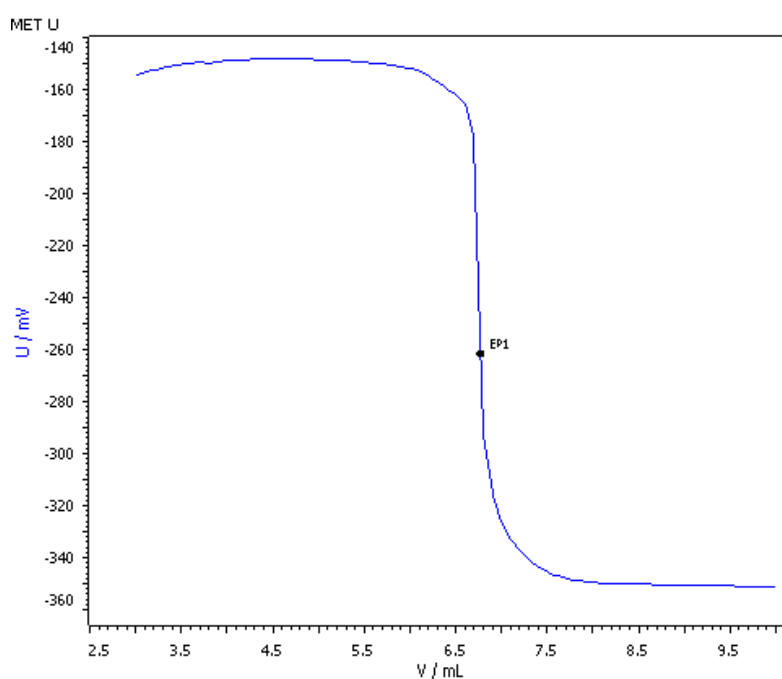


Titration Application Note T-157

Automatic potentiometric titration of sulfanilamide



The purity of sulfanilamide was determined by automatic, potentiometric titration using sodium nitrite as titrant. Potassium bromide was added to the solution because bromide ions act as catalyst for the diazotization titration.

Method description

Sample

Sulfanilamide, purum $\geq 98\%$

Sample preparation

No sample preparation is required.

System preparation

The waterbath rack is filled with deion. H_2O and the thermostat temperature is set to $5\text{ }^\circ\text{C}$. The thermostat is started several hours before the analysis to cool down the water in the waterbath rack.

Configuration

905 Titrande	2.905.0010
815 Robotic USB Sample Processor XL (1T/0P)	2.815.0030
802 Rod stirrer	2.802.0010
843 Pump Station (peristaltic)	2.843.0150
786 Swing Head	2.786.0040
800 Dosino, 4x	2.800.0010
Robotic arm with holder for titration head, left swinging	6.1462.060
Titration Head, 3x SGJ 14	6.1458.040
Replacement tubing for retracted tubing in titration heads and robotic arms	6.1829.050
Drip pan for 789/815/855	6.2711.070
Waterbath rack	6.2840.000
Insert for waterbath 30 * 120 mL	6.9920.116
Sample beaker 120 mL/250 pieces	6.1459.300
Dosing unit 5 mL	6.3032.150
Dosing unit 20 mL, 2	6.3032.220
Dosing unit 50 mL	6.3032.250
Thermostat Lauda E 300	LCK 0863
Pt Titrode	6.0431.100
Pt 1000 temperature sensor for sample changers	6.1110.110

Solutions

Titrand	$c(\text{NaNO}_2) = 0.1\text{ mol/L}$ 6.90 g NaNO_2 is weighed into a 1 L volumetric flask and dissolved in deion. H_2O .
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	The flask is then filled up to the mark with deion. H_2O .
Hydrochloric acid	$w(\text{HCl}) = 20\%$ Approx. 560 g HCl is weighed into a 1 L volumetric flask and dissolved in approx. 200 mL deion. H_2O . The flask is then filled up to the mark with deion. H_2O .
Potassium bromide solution	$c(\text{KBr}) = 2.5\text{ mol/L}$ 29.75 g KBr is weighed into a 100 mL volumetric flask and dissolved in deion. H_2O . The flask is then filled up to the mark with deion. H_2O .

Analysis

90–110 mg sample is weighed into the titration vessels and put onto the rack. 7.5 mL $w(\text{HCl}) = 20\%$ and 42.5 mL deion. H_2O are dosed into the titration vessels before starting any titration. Care has to be taken, that for this pre-dosing the titration equipment does not reach the solution level to prevent any carryover.

The following procedure is separately carried out for each titration vessel: 2 mL $c(\text{KBr}) = 2.5\text{ mol/L}$ is pipetted into a titration vessel. Only after the solution temperature is below $13\text{ }^\circ\text{C}$, titration with $c(\text{NaNO}_2) = 0.1\text{ mol/L}$ is carried out until after the equivalence point.

Parameters

Mode	MET U
Stirring rate	6
Start volume	3 mL
Pause	30 s
Signal drift	30 mV/min
Min. waiting time	5 s
Max. waiting time	60 s
Volume increment	0.1 mL
EP criterion	30 mV
EP recognition	greatest

Results

Content / %	$s(\text{rel}) / \% (n = 5)$
100.94	0.23

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