Electroless Nickel Bath: Determination of Nickel

Method for determination of nickel content in electroless nickel bath.

Sample	Electroless nickel bath, 5 mL
Compound	Nickel, Ni M= 58.69 g/mol, z = 1
Chemicals	Deionized water, 50 mL Indicator : 0.2 g Murexide trituration with NaCl (1 : 500). Buffer pH10, 10 mL
Titrant	Ethylenediaminetetraacetic acid disodium, $C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$ c(EDTA- Na_2) = 0.1 mol/L
Standard	Zinc Sulfate, $ZnSO_4$ c($ZnSO_4$) = 0.1 mol/L
Indication	DP5 Phototrode™ (555 nm) (Yellow to blue-violet)
Chemistry	Ni^{2+} + Murexide$^-$ → Ni-Murexide$^+$ Ni-Murexide$^+$ + $EDTA^{4-}$ → Ni-$EDTA^{2-}$ + Murexide$^-$
Calculation	R = Q*C/m*d, g/L Q = Titrant consumption in mmol. C = M/z. M = Molar mass of sample in g. z = Equivalent no. sample, z=1 d = density of sample in g/mL. m = mass of sample in mL.
Waste disposal	Nickel solutions : If necessary, neutralize the solution before final disposal as special waste.
Author, Version	Ruby Das, IMSG AnaChem, V2.0 Revised: C. De Caro, MSGAnaChem

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.

Sample Preparation:

- **Electroless nickel bath** : Pipette 20 mL Ginplate Ni 426-A and 20 mL Ginplate Ni 426-B in 200 mL volumetric flask and dilute it upto the mark with deionized water.

Sample titration:

- Add 0.25 g murexide trituration with NaCl (1:500) in the beaker placed on sample changer.
- Dispense 5 mL sample from dosing unit.
- Add 50 mL of deionized water from dosing unit.
- Add 10 mL of buffer pH10 from dosing unit.
- Titrate with 0.1mol/L EDTA.
- After completion of each sample sensor, stirrer and titration tubes are rinsed by deionized water by means of membrane pump.
- Sensor is cleaned with deion. water in the conditioning beaker placed on sample changer after each sample.

Remarks

- Prior to use , adjust the output signal of the DP5 Phototrode™ to approx. 1000 mV in deion.water before starting titration (100% transmission) by turning the small knob on the housing.
- Rinsing and conditioning of the Phototrode is crucial to achieve accurate and precise results.
- Avoid formation of bubbles during titration by low speed rate of stirrer, as they disturb photometric indication.
- This method allows a fully automated analysis procedure. This method can be easily modified for manual operation. Select “Manual stand” in the method function “Titration stand”.
- Sample may be added manually using a pipette instead of using an additional dosing unit.

Literature :

- Ginplate NI 426, (http://www.growel.com/tds/549.pdf), a trademark of Grauer & Weil India Ltd, www.growel.com
- Mettler-Toledo Application M066 and M007

Instruments	- Titration Excellence T50/T70/T90 (Other Titrators: depending on instrument type, manual operation and method changes are necessary) - XP205 Balance (MT-1106024) - Rondo 20 with PowerShower™ (MT-51108003)
Accessories	- 3 x Additional dosing unit (MT-51109030) - 1 x 20 mL DV1020 glass burette (MT-51107502) - 2 x 10 mL DV1010 glass burette (MT-51107501) - 1 x 5 mL DV 1005 glass burette (MT-51107500) - 100 mL Propylene titration beakers (MT-00101974)

Results

All results

Method-ID	Nickel determination
Sample	5 mL (1/6)
R1 (Nickel)	5.44 g/L
Sample	5 mL (2/6)
R2 (Nickel)	5.44 g/L
Sample	5 mL (3/6)
R3 (Nickel)	5.45 g/L
Sample	5 mL (4/6)
R4 (Nickel)	5.43 g/L
Sample	5 mL (5/6)
R5 (Nickel)	5.43 g/L
Sample	5 mL (6/6)
R6 (Nickel)	5.44 g/L

