

Content Determination of Sodium Starch Glycolate

Method for the acidimetric content determination of sodium in the sodium salt of starch glycolate in a non-aqueous medium

Sample	Sodium Starch Glycolate	Preparation and Procedures Dry and crush the resulting residue from the determination of sodium chloride in sodium starch glycolate. Weigh 0.49 to 0.51 g of the crushed residue, accurately to 0.1 mg, in a round bottomed flask. Add 80 mL acetic acid anhydrous to the flask as well as to an additional 3 empty flasks for the blank determinations, and reflux for 2 hours. Transfer the contents of the round bottomed flasks into a titration beaker, add 20 mL anhydrous acetic acid and titrate.
Substance	Sodium MG= 22.99 g/mol	
Chemicals	Acetic acid, anhydrous R	
Titrant	$c(\text{HClO}_4) = 0.1 \text{ mol/L}$	
Standard	THAM Tris-hydroxymethyl	
Instruments	DL70ES/DL77 and DL58/55+/53+	
Accessories	Titration beaker ME-101974 SP250 Peristaltic pump Temperature sensor DT120	Remarks As a result of the high expansion coefficient of the perchloric acid solution, its temperature is measured during standardisation (H16) and titration (T) and a correction factor (H5) is then calculated.
Indication	DG 113-SC	
Chemistry		
Calculation	$R(\%) = (Q[2] - x) \cdot H5 \cdot M/10 \cdot m$ Q[2] = Titrant consumption in mmol x = Mean value of the blank determination in mmol M = Molar mass Sodium m = Sample size in g H5 = Volume correction factor	
Waste disposal	Halogen-free organic solvents waste.	
Author	L. Pludra	

Results

METTLER DL77 Titration V3.1 Dr. Kade Pharm. Fabrik GmbH
Tante Elfriede Quality Control Laboratory

Method: G032 Na Cont. in Primojel Date: 10-06-1999 8:47
User: Kuntze/Rechenb. Measured: 10-06-1999 11:48

RESULTS

No.	ID1	Sample size and results			
1/1	992512	0.4904 g	R2 = 2.948	%	Na
1/2	992512	0.5018 g	R2 = 2.960	%	Na
1/3	992512	0.5027 g	R2 = 2.955	%	Na
			R3 = 22.7	oC	T Titer det.
			R4 = 24.0	oC	T measured

STATISTICS

Number of results R2	n = 3		
Mean value	= 2.954	%	Na
Standard deviation	s = 0.005867	%	Na
Rel. standard deviation	srel = 0.199	%	

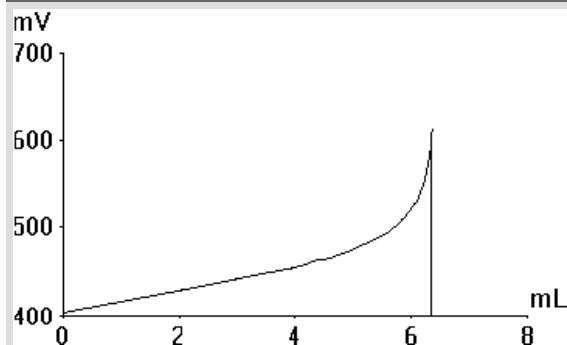
AUXILIARY VALUE

H5 = 0.998588 Volume corr. factor

Table of measured values

EQP	ET	Volumen mL	Signal mV	1. Ableit. mV/mL	Zeit min:s
EQP1	ET1	0.0000	403.7		0:30
		2.2850	431.2	12.0	0:41
		3.4270	446.9	13.7	0:49
	ET2	4.0000	454.3	12.9	0:55
		4.2000	458.1	18.9	1:04
		4.4000	463.3	26.2	1:15
		4.6000	466.7	16.8	1:20
		4.8000	471.0	21.4	1:24
		5.0000	475.1	20.7	1:28
		5.2000	481.3	31.1	1:33
		5.3920	487.3	31.0	1:40
		5.5920	494.5	36.4	1:45
		5.7830	505.2	55.7	1:54
		5.8890	511.1	56.1	1:58
		6.0300	523.4	86.9	2:05
		6.0980	530.4	104.0	2:13
		6.1640	540.5	152.7	2:20
		6.2030	547.9	190.3	2:24
		6.2380	556.0	230.0	2:29
		6.2670	565.0	309.0	2:34
		6.2870	572.8	392.0	2:38
		6.3070	582.2	472.5	2:43
		6.3270	594.2	598.5	2:49
		6.3470	603.3	455.0	2:53
		6.3720	614.7	456.4	2:58

