

Content Determination of Magnesium in Magnesium Stearate

Method for the complexometric determination of magnesium in Magnesium Stearate

Sample	Magnesium stearate, $C_{36}H_{70}O_4Mg$	Preparation and Procedures Weigh a sample of 0.74 to 0.76 g magnesium stearate, accurately to 0.1 mg, in a 100 mL titration beaker and add 50 mL of a mixture of equal volumes 1-butanol and anhydrous ethanol. Pipette into the sample 5 mL ammonium solution 26%, 3 mL ammonium chloride buffer solution pH10 and 30 mL 0.1M Na_2 -EDTA. Place the titration beaker in an ultrasonic bath at 45 - 50 °C, and leave for approximately five minutes until the sample is completely dissolved. Prepare three blank determination samples in the same way.
Substance	Magnesium $M = 24.31 \text{ g/mol}$	
Chemicals	1-Butanol R Ammonium chloride buffer solution pH 10 R 0.1M Na_2 -EDTA	
Titrant	$c(ZnSO_4) = 0.1 \text{ mol/L}$	
Standard	EDTA-Dihydrate (M539)	
Instruments	DL77/DL70ES and DL58/55+/53+	
Accessories	Titration beaker ME-101974, SP250 Peristaltic pump, Rondo60 Sample changer	
Indication	DM 140-SC (amalgamated)	Remarks The previously determined loss on drying content is saved as the auxiliary value H20.
Chemistry		
Calculation	$R(\%) = (x-Q) \cdot M \cdot F/10 \cdot m$ Q = Titrant consumption in mmol x = Mean value of the blank determination in mmol F = $100/(100-H20)$ Formula for the loss on drying of Magnesium Stearate M = Molar mass Magnesium m = Sample size in g	
Waste disposal	Halogen-free organic solvents waste, zinc containing.	
Author	L. Pludra	

Results

METTLER DL70ES Titrator V3.1 Dr. Kade Pharm. Fabrik GmbH
Onkel Tuka Quality Control Laboratory

Method: G061 Cont. Magnesium stearate Date: 29-10-1999 10:02
User: Kuntze/Karasch Measured: 29-10-1999 13:19

RESULTS

No.	ID1	Sample size and results			
1/1	992563	30.0 mL	R1 = 3.003	mmol	Blank
1/2	992563	30.0 mL	R1 = 3.003	mmol	Blank
2/1	992563	0.7426 g	R2 = 4.636	%	Magnesium
2/2	992563	0.7447 g	R2 = 4.639	%	Magnesium
2/3	992563	0.7554 g	R2 = 4.631	%	Magnesium
2/4	992563	0.7522 g	R2 = 4.635	%	Magnesium
2/5	992563	0.7505 g	R2 = 4.630	%	Magnesium
2/6	992563	0.7441 g	R2 = 4.622	%	Magnesium

STATISTICS

Number of results R1 n = 2
Mean value = 3.003 mmol Blank
Standard deviation s = 0.000080 mmol Blank
Rel. standard deviation srel = 0.003 %

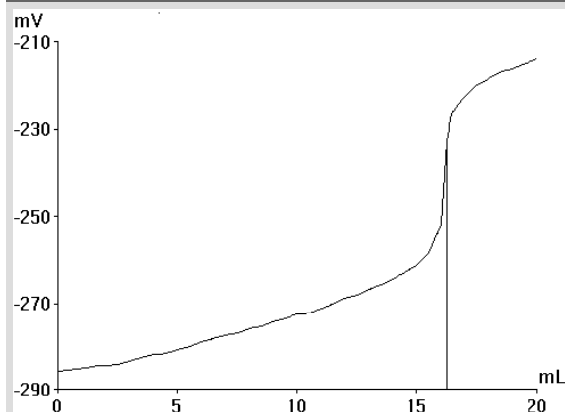
STATISTICS

Number of results R2 n = 6
Mean value = 4.632 % Magnesium
Standard deviation s = 0.006120 % Magnesium
Rel. standard deviation srel = 0.132 %

