Determination of Nickel and Zinc

Description

The determination of nickel next to zinc can be done with the Optiline 6 and a color indicator.
In the method described here, EDTA is added to the sample in excess and the excess is titrated with magnesium sulfate. This gives the total content of nickel and zinc. 2,3-Dimercapto-1-propanol is then added to the sample. 2,3-Dimercapto-1-propanol forms a very stable complex with zinc. This releases EDTA from the Zn(EDTA) complex, which is titrated with magnesium sulfate. Eriochrome black T is suitable as an indicator, the optical sensor Optiline 6 is used for detection.
This titration can best be done with Titrisoft 3.4, since the entire process can be automated in one method. Without Titrisoft, 2 methods have to be created: the first method for the titration of Ni + Zn together, the second method for the titration of Zn after the addition of 2,3-Dimercapto-1-propanol.

Instruments

Titrator	TL 7000 or higher
Exchangeable head	WA 20
Electrode	Optiline 6
Cable	---
Stirrer	TM 235
Lab accessory	Beaker 150 mL
	Magnetic stirrer bar 30 mm

Reagents

1	EDTA – solution 0,1 mol/L
2	Magnesium sulfate solution 0,1 mol/L
3	Ammonium chloride / Ammonia buffer pH 10
4	Eriochrome black T trituration with NaCl
5	2,3-Dimercapto-1-propanol.
6	Ethanol 96%
7	Distilled water
All reagents should be of analytical grade or better.	

Titration procedure

Reagents

EDTA-solution 0.1 mol/L

Na_2EDTA solution 0.1 mol / L is available as a ready-to-use solution.

Magnesium sulfate-solution 0.1 mol/L

$MgSO_4$ solution 0.1 mol / L is available as a ready-to-use solution.

Buffer solution pH 10

54.0 g Ammonium chloride and 350 ml of Ammonia solution 25% are dissolved in dist. water and made up to 1.0 L with dist. water.

Eriochrome black T trituration

1.0g Eriochrome Black T and 49.0g NaCl are rubbed in a mortar until a homogeneous mixture is obtained.

2,3-Dimercapto-1-propanol solution

20,0g 2,3-Dimercapto-1-propanol are made up to 100 mL with Ethanol 96%.

Cleaning and storage of the electrode

The Optiline 6 is cleaned with distilled water. It is stored dry and clean.

Blank value

10 mL buffer solution pH 10 and approx. 50 mg Eriochrome black T trituration are placed in a 150 mL beaker and made up to approx. 80 mL with dist. water. 20.0 mL EDTA solution 0.1 mol/L are added. The mixture is titrated with MgSO4 0.1 mol/L up to the first equivalence point (color change, Optiline 6, wavelength 625 nm).

Sample preparation

The sample is pipetted into a 150 mL beaker, 10 mL buffer solution pH 10, approx. 50 mg Eriochrome black T trituration are added and made up to approx. 80 mL with dist. Water.

20.0 mL EDTA solution 0.1 mol/L are added and the mixture is titrated with MgSO4 0.1 mol/L up to the 1st equivalence point (color change, Optiline 6, wavelength 625 nm).

