

Glycerin content determination according to USP 26

The glycerin content is determined according to USP 26 with acid base titration: Formic acid that is released from glycerin by quantitative reaction with sodium periodate is titrated with sodium hydroxide. Beforehand the suitability of the periodate solution is validated by redox titration using sodium thiosulfate as titrant.

Sample	Approx. 500 mg Glycerin (anhydrous, > 98%)	Preparation and Procedures 1.1) <i>Sodium(meta)periodate solution</i> : 60 g of sodium(meta)periodate are dissolved in a 1 L flask with 12% v/v sulfuric acid. The solution is stored in a brown titration glass bottle. 1.2) <i>Suitability test of the sodium(meta)periodate solution</i> : - 10 mL of sodium periodate solution are pipetted into a 250 mL flask and filled up with water. - 500 mg of glycerin are dissolved in 50 mL water and 50 mL of the diluted periodate solution was added. Wait for 30 min. - Blank: 50 mL of the diluted periodate solution was added to 50 mL water. Wait for 30 min. - 20 mL aliquots of both solutions were acidified with 1 mL of 1.0 mol/L HCl. 10 mL 0.6 mol/L KI-solution are added. Deion. water was added to achieve a volume of 50 mL. The released iodine was titrated with 0.1 mol/L sodium thiosulfate. 2) <i>Acid base titration</i> : 500 mg glycerin are weighed in a titration beaker and 50 mL deion. water were added. For a blank determination 50 mL deionized water were added separately into a titration beaker. The pH was adjusted to 7.8 for both solutions. 50 mL of the sodium periodate solution was pipetted into each beaker and covered with a watch glass. Both solutions were allowed to stand for 30 min at room temperature. 10 mL of a PEG (polyethyleneglycol)/water mixture (1:1, v:v) were added to both solutions and allowed to stand for 20 min.
Substance	Glycerin, M = 92.10, z = 1	
Chemicals	Potassium hydrogen phthalate (KHP) Sodium(meta)periodate (NaIO ₄) Potassium iodide, 0.6 mol/L Sodium thiosulfate c = 0.1 mol/L	
Titrant	Sodium hydroxide (NaOH) c(NaOH) = 0.1 mol/L	
Standard	Potassium hydrogen phthalate, 80 mg	
Instruments	T50, T70 and T90 Excellence Titrators	
Accessories	Titration beakers ME-101974 OKI B4250 Printer (parallel cable ME-51 108 780)	Remarks 1) <i>Suitability test</i> : According to USP26 the ratio between the volume of 0.1 mol/L sodium thiosulfate required for the glycerin containing mixture to that for the blank should be between 0.750 and 0.765. The obtained ratio was 0.750. This ratio was regarded as a proof that the periodate solution is suitable for the analysis of the glycerin content. 2) <i>Acid base titration</i> : The nominal concentration of the glycerin solution was 0.04995 mol/L (0.5061 g in 110 mL). This corresponds to a recovery of 98.90% (see results: mean value of the obtained glycerin concentration). Blank and sample determinations were performed in the same EQP-titration mode and the obtained blank value (mmol) was subtracted from the sample value (mmol) in order to account for the acidity in the solvent not corresponding to the released formic acid. 3) <i>Oxidation of glycerin with periodate</i> : $\text{CH}_2\text{OH}-\text{CHOH}-\text{CH}_2\text{OH} + 2\text{IO}_4^- = \text{HCOOH} + 2\text{HCHO} + 2\text{IO}_3^- + \text{H}_2\text{O}$ One mole of glycerin corresponds therefore to one mole of formic acid. Formic acid (M = 46.025, z = 1) was titrated with sodium hydroxide. Literature: "USP 26 -Official monographs / Glycerin" p. 867
Indication	DM140-SC (Redox titration) DG111-SC (Acid base titration)	
Chemistry	1) Suitability test (Redox): $\text{IO}_4^- + 7\text{I}^- + 6\text{H}^+ + \text{H}_2\text{O} = 4\text{I}_2 + 2\text{OH}^- + 3\text{H}_2\text{O}$ $4\text{I}_2 + 8\text{S}_2\text{O}_3^{2-} = 4\text{S}_4\text{O}_6^{2-} + 8\text{I}^-$, IO_4^- : z = 8 2) Acid base titration: $\text{HCOOH} + \text{OH}^- = \text{H}_2\text{O} + \text{HCOO}^-$	
Calculation	R1=Q-B[Glycerin]; mmol R2=(Q-B[Glycerin])*C/m C=1/z ; mol/L B[Glycerin]: Blank value	
Waste disposal	Neutralization before final disposal	
Author	Thomas Hitz	

