Determination of the Sodium Borohydride – Hypochlorite method

Description

$NaBH_4$ is treated with excess $NaClO_4$. The not reacted NaClO4 is back titrated with $Na_2S_2O_4$ after the addition of KI and the release of iodine.

Instrumentation

Titrator	TL 5000/50-M1, TL 7000 or higher with WA 50
Sensor	Pt 62, Pt 62 RG or Pt 61
Cable	L 1 A
Stirrer	Magnetic stirrer TM 235
Laboratory instruments	100 and 500 ml volumetric flasks with stopper
	250 ml glass beaker tall form
	10 and 25 ml volumetric pipette

Reagents

1	$Na_2S_2O_3$ 0.2 mol/L
2	NaClO 0.18 mol/L
3	NaOH 0.1 mol/L
4	Potassium iodide (KI)
5	H_2SO_4 25 %
6	Deionized water
All reagents should be of analytical grade or better	

Reagents

$Na_2S_2O_3$ 0.2 mol/L, NaOH 0.1 mol/L and H2SO4 25 %

We recommend ready to use titrant and reagents.

NaClO 0.18 mol/l

Example: 180 ml NaClO 3,5 % (Cl2) + about 350 ml deionized H2O + 0.5 ml NaOH 0,1 mol/L (for stabilization) are mixed and then filled up to 500 ml in a 500 ml volumetric flask with deionized water.

Titration procedure

Sample preparation

0.2 – 0.22 g of high concentrated $NaBH_4$ are weighed exactly into a 100 ml volumetric flask. The sample is dissolved with about 70 ml NaOH 0.1 mol/L shaking the volumetric flask carefully. Fill up to 100 ml with NaOH 0.1 mol/L. This sample solution is only stable for about 1 – 2 hours only.

Pipette 10 ml from the sample solution into a 250 ml glass beaker. Add exactly 25 ml of the NaClO solution. Add a magnetic stirrer bar and stir for 1 minute carefully.

To this solution add 4 – 4.2 g KI and 100 ml deionized water. Mix the sample with higher stirring speed until it is completely mixed. Add 10 ml of the $H2SO_4$ 25 % carefully to the sample solution.

The released iodine is back titrated with the $Na_2S_2O_3$ 0.2 mol/L titrant. Add the exact sample weighed from the sample solution (0.20950 g in our example).

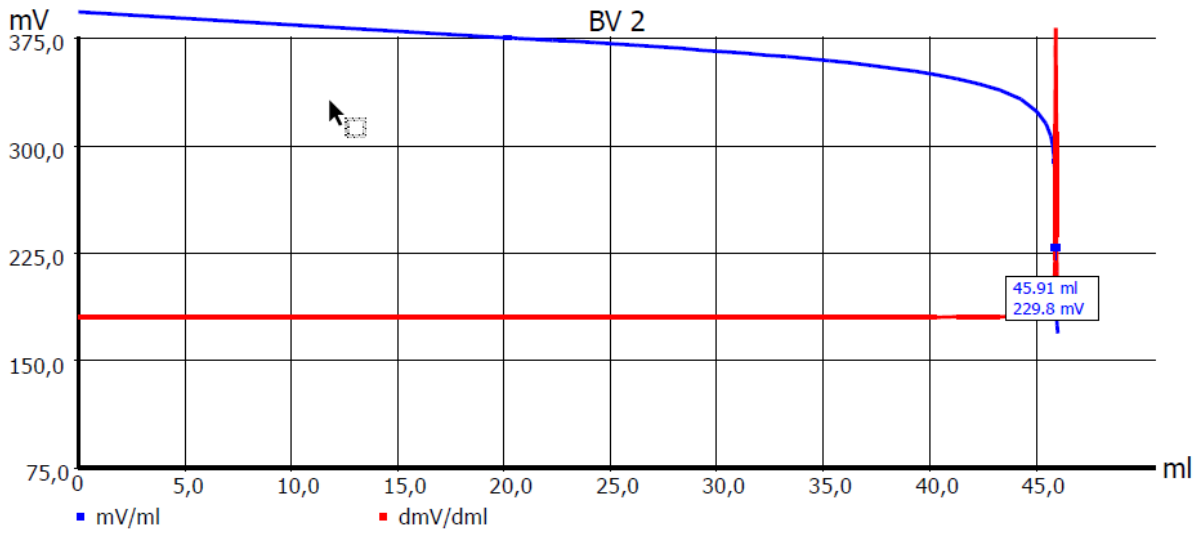
Carry out two blank titrations before with 10 ml NaOH 0.1 mol/L instead of the sample. Use the average value as blank value.