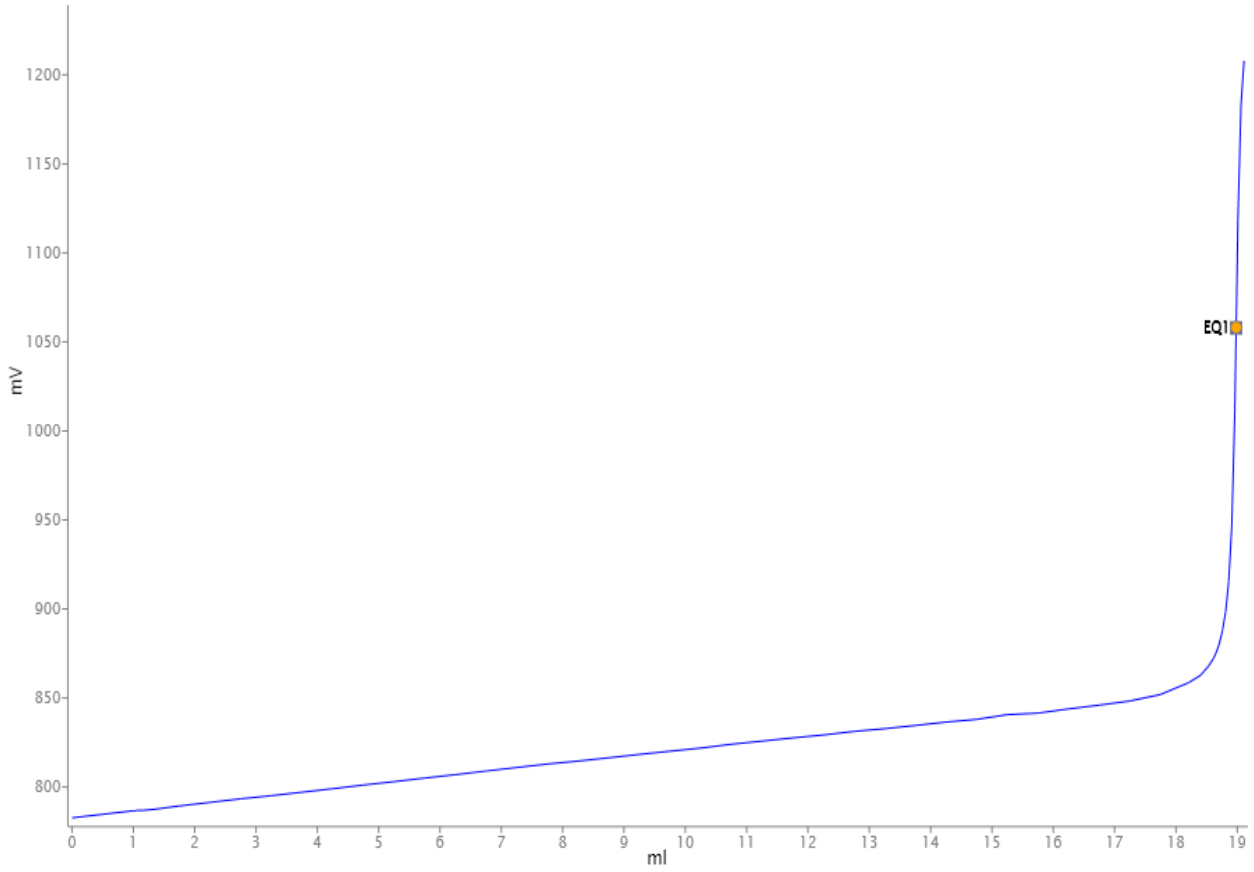
Then 2 mL 2,3-dimercapto-1-propanol solution are added and titrated again to the equivalence point (color change, Optiline 6, wavelength 625 nm).

The required sample amount can be estimated according to this rule of thumb:

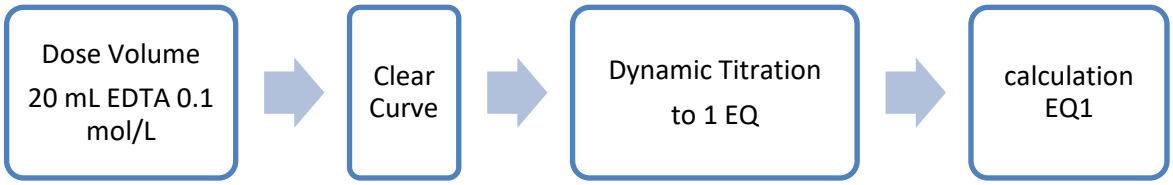
$$V(ml) = \frac{620 * Titer\ [\frac{mol}{L}]}{expected\ Ni/Zn - content\ [g/L]}$$

