

Thermo. Titr. Application Note No. H-060

Title: Standardization of 0.1M perchloric acid in glacial acetic acid

Scope: Standardization of 0.1mol/L perchloric acid in glacial acetic acid by catalyzed endpoint thermometric titration.

Principle: Standardization 0.1mol/L perchloric acid in perchloric acid with standard 0.1mol/L potassium hydrogen phthalate in glacial acetic acid, using hydroxyacetone (acetol) as a thermometric endpoint indicator.

Reagents:

Titrant: 0.1mol/L perchloric acid in glacial acetic acid (BDH Cat. No.190934T .

Endpoint indicator: Hydroxyacetone (acetol) - Aldrich Cat. No. 138185-100G

Solvent: Glacial acetic acid - Ajax Univar

Standard: Potassium hydrogen phthalate – Sigma Ultra (minimum purity 99.95%, Cat. No. P1088-100G)

Method:

Basic Experimental Parameters:

Titrant delivery rate (mL/min.)	2
No. of exothermic endpoints	1
Data smoothing factor (DSF)	60
Stirring speed (802 stirrer)	8

A standard solution of 0.1mol/L potassium hydrogen phthalate was prepared by accurately weighing 1.7030g of the substance into a 200mL volumetric flask, dissolving and making to volume with glacial acetic acid. For highest precision, aliquots between 1 – 5mL were dispensed with a 10mL Dosino burette into a vessel containing 30mL glacial acetic acid. A 1mL dose of acetol was added to the vessel immediately prior to the commencement of the titration.

Example: Titration of 0.10017mol/L potassium hydrogen phthalate solution (KHPht)

	mL KHPht solution	mmole KHPht solution	Titre HClO ₄ solution
	5.00	0.5019	5.035
	4.00	0.4005	4.023
	3.00	0.3017	3.037
	2.00	0.2010	2.031
	1.00	0.1003	1.027

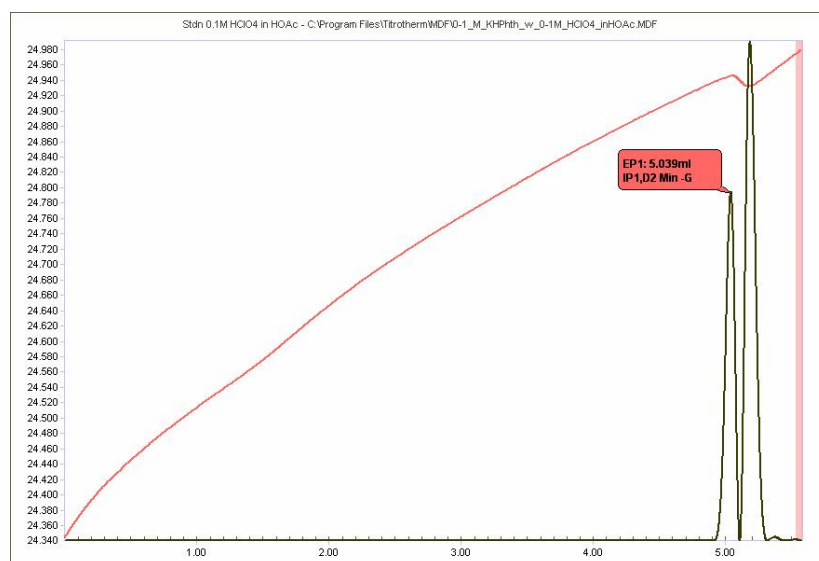
Thermometric Titration Plot:

Legend:

Red = solution

temperature curve

Black = second derivative curve (for endpoints)



Titration of 5mmole potassium hydrogen phthalate

Determination of molarity:

$$\begin{aligned} \text{Molarity} &= 1/\text{gradient} \\ &= 1/9.98293 \\ &= 0.10017 \end{aligned}$$

Titration blank = 0.025mL

