Titration of Carboxyl end group content according to ASTM D7409-15: Potentiometric Titration

Description

The PET sample is dissolved in an appropriate solvent at 80 °C. The carboxyl end groups are titrated with methanolic KOH after dilution with an additional solvent. According to ASTM D7409-15 two different indications are possible, potentiometric or photometric. The application note describes the potentiometric indication.

Instrumentation

Titrator	TL 7000 oder höher mit WA 10
Electrode	N 6480 eth
Cable	L 1 A
Stirrer	Magnetic stirrer TM 235
Laboratory accessory	Beaker 100 ml
	Watch glasses
	Magnetic stir bar
	Heating plate

Reagents

1	KOH in methanol 0.005 mol/L
2	Ortho-Cresol
3	Dichloromethan
All reagents should be of analytical grade or better.	

Titration procedure

Rinsing of the electrode

After each titration the electrode should be rinsed in a solvent mixture of 20 ml o-Cresol plus 40 ml Dichloromethan for approx. 5 minutes. Afterwards both the electrodes and titration tip are rinsed with Ethanol. Rinse the glas membrane additionaly with water followed by ethanol. Let the sensor dry for approx. 2 minutes before starting the next titration.

Sample preparation

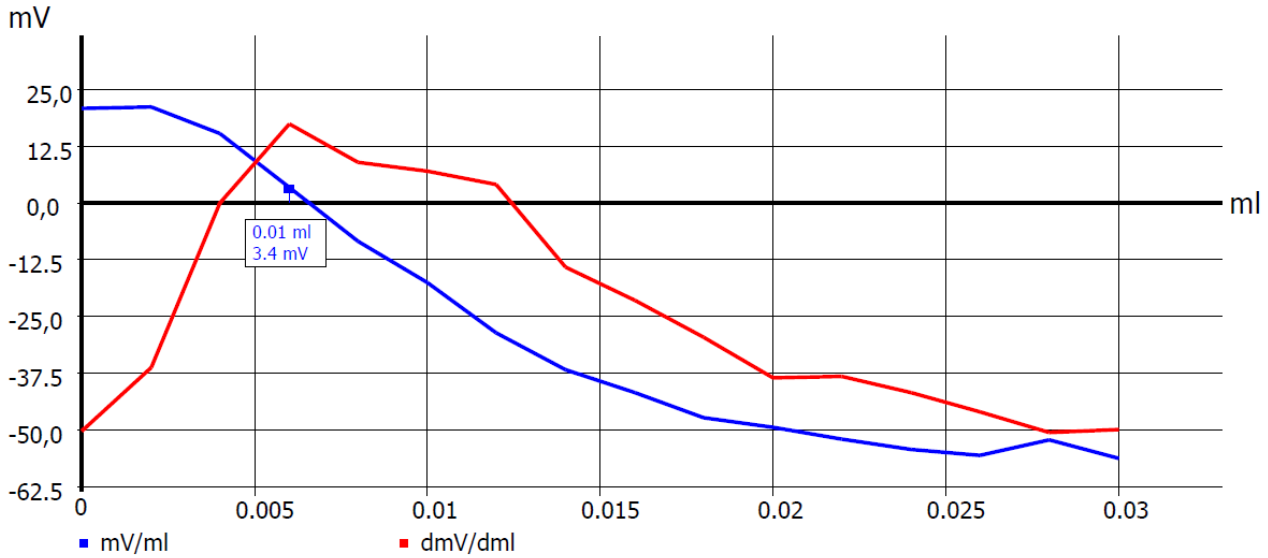
0.25 g of the sample are weight in into a 100 ml beaker. 15 ml of o-Cresol is added. Cover the beaker with a watch glass and heat the sample to 80 °C while stirring. Keep the temperature until the entire sample is dissolved.

After dissolving 60 ml of Dichloromethan is added and titrated using 0.005 molar methanolic KOH.

A blank value is measured as described but without adding the sample. Eine Blindwertbestimmung wird durchgeführt. Die Durchführung erfolgt wie beschrieben, jedoch ohne Zugabe der Probe.

