7.9	NaN	108

Comments

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Method

001 Title

Type	General titration
Compatible with	T50 / T70 / T90
ID	AgDM148
Title	AgCont
. . .	

002 Sample

Number of IDs	1
ID 1	SILVER
Entry type	Fixed volume
Volume	3.0 mL
Density	1.0 g/mL
Correction factor	1.0
Temperature	25.0°C

003 Titration stand (Manual stand)

Type	Manual stand
Titration stand	Manual stand 1

004 Stir

Speed	35%
Duration	15 s
Condition	No

005 Titration (EQP) [1]

Titrant

Titrant	KSCN
Concentration	0.1 mol/L

Sensor

Type	mV
Sensor	DM148
Unit	mV

Temperature acquisition

Temperature measurement	No

Stir

Speed	35%

Predispense

Mode	Volume
Volume	0.5 mL
Wait time	5 s

Control

Control	User
Titrant addition	Dynamic
dE (set value)	8.0 mV
dV (min)	0.008 mL
dV (max)	0.4 mL
Meas. val. Acquisition	Equilibrium controlled
dE	1.0 mV
dt	1 s
t (min)	3 s
t (max)	30 s

Evaluation and recognition

Procedure	Standard
Threshold	200.0 mV/mL
Tendency	Positive
Ranges	0
Add. EQP criteria	No

Termination

At Vmax	10.0 mL
At potential	No
At slope	No
After number of recognized EQPs	Yes
Number of EQPs	1
Combined termination criteria	No

006 Calculation R1

Result	Content
Result unit	mol/L
Formula	R1=Q*C/m
Constant	C=1
M	M[None]
z	z[None]
Decimal places	3
Result limits	No
Record statistics	Yes
Extra statistical func.	No
Send to buffer	No
Condition	No

007 End of sample