Sensor cleaning

The sensor is cleaned with water.

Titration parameter

Blank



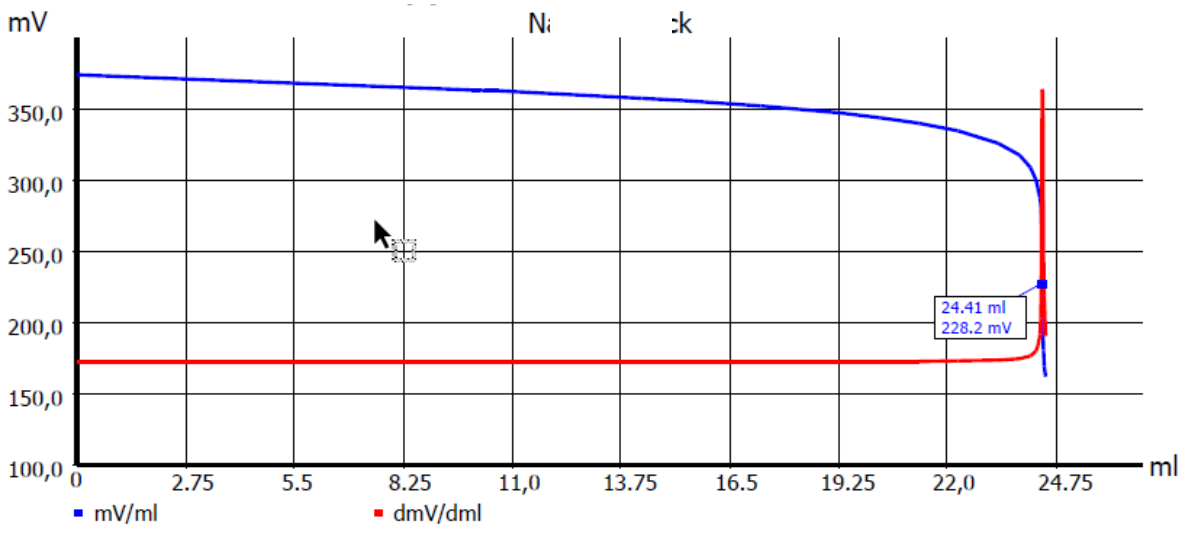
Default method	---		
Method type	Automatic Titration		
Mode	Dynamic		
Measured value	mV		
Measuring speed / Drift	User defined	Minimum holding time	3 s
		Maximum holding time	15 s
		Measuring time	3 s
		Drift	10 mV/min
Initial waiting time	0 s		
Dynamic	average	Max. step size	1.0 ml
		Slope max ml	10
		Min. step size	0.02 ml
		Slope min ml	120
Damping	Off	Titration direction	decrease
Pre-titration	Off	Delay time	0 s
End value	Off		
EQ	On(1)	Slope value	700 (steep)
Max. Titration volume	50 ml		
Dosing Speed	100%	Filling speed	30 s

Calculation:

$$ml = EQ1$$

The result is stored in the Global Memory as for example M01. It is recommendable to determine the mean of two or three blank titrations (statistic: 2 or 3).

Sample titration



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		Slope max ml	10
		Min. step size	0.02 ml
		Slope min ml	120
Damping	Off	Titration direction	decrease
Pre-titration	Off	Delay time	0 s
End value	Off		
EQ	On(1)	Slope value	350
Max. Titration volume	50 ml		
Dosing Speed	100%	Filling speed	30 s

Calculation:

$$NaBH4\ [\%] = \frac{(B - EQ1) * T * M * F1}{V * F2}$$

B	M01	Consumption of titration reagent of the blank determination
EQ1		Consumption of the titration reagent at the EQ
T	WA	Exact concentration of the titration reagent in [mol/L]. 0.2 mol/L
M	4.72880	Equivalent weight including dilution factor
W	man	Sample amount [g]
F1	1	Conversion factor 1
F2	1	Conversion factor 2

Xylem Analytics Germany Sales GmbH & Co. KG,
SI Analytics
Erich-Dombrowski-Straße 4 • 55127 Mainz, Germany

Tel+ 49 6131 894-5111
TechInfo.xags@xylem.com
xylemanalytics.com

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