Titration curve



Method

Methode: G032 Gehalt an Na in Primojel
Version: 06-10-1999 8:47

Titel

Methoden ID G032
Titel Gehalt an Na in
Primojel
Datum/Zeit 16-12-1999 14:01

Temperatur

Sensor TEMP A
Messeinheit oC
dT [oC,oF,K] 0.02
dt [s] 2.0
t(min) Modus Fix
t(min) [s] 3.0
t(max) [s] 30.0

Probe

Anzahl Proben 2
Titrierstand ST20 1
Eingabeart Fixes Volumen U
Volumen [mL] 80.0
ID1
Molmasse M 100.0
Aquivalentzahl z 1
Temperatursensor Manuell

Ruhren

Drehzahl [%] 50
Zeit [s] 10

Titration

Titriermittel HClO4
Konzentration [mol/L] 0.1
Sensor DG113-SC
Messeinheit mV
Titrationsart EQP
Vordosierung 1 mL
Volumen [mL] 0.0
Titriermittelzugabe DYN
dE(soll) [mV] 8.0
Limiten dV Absolut
dV(min) [mL] 0.01
dV(max) [mL] 0.1
Messart EQU
dE [mV] 0.5
dt [s] 1.0
t(min) [s] 3.0
t(max) [s] 30.0
Schwelle 50.0
Maximalvolumen [mL] 1.0
Auswerteverfahren Standard
Stopp fur Nachauswertung Ja
Bedingung neq=0

Berechnung

Resultatname Blindwert
Formel R1=Q
Konstante
Resultateinheit mmol
Anzahl Dezimalstellen 3

Protokoll

Ausgabegerat Computer
Rohresultate letzte Probe Ja
Messwerttabelle Ja
E - V Kurve Ja

Statistik

Ri (i=Index) R1
Standardabweichung s Ja
Rel. Standardabweichung srel Ja

Probe

Anzahl Proben 4
Titrierstand ST20 1
Eingabeart Gewicht m
Untere Limite [g] 0.49
Obere Limite [g] 0.51
ID1 Pruef.Nr
Molmasse M 22.99
Aquivalentzahl z 1
Temperatursensor TEMP A

Ruhren

Drehzahl [%] 75
Zeit [s] 15

Titration

Titriermittel HClO4
Konzentration [mol/L] 0.1
Sensor DG113-SC
Messeinheit mV
Titrationsart EQP
Vordosierung 1 mL
Volumen [mL] 4.0
Titriermittelzugabe DYN
dE(soll) [mV] 8.0
Limiten dV Absolut
dV(min) [mL] 0.02
dV(max) [mL] 0.2
Messart EQU
dE [mV] 0.5
dt [s] 1.0
t(min) [s] 3.0
t(max) [s] 30.0
Schwelle 100.0
Maximalvolumen [mL] 13.0
Abbruch nach n EQP's Ja
n = 1
Auswerteverfahren Standard
Nur steilster Sprung Ja
Stopp fur Nachauswertung Ja
Bedingung neq=0

Hilfswert

ID-Text Volumen Korrfaktor
Formel H5=1+(H16-T)*0.0011

Berechnung

Resultatname Na
Formel R2=(Q[2]-x)*H5*C2/m
Konstante C2=M/(10*z)
Resultateinheit %
Anzahl Dezimalstellen 2

Protokoll

Ausgabegerat Computer
Rohresultate letzte Probe Ja
Messwerttabelle Ja
E - V Kurve Ja

Statistik

Ri (i=Index) R2
Standardabweichung s Ja
Rel. Standardabweichung srel Ja

Berechnung

Resultatname T Titerbest.
Formel R3=H16
Konstante
Resultateinheit oC
Anzahl Dezimalstellen 1

Berechnung

Resultatname T gemessen
Formel R4=T
Konstante
Resultateinheit oC
Anzahl Dezimalstellen 1

Protokoll

Ausgabegerat Drucker
Methode (Kurzfassung) Ja
Alle Resultate Ja