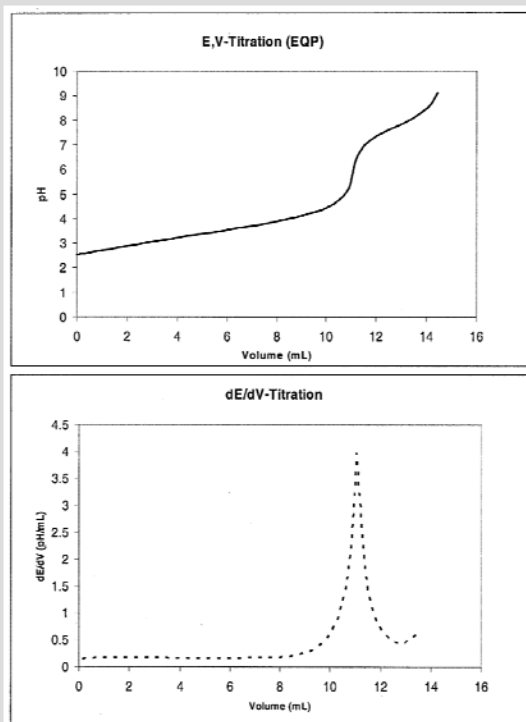
Results

Name: Thomas Hitz, ID			Glycerin Content			
			Rx	Result	Unit	Name
1/5	--	27.03.2007	11:30:27			
			R1 =	0.98657	mmol	Content
			R2 =	0.04933	mol/L	Concentration
2/5	--	27.03.2007	11:34:16			
			R1 =	0.98932	mmol	Content
			R2 =	0.04947	mol/L	Concentration
3/5	--	27.03.2007	11:41:09			
			R1 =	0.98976	mmol	Content
			R2 =	0.04949	mol/L	Concentration
4/5	--	27.03.2007	11:47:34			
			R1 =	0.98991	mmol	Content
			R2 =	0.04950	mol/L	Concentration
5/5	--	27.03.2007	11:53:14			
			R1 =	0.98406	mmol	Content
			R2 =	0.04920	mol/L	Concentration
Statistics						
Rx	Name	n	Mean	Unit	s	srel [%]
R1	Cont.	5	0.98792	mmol	0.00255	0.258
R2	Conc.	5	0.04940	mol/L	0.00013	0.264

Table of measured values

Volume mL	Increment mL	Signal pH	Change pH	1st deriv. pH/mL	Time s
0	NaN	2.544	NaN	NaN	0
0.005	0.005	2.545	0.001	NaN	3
0.01	0.005	2.545	0	NaN	6
0.022	0.012	2.547	0.002	NaN	9
0.054	0.032	2.552	0.005	NaN	12
0.131	0.077	2.563	0.011	0.15	15
0.325	0.194	2.593	0.03	0.16	18
0.51	0.485	2.671	0.078	0.17	21
1.31	0.5	2.762	0.091	0.17	24
1.81	0.5	2.846	0.086	0.18	27
2.31	0.5	2.939	0.091	0.18	30
2.81	0.5	3.024	0.085	0.17	34
3.31	0.5	3.109	0.085	0.17	36
3.81	0.5	3.193	0.084	0.16	40
4.31	0.5	3.274	0.081	0.16	43
4.81	0.5	3.353	0.079	0.16	46
5.31	0.5	3.431	0.078	0.16	49
5.81	0.5	3.511	0.08	0.16	53
6.31	0.5	3.591	0.08	0.16	56
6.81	0.5	3.673	0.082	0.17	59
7.31	0.5	3.756	0.083	0.17	62
7.81	0.5	3.849	0.093	0.18	66
8.31	0.5	3.946	0.097	0.2	70
8.81	0.5	4.06	0.114	0.24	73
9.31	0.5	4.193	0.133	0.31	77
9.81	0.5	4.363	0.17	0.48	81
10.31	0.5	4.606	0.242	0.88	112
10.66	0.35	4.894	0.270	1.47	116
10.815	0.155	5.093	0.209	2.08	119
10.904	0.089	5.271	0.178	2.71	123
10.972	0.068	5.492	0.191	3.22	127
11.025	0.053	5.676	0.214	3.39	130
11.059	0.034	5.839	0.163	3.99	134
11.060119	NaN	5.844	NaN	3.99	NaN
11.095	0.036	6.014	0.175	3.88	137
11.136	0.041	6.19	0.176	3.37	140
11.19	0.054	6.379	0.189	3.12	143
11.26	0.07	6.57	0.191	2.61	147
11.356	0.096	6.75	0.18	2	150
11.614	0.158	6.955	0.205	1.43	163
11.74	0.226	7.16	0.205	0.99	157
12.058	0.318	7.372	0.212	0.66	161
12.476	0.418	7.588	0.216	0.48	166
12.976	0.5	7.826	0.238	0.43	169
13.437	0.461	8.063	0.237	0.6	173
13.604	0.367	8.301	0.238	NaN	178
14.053	0.249	8.515	0.214	NaN	182
14.229	0.176	8.73	0.215	NaN	186