Titration parameter

Blank value



In Titrisoft the following method sequence can be used to determine the blank value:

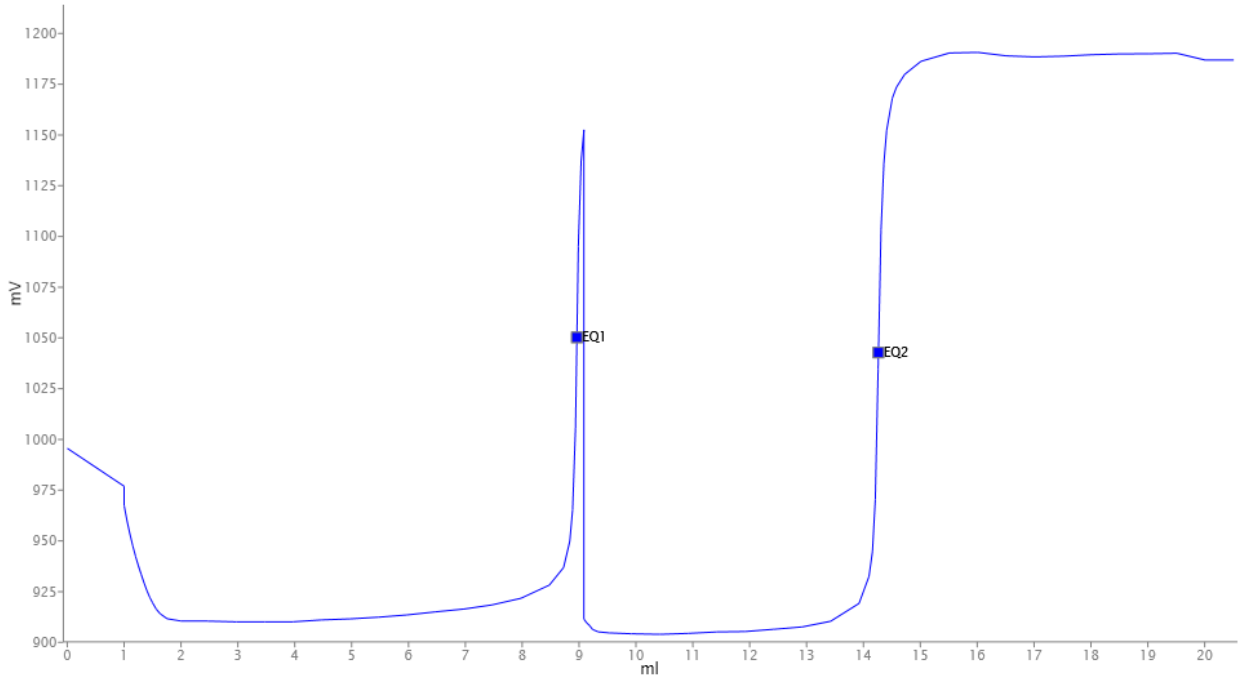


The result should be written to a global variable. So the blank value is available for calculations in the sample method.