Titration parameter

Blank



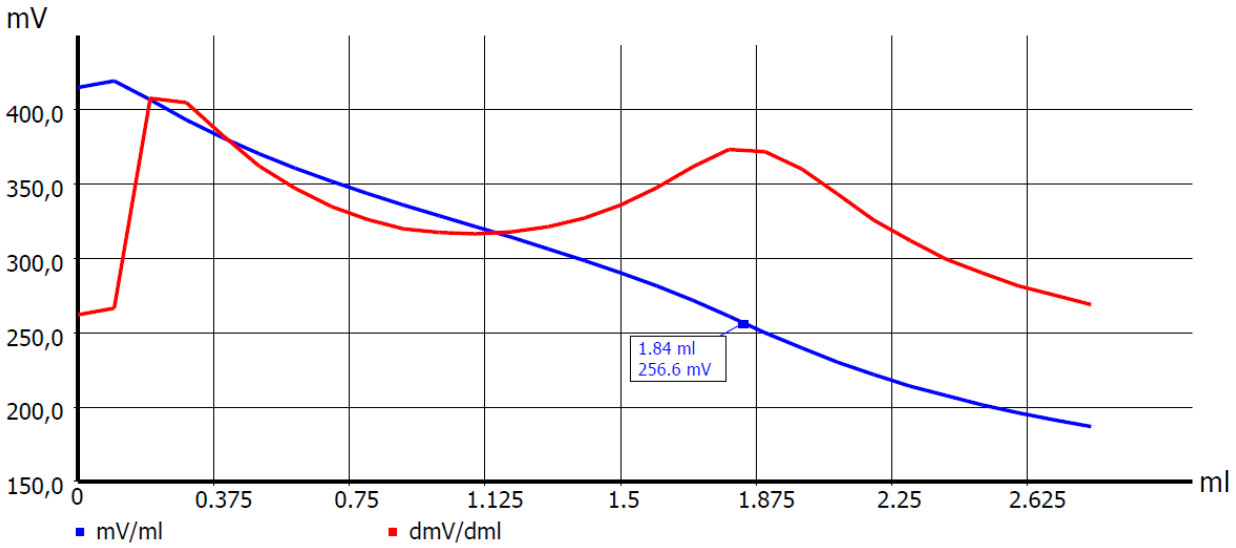
Default method	---		
Method type	Automatic titration		
Modus	linear		
Measured value	mV		
Measuring speed / drift	User defined	Fixed delay time	35 s
Initial waiting time	0 s		
Linear Steps	0.002 ml		
Damping	strong	Titration direction	decrease
Pretitration	off	Delay time	0 s
End value	off		
EQ	off	Slope value	---
Max. titration volume	0.1 ml		
Dosing speed	100%	Filling speed	30 s

Calculation:

$$ml = EQ1$$

The result is stored as a Global memory (for example M01). A three-fold determination is recommendable.

Sample titration



Default method	---		
Method type	Automatic titration		
Modus	linear		
Measured value	mV		
Measuring speed / drift	User defined	Fixed delay time	35 s
Initial waiting time	0 s		
Linear Steps	0.05 – 0.1 ml		
Damping	strong	Titration direction	decrease
Pretitration	off	Initial waiting time	0 s
End value	off		
EQ	off	Slope value	---
Max. titration volume	3 ml (Depending on sample)		
Dosing speed	100%	Filling speed	30 s

Calculation:

$$COOH - R\ [mmol/kg] = \frac{(EQ1 - B) * T * M * F1}{W * F2}$$

B	M01	Consumption of the titration reagent used by the blank determination
EQ1		Consumption of the titration reagent at the first EQ
T	WA	Exact Concentration of the titration reagent [mol/l]
M	1	Molecular Weight (according to the unit mmol/kg here 1)
W	man	Sample amount [g]
F1	1000	Calculation factor 1
F2	1	Calculation factor 2

Fragen? Bitte kontaktieren Sie unser Applikationsteam:

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