Titration curve



Method

Method	M351	Glycerin
Author	METTLER	TOLEDO
001 Title		
Type	General titration	
Compatible	with T50/T70/T90	
Method ID	M351	
Title	Glycerin content	
Author	METTLER TOLEDO	
Date/Time	26.03.2007/15:00:00	
Modified	--	
Modified by	--	
Protect	No	
SOP	None	
002 Sample		
Number of IDs	1	
ID 1	--	
Entry type	Fixed Volume	
Volume [mL]	20	
Density [g/mL]	1.0	
Correction factor	1.0	
Temperature[°C]	25.0	
003 Titration stand		
Type	Manual stand	
Titration stand	Manual stand1	
004 Stir		
Speed [%]	35	
Duration [s]	60	
005 Titration (EQP) [1]		
Titrant		
Titrant	NaOH	
Concentration[mol/L]	0.1	
Sensor		
Type	pH	
Sensor	DG111-SC	
Unit	pH	
Temperature acquisition		
Temperature acquisition	No	
Stir		
Speed[%]	35	
Predispense		
Mode	None	
Wait time[s]	0	
Control		
Control	User	
Titrant addition	Dynamic	
dE(set value)[mV]	12.0	
dV(min)[mL]	0.002	
dV(max)[mL]	0.5	
Meas. val. acquisition	Equilibrium controlled	
dE[mV]	1.0	
dt[s]	1.0	
t(min)[s]	3.0	
t(max)[s]	30.0	
Evaluation and Recognition		
Procedure	Standard	
Threshold[pH/mL]	3.0	
Tendency	Positive	
Ranges	0	
Add. EQP criteria	No	
Termination		
At Vmax [mL]	10.0	
At potential	Yes	
Potential	9	
Termination tendency	None	
At slope	No	
After number of recognized EQPs	No	
Combined termination criteria	No	
Accompanying stating		
Accompanying stating	No	
Condition		
Condition	No	

006 Calculation R1		
	Result	Content
	Result unit	mmol
	Formula	R1=Q-B[Glycerin]
	Constant	--
	M	--
	z	--
	Decimal places	5
	Result limits	No
	Record statistics	Yes
	Extra statistical functions	No
	Condition	No
007 Calculation R2		
	Result	Concentration
	Result unit	mol/L
	Formula	R2=(Q-B[Glycerin])*C/m
	Constant	C= 1/z
	M	M[FormicAcid]
	z	z[FormicAcid]
	Decimal places	5
	Result limits	No
	Record statistics	Yes
	Extra statistical functions	No
	Condition	No
008 Record		
	Results	Per series
	Raw results	Per series
function	Table of measured values	Last titration
	Sample data	Per series
	Resource data	No
function	E - V	Last titration
function	dE/dV - V	Last titration
	log dE/dV - V	No
	d2E/dV2 - V	No
	E - t	No
	V - t	No
	dV/dt - t	No
	T - t	No
	E - V & dE/dV - V	No
	V - t & dV/dt - t	No
	Calibration curve	No
	Method	No
	Series data	No
009 End of sample		