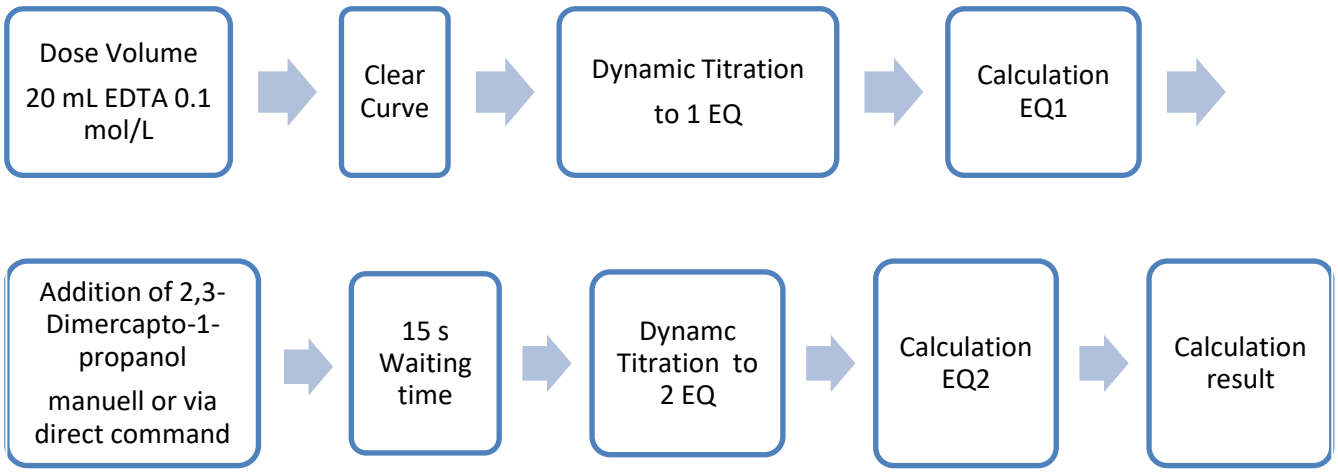
For the dynamic titration this parameters are suitable:

Default method	---		
Method type	Automatic titration		
Modus	Dynamic		
Measured value	mV(E)		
Measuring speed / drift	Individual	Minimum holding time	5 s
		Maximum holding time	10 s
		Measuring time	3 s
		Drift	10 mV/min
Optiline 6 settings		Wave length	625 nm
		Intensity	50
		Smoothing	Normal
Initial waiting time	5 s		
Dynamic	Individual	Max step size	0.5 ml
		Slope max ml	10
		Min. step size	0.05 ml
		Slope min. ml	60
Damping	none	Titration direction	increase
Pretitration	off	Delay time	0 s
End value	off		
EQ	On (1)	Slope value	300
Max. titration volume	50 ml		
Dosing speed	100%	Filling speed	30 s

Sample titration



In Titrisoft, both equivalence points can be displayed in one titration curve. The following method sequence can be used for this:



If the titration curve is too noisy at the beginning, a linear titration with 1 dosing step of 1 ml can be set as a pre-titration before the first dynamic titration.

For both dynamic titrations this parameters are suitable:

Default method	---		
Method type	Automatic titration		
Modus	Dynamic		
Measured value	mV(E)		
Measuring speed / drift	Individual	Minimum holding time	5 s
		Maximum holding time	10 s
		Measuring time	3 s
		Drift	10 mV/min
Optiline 6 settings		Wave length	625 nm
		Intensity	50
		Smoothing	Normal
Initial waiting time	5 s		
Dynamic	Individual	Max step size	0.5 ml
		Slope max ml	10
		Min. step size	0.05 ml
		Slope min. ml	60
Damping	none	Titration direction	increase
Pretitration	off	Delay time	0 s
End value	off		
EQ	On (1)	Slope value	300
Max. titration volume	50 ml		
Dosing speed	100%	Filling speed	30 s

The same parameters can be used for titration without Titrisoft. However, a method must be created for each of the two titration loops.

Calculation:

$$Zn\ [g/L] = \frac{(EQ2 - EQ1) * T * M * F1}{V * F2}$$

EQ1		Consumption of titrant at first Equivalence point
EQ2		Consumption of titrant at second Equivalence point
T	WA	Actual concentration of the titrant
M	65,38	Molecular weight Zn
V	man	sample volume [mL]
F1	1	Conversion factor
F2	1	Conversion factor

$$Ni\ [g/L] = \frac{\big((B - EQ1) - (EQ2 - EQ1)\big) * T * M * F1}{V * F2}$$

B	M01	Blank value from global memory
EQ1		Consumption of titrant at first Equivalence point
EQ2		Consumption of titrant at second Equivalence point
T	WA	Actual concentration of the titrant
M	58,69	Molecular weight Ni
V	man	sample volume [mL]
F1	1	Conversion factor
F2	1	Conversion factor

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