Metrohm

Application Bulletin 443/1 e

Determination of Glycerin Purity by Potentiometric Titration

Branch

Chemical; Food & beverage

Keywords

Glycerin; raw materials; potentiometric titration; periodate; glycols; Unitrode; 6.0260.020; S01; S010; S07; S071

Summary

This method is applicable to all samples containing glycerin in the absence of other triols or other compounds that react with periodate to produce acidic products. Glycerin may be determined in the presence of glycols.

A periodate solution reacts slowly with diols and triols in acidic aqueous media at room temperature. A quantitative amount of formic acid is generated from the reaction with glycerin (a triol). The reaction with diols produces neutral aldehydes.

$C_3H_8O_3 + 2\ IO_4^- \rightarrow 2\ HCHO + HCOOH + H_2O + 2\ IO_3^-$

The amount of formic acid generated by this reaction is determined by titration against sodium hydroxide.

Samples

Glycerin raw materials

Instruments

- Titrator with DET mode
- 50 mL buret
- Stirrer

Electrode

Unitrode Easy-Clean	6.0260.020

Reagents

- Sodium periodate, puriss p.a.
- Propylene glycol, USP reagent
- Sodium hydroxide, c(NaOH) = 0.1 mol/L
- Sulfuric acid, w(H_2SO_4) = 10% (v/v)
- Potassium hydrogen phthalate
- Ultrapure water

Solutions

Titrant	c(NaOH) = 0.1 mol/L
Periodate solution	30 g of sodium periodate and approx. 300 mL ultrapure water are added to a 500 mL volumetric flask. 4 mL of w(H_2SO_4) = 10% (v/v) is added to this mixture and the flask is filled up to the mark with ultrapure water. The solution must be stored in an opaque container.
Propylene glycol solution	Φ(propylene glycol) = 50% (v/v) in ultrapure water

Titer Determination

0.1 g of potassium hydrogen phthalate is weighed into a 120 mL disposable beaker. Approximately 40 mL of ultrapure water is added. The standard is stirred for two minutes to dissolve it completely. The system is then titrated with c(NaOH) = 0.1 mol/L. The electrode and tips are rinsed with ultrapure water between standards. The titer is determined in triplicate.

Blank

A stir bar is added to a clean 250 mL beaker. 30.0 mL of periodate solution is added, along with approximately 150 mL of ultrapure water. The solution is mixed for 30 seconds, then the beaker is covered and placed in the dark for 30 min. Mixing is resumed, then exactly 10.0 mL of the propylene glycol solution is added. Once mixed, the beaker is covered and placed in the dark for an additional 20 min. After removing the

solution from the dark, it is uncapped and titrated with c(NaOH) = 0.1 mol/L to the last potentiometric endpoint. The electrode and tips are rinsed with ultrapure water between titrations. The blank is saved as a common variable and used in the calculations. The blank should be prepared at the same time as the samples.

Analysis

Aliquots of sample (0.25–0.30 g) are weighed into clean 250 mL beakers. A stir bar is added to each beaker. 30.0 mL of periodate solution is added along with approximately 150 mL of ultrapure water. The solution is mixed for 30 seconds, then the beaker is covered and placed in the dark for 30 min. Mixing is resumed and exactly 10.0 mL of the propylene glycol solution is added. Once mixed, the beaker is covered and placed in the dark for an additional 20 min. The sample is then removed from the dark, uncapped, and titrated with c(NaOH) = 0.1 mol/L to the last potentiometric endpoint. The electrode and tips are rinsed with ultrapure water between titrations. The samples are analyzed in duplicate.

Parameters

Mode	DET U
Signal Drift	25 mV/min
Min. Waiting Time	0 s
Max. Waiting Time	20 s
Measuring Point Density	4
Min. Increment	50 µL
Max. Increment	off
Dosing Rate	20 mL/min
Stirring Rate	6
Stop Volume	50 mL
EP Criterion	5
EP recognition	last

Calculations

Titer

$$f = \frac{m_s}{V_{EP1} \times c_{NaOH} \times M_{KHP}}$$

f: Titer of titrant
m_s: Sample size in mg
V_{EP1}: Titrant consumption until the first equivalence point in mL
c_{NaOH}: Concentration of titrant, c(NaOH) = 0.1 mol/L
M_{KHP}: Molecular weight of potassium hydrogen phthalate, M(KHP) = 204.22 g/mol

Blank

Blank = V_{EP1}

Blank: Blank value in mL
V_{EP1}: Titrant consumption until the last equivalence point in mL

Glycerin content

$$w_{Glycerin} = \frac{(V_{EP1} - Blank) \times c_{NaOH} \times f \times M_{Glycerin}}{m_s \times 10}$$

$w_{Glycerin}$: Glycerin content in %
V_{EP1}: Titrant consumption of the last equivalence point in mL
Blank: Blank value in mL
c_{NaOH}: Concentration of titrant, c(NaOH) = 0.1 mol/L
f: Titer of titrant
$M_{Glycerin}$: Molecular weight of glycerin, M(Glycerin) = 92.10 g/mol
m_s: Sample size in g
10: Conversion factor for %

Example determinations

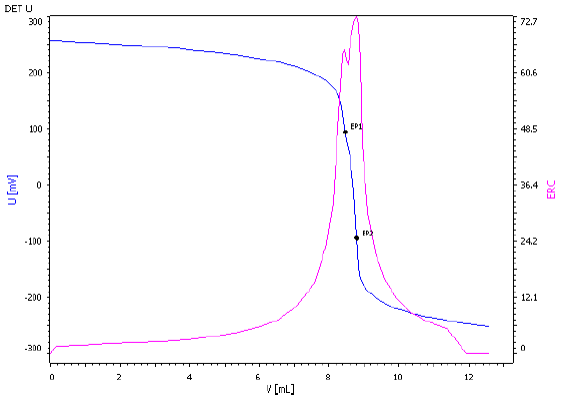

Fig. 1: Determination of a blank. The second endpoint that occurs in the alkaline region is the correct endpoint for choosing the blank volume.

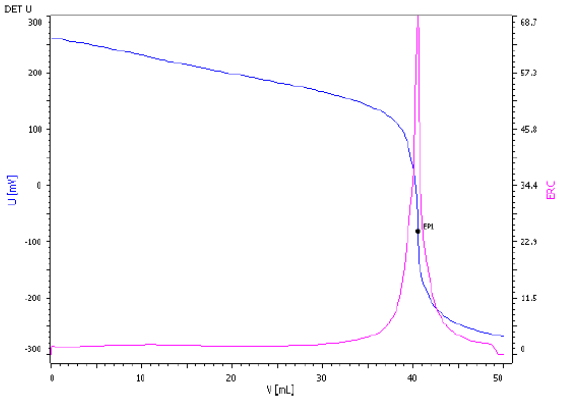

Fig. 2: Determination of a sample with glycerol.

Comments

- This method was adapted for automatic titration from the USP monograph.
- The endpoint recognition for the blank can be set to last to ensure the correct endpoint is automatically picked.

References

- Practical Titration Monograph, Metrohm.
- USP Monograph, Glycerin.

Date

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