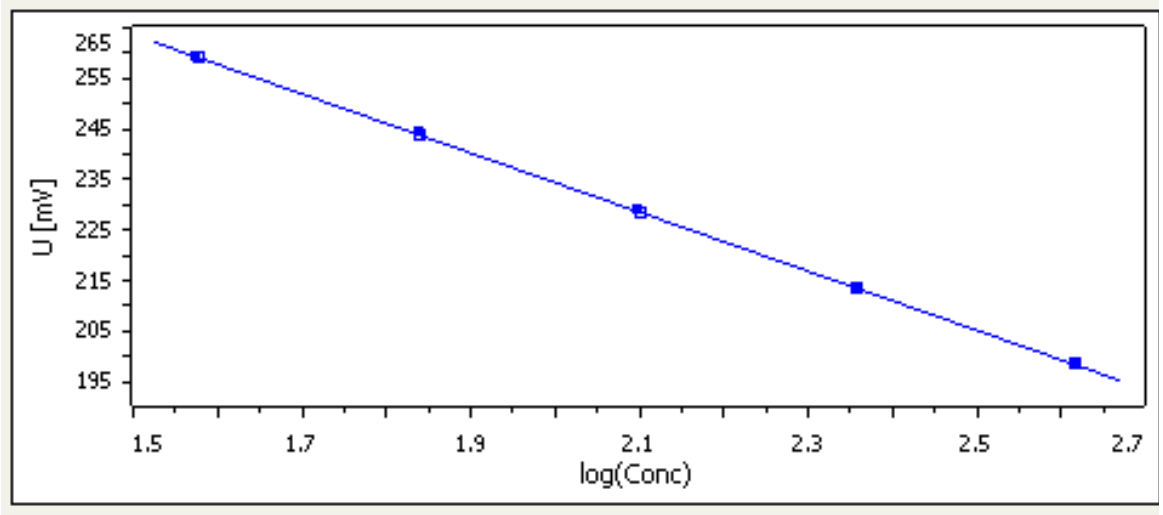
ISE Application Note I-025

Purity of lucigenin by nitrate determination

Fast and inexpensive determination by standard addition



Lucigenin is one of the most often used chemiluminescent reagents and might be used for e.g., the indication of the presence of superoxide anion radicals.

Lucigenin is rather expensive to buy, however, its synthesis only includes a two stage synthesis starting from acridanone. The first stage includes an N-methylation, the second forms the lucigenin chloride, which is finally transformed into lucigenin nitrate. To check the purity of the synthesized lucigenin, ion measurement can be applied using a nitrate selective electrode. This is a fast and inexpensive method compared to competing methods such as ion chromatography.

Metrohm

Method description

Sample

In-house synthesized lucigenin samples

Sample preparation

Approx. 70 mg of dried lucigenin is weighed into a 50 mL volumetric flask, dissolved in approx. 30 mL deionized water and filled to the mark with deionized water.

Configuration

814 USB sample processor (1T/2P)	2.814.0020
Titration head, 3x SGJ 14	6.1458.040
Sample rack 22 x 120 mL	6.2041.470
Sample beakers plastic (PP), 120 mL, 250 pieces	6.1459.300
Propeller for 120 mL beaker	6.1909.050
802 Rod stirrer	2.802.0020
tiamo™ 2.5 full	6.6056.252
867 pH module	2.867.0010
800 Dosino	2.800.0010
Dosing unit, 10 mL	6.3032.210
Cable USB A- mini DIN 8 pin	6.2151.000
Electrode cable 2 m / F	6.2104.030
Electrode cable 2 m, 2 x 2 mm	6.2104.150
Combined NO_3-ISE	6.00510.120
Temperature sensor Pt1000	6.1110.100

Solutions

ISA	$c(Al_2(SO_4)_3)$ = 0.1 mol/L 66.6 g aluminum sulfate octadeca-hydrate is weighed into a 1 L volumetric flask, dissolved in approx. 500 mL deionized water and filled to the mark with deionized water.

Analysis of samples

2 mL ISA is dosed to 5 mL prepared sample and 43 mL of deionized water is added. The standard addition is carried out using $\beta(NO_3^-)$ = 6200 mg/L

In between each measurement the electrode is conditioned for 30 s in $c(KNO_3)$ = 0.01 mol/L and then well rinsed with deionized water.

Parameters

Mode	STDADD auto
Number of additions	4
Volume auxiliary solution	45 mL
Stop volume	5 mL
Dosing rate	fast
Delta U	15 mV
Signal drift	0.5 mV/min
Min. waiting time	10 s
Max. waiting time	300 s
Measuring interval	2.0 s
Stirring rate	8

Result

Sample	Purity in %
Batch 1	98.7% (s(rel) = 1.3%, n = 5)
Batch 2	99.6% (s(rel) = 1.5%, n = 5)