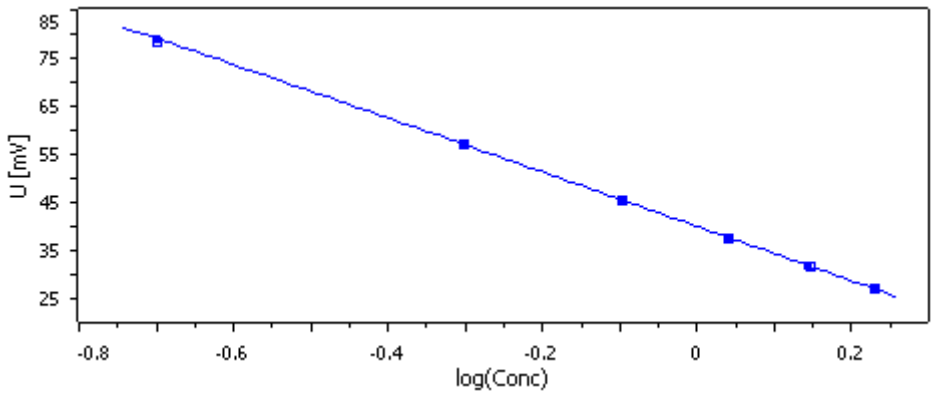
ISE Application Note I–012

Automated calibration of the NH_3-ISE for low ammonia concentrations



For the determination of ammonia with the NH_3-ISE, a correct calibration of the electrode is crucial. This Application Note shows the best way to calibrate the ammonium ISE.

Metrohm

Method description

Sample

Ammonium standard solutions of different concentrations, see *Standard preparation*.

Sample preparation

The standard solutions are automatically prepared by the system, see *Standard preparation*.

Electrode preparation

For the preparation of the electrode, the electrolyte is diluted 1:9 with deion. water. 2.5 mL of the diluted electrolyte is filled into the membrane. After connecting the diaphragm with the shaft, the electrode is shaken like a fever thermometer to assure that no bubbles stick to the inner side of the electrode.

Configuration

815 Robotic USB Sample Processor XL	2.815.0030
836 Titrando	2.836.0020
800 Dosino (3×)	2.800.0010
Robotic arm with holder for titration head, right swinging	6.1462.070
Sample rack 28 × 250 mL	6.2041.820
Dosing unit 2 mL	6.3032.120
Dosing unit 10 mL	6.3032.210
Dosing unit 50 mL	6.3032.250
Disposable PP sample beaker, 200 mL	6.1459.310
NH_3-selective electrode	6.0506.100

Solutions

c(NaOH) = 10 mol/L	400 g NaOH is dissolved in approx. 800 mL deion. water. The solution is cooled down, transferred into a 1 L volumetric flask and filled up to the mark with deion. water.
Ammonium stock standard solution (100 mg/L)	0.297 g NH_4Cl is given into a 1 L volumetric flask and dissolved. After dissolution, the solution is filled up to the mark with deion. water. The stock solution should not be older than 12 h.

Standard preparation

$\beta(NH_3)$ in deionized water [mg/L]

Std. 1	Std. 2	Std. 3	Std. 4	Std. 5	Std. 6
0.2	0.5	0.8	1.1	1.4	1.7

The standards for the calibration are each prepared directly before measurement by dosing appropriate amounts of ammonium stock standard solution (0.10 to 0.85 mL) into a 200 mL PP beaker and subsequent dilution to 50 mL deion. water.

Analysis

Before the measurement 1 mL c(NaOH) = 10 mol/L is added and the calibration is performed. Between each measurement the electrode is dip-rinsed two times in deion. water for 10 s each.

To verify the calibration the recoveries for the different standards is determined. This analysis is performed with the same parameters as the calibration.

Parameters

Mode	CAL MEAS Conc
Measurement type	Measurement with drift control
Signal drift	0.5 mV/min
Min. waiting time	100 s
Max. waiting time	600 s

Method description

Results

Calibration

Slope / mV	Blank / ppm	Variance
-57.2	0.01	0.131

Recovery (n = 5)

Standard	Recovery / %	s(rel) / %
Std. 1	101.50	3.86
Std. 2	99.60	1.70
Std. 3	99.90	2.02
Std. 4	99.56	2.24
Std. 5	99.94	1.92
Std. 6	99.54	2.39

Comments

The use of freshly prepared standards immediately before the measurement ensures the best results for the calibration.