Statistics

Method-ID	Nickel determination
R1	Nickel
Samples	6
Mean	5.44
s	0.01
srel	0.138%

Titration curve

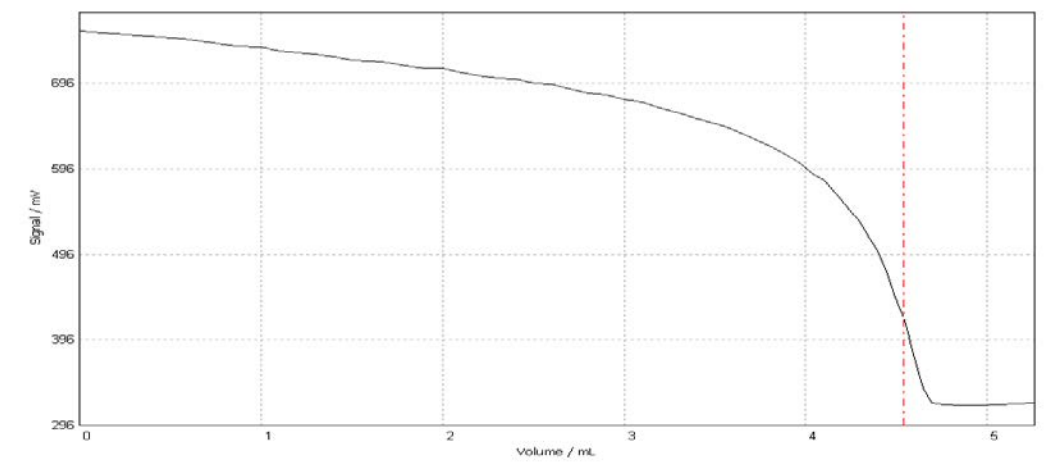

Table of measured values

	Volume mL	Increment mL	Signal mV	Change mV	1st deriv. mV/mL	Time s	Temperature °C
	0.000	NaN	757.2	NaN	NaN	0	25.0
	0.5715	0.5715	748.2	-9.0	NaN	5	25.0
	0.8570	0.2855	739.0	-9.2	NaN	13	25.0
	1.0000	0.1430	738.4	-0.6	NaN	16	25.0
	1.1000	0.1000	734.0	-4.4	NaN	20	25.0
	1.2000	0.1000	732.1	-1.9	-25.96	24	25.0
	1.3000	0.1000	729.6	-2.5	-24.64	28	25.0
	1.4000	0.1000	727.1	-2.5	-26.16	32	25.0
	1.5000	0.1000	723.3	-3.8	-24.72	37	25.0
	1.6000	0.1000	721.7	-1.6	-24.40	42	25.0
	1.7000	0.1000	720.6	-1.1	-23.28	44	25.0
	1.8000	0.1000	716.7	-3.9	-24.36	50	25.0
	1.9000	0.1000	713.5	-3.2	-28.28	55	25.0
	2.0000	0.1000	713.0	-0.5	-29.78	58	25.0
	2.1000	0.1000	708.5	-4.5	-30.43	62	25.0
	---	---	---	---	---	---	---
	4.3440	0.0500	518.0	-17.4	-358.17	184	25.0
	4.3940	0.0500	499.4	-18.6	-421.44	190	25.0
	4.4440	0.0500	476.6	-22.8	-538.07	196	25.0
	4.4940	0.0500	446.3	-30.3	-658.13	202	25.0
EQP1	4.540268	NaN	420.7	NaN	-659.56	NaN	NaN
	4.5440	0.0500	418.6	-27.7	-657.52	210	25.0
	4.5940	0.0500	379.1	-39.5	-544.67	218	25.0
	4.6440	0.0500	339.4	-39.7	-393.50	230	25.0
	4.6940	0.0500	322.0	-17.4	-269.40	239	25.0
	4.7600	0.6600	320.1	-1.9	-148.20	243	25.0
	4.8600	0.1000	319.3	-0.8	NaN	245	25.0
	4.9600	0.1000	319.6	0.3	NaN	248	25.0
	5.0600	0.1000	320.4	0.8	NaN	250	25.0
	5.1600	0.1000	321.2	0.8	NaN	253	25.0
	5.2600	0.1000	321.9	0.7	NaN	255	25.0