Table of measured values

EQP	ET	Volumen mL	Signal mV	1. Ableit. mV/mL	Zeit min:s
EQP1	ET1	0.0000	-286.0		0:02
		1.1420	-284.0	1.8	0:07
		1.7130	-282.7	2.3	0:10
	ET2	2.0000	-282.0	2.2	0:13
		2.5000	-281.3	1.5	0:17
		3.0000	-280.6	1.4	0:21
		\\	\\	\\	\\
		14.0000	-264.7	2.9	2:20
		14.5000	-263.1	3.2	2:25
		15.0000	-261.0	4.3	2:29
		15.5000	-258.2	5.6	2:34
		16.0000	-251.9	12.6	2:42
		16.2550	-232.7	75.2	2:56
		16.2910	-230.8	52.5	3:00
		16.3630	-228.1	37.9	3:07
		\\	\\	\\	\\

Titration curve



Method

Methode: G061 Gehalt Magnesiumstearat
Version: 29-10-1999 10:02

Titel

Methoden ID G061
Titel Gehalt
Magnesiumstearat
Datum/Zeit 29-Okt-1999 10:02

Probe

Anzahl Proben 3
Titrierstand ST20 1
Eingabeart Fixes Volumen U
Volumen [mL] 30.0
ID1
Molmasse M 0.0
Äquivalentzahl z 1
Temperatursensor Manuell

Ruhren

Drehzahl [%] 50
Zeit [s] 10

Titration

Titriermittel ZnSO4
Konzentration [mol/L] 0.1
Sensor DMI41-SC
Messeinheit mV
Titrationsart EQP
Vordosierung 1 mL
Volumen [mL] 28.0
Titriermittelzugabe DYN
dE(soll) [mV] 5.0
Limiten dV Absolut
dV(min) [mL] 0.02
dV(max) [mL] 0.5
Messart EQU
dE [mV] 0.25
dt [s] 1.0
t(min) [s] 2.0
t(max) [s] 20.0
Schwelle 40.0
Maximalvolumen [mL] 32.0
Auswerteverfahren Standard
Nur steilster Sprung Ja
Stopp für Nachauswertung Ja
Bedingung neq=0

Berechnung

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

Spulen

Hilfsreagenz Wasser
Volumen [mL] 20.0

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 6
Titrierstand ST20 1
Eingabeart Gewicht m
Untere Limite [g] 0.74
Obere Limite [g] 0.76
ID1
Molmasse M 24.31
Äquivalentzahl z 1
Temperatursensor Manuell

Ruhren

Drehzahl [%] 50
Zeit [s] 10

Titration

Titriermittel ZnSO4
Konzentration [mol/L] 0.1
Sensor DMI41-SC
Messeinheit mV
Titrationsart EQP
Vordosierung 1 mL
Volumen [mL] 2.0
Titriermittelzugabe DYN
dE(soll) [mV] 5.0
Limiten dV Absolut
dV(min) [mL] 0.02
dV(max) [mL] 0.5
Messart EQU
dE [mV] 0.25
dt [s] 1.0
t(min) [s] 2.0
t(max) [s] 20.0
Schwelle 50.0
Maximalvolumen [mL] 20.0
Auswerteverfahren Standard
Nur steilster Sprung Ja
Stopp für Nachauswertung Ja
Bedingung neq=0

Berechnung

Resultatname Magnesium
Formel $R2 = ((x - Q[2]) * C2 / m)$
Konstante $C2 = M * 10 / (z * (100 - H2O))$
Resultateinheit %
Anzahl Dezimalstellen 3

Spulen

Hilfsreagenz Wasser
Volumen [mL] 20.0

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

Protokoll

Ausgabegerat Drucker+Computer
Alle Resultate Ja