Comments

- Titer determination of 0.1 mol/L EDTA-Na_2 is done as per the Mettler –Toledo method application M007 and mean value found is 0.99163.
- The mean value of the titer is automatically stored as part of the setup by the function TITER.
- The buffer pH 10 is prepared by dissolving 64 g NH_4Cl in 600mL 25% Ammonia solution and diluting it upto the mark with deionized water in a 1L volumetric flask..
- The shape of the titration curve is somewhat affected by the concentration of the indicator. The results, however do not differ significantly (tested range: 25-500 mg of Murexide trituration with NaCl(1 :500).
- Add the indicator before starting analysis. Keep sample free of air bubbles during titration. Air bubbles and undissolved impurities affect the photometric indication.
- Due to the steep signal change, an EQP titration with fixed increments is used. The low threshold value allows for different amounts of indicator.

Principle :

- Nickel ions forms yellow complex with murexide in alkaline solution:

  Ni^{2+} + Murexide^{-} → Ni-Murexide^{+}

- By adding EDTA , Ni forms a more stable complex with EDTA:

  Ni-Murexide^{+} + $EDTA^{4-}$ → Ni-$EDTA^{2-}$ + Murexide^{-}

At the equivalence point, all Ni ions have been complexed by EDTA and murexide is free in the alkaline solution. There is a colour change from yellow to blue-violet.

Method

001 Title

Type	General titration
Compatible with	T50 / T70 / T90
ID	Nickeldetermination
Title	Ni determination
. . .	

002 Sample

Number of IDs	1
ID 1	Nickel
Entry type	Fixed volume
Volume	5.0 mL
Density	1.03 g/mL
Correction factor	1.0
Temperature	25.0°C
Entry	Arbitrary

003 Titration stand (Rondo/TowerA)

Type	Rondo/TowerA
Titration stand	Rondo60/1A
Lid handling	No

004 Dispense (normal) [1]

Titrant	NI SAMPLE
Concentration	1
Volume	5.0 mL
Dosing rate	60.0 mL/min
Condition	No

005 Dispense (normal) [2]

Titrant	Water..
Concentration	1
Volume	50.0 mL
Dosing rate	60.0 mL/min
Condition	No

006 Dispense (normal) [3]

Titrant	BUFFER 10PH
Concentration	1
Volume	10.0 mL
Dosing rate	60.0 mL/min
Condition	No

007 Stir

Speed	10%
Duration	60 s
Condition	No

008 Titration (EQP) [1]

Titrant

Titrant	EDTA(0.1M)
Concentration	0.1 mol/L

Sensor

Type	Phototrode
Sensor	DP5
Unit	mV

Temperature acquisition

Temperature measurement	No

Stir

Speed	10%

Predispense

Mode	Volume
Volume	1.0
Wait time	0 s

Control

Control	User
Titrant addition	Dynamic
dE (set value)	10 mV
dV (min)	0.05 mL
dV (max)	0.1 mL
Mode	Equilibrium controlled
dE	1.0 mV
dt	2 s
t (min)	2 s
t (max)	12 s

Evaluation and recognition

Procedure	Standard
Threshold	200.0 mV/mL
Tendency	None
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

Accompanying stating

Accompanying stating	No

Condition

Condition	No

009 Calculation R1

Result	Ni content
Result unit	g/L
Formula	R1= Q*C/m*d
Constant	C=M/z
M	M[Nickel]
z	z[Nickel]
Decimal places	2
Result limits	No
Record statistics	Yes
Extra statistical func.	No
Send to buffer	No
Condition	No

010 Rinse

Auxillary reagent	WATER
Rinse cycles	1
Vol.per cycle	10 mL
Position	Current position
Drain	No
Condition	No

011 Condition

Type	Fix
Interval	1
Position	Conditioning beaker
Time	20 s
Speed	10 %
Condition	No

012 End of sample