Thermo. Titr. Application Note No. H-003

Title:	**Determination of Sulfate in Phosphoric Acid**

***Scope*:**	Determination of the sulfate content of wet process phosphoric acid.

***Principle*:**	An aliquot of concentrated phosphoric acid is titrated with standard barium chloride solution to a single thermometric endpoint.

Reagent:	Standard c ($BaCl_2$) = 1mol/L

Method:

Basic Experimental Parameters:

Titrant delivery rate (mL/min.)	6
No. of endothermic endpoints	1
Data smoothing factor	40
ERC (2^{nd} derivative) value	-12

Procedure:

Prepare a titration vessel with approximately 30mL deionized water and tare on a balance. Slowly pipette in approximately 10mL of industrial phosphoric acid sample and weigh. Record the mass of the sample. Carefully swirl until thoroughly mixed. If necessary, cool in a water bath to near room temperature. Titrate with standard c($BaCl_2$) = 1 mol/L solution to a single thermometric endpoint.

It is good practice to remove the titration vessel from the thermometric sensor immediately after each titration is finished, rinsing the titration assembly thoroughly with water.

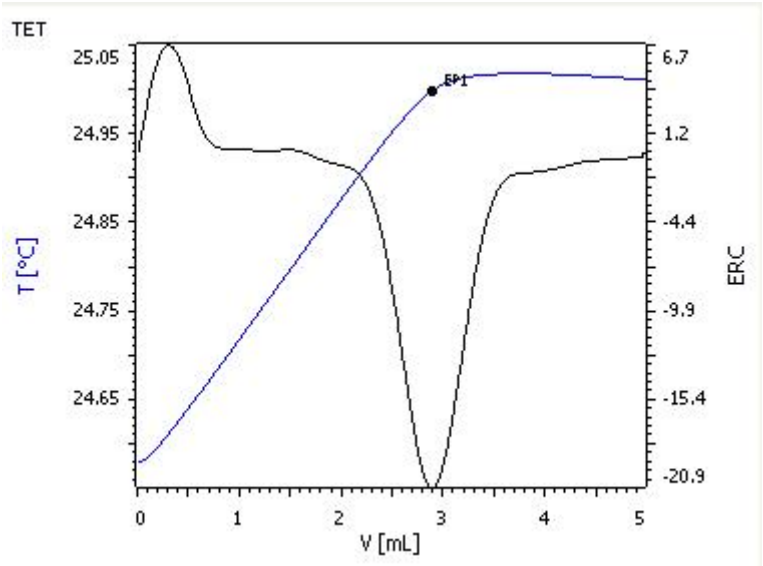
Blank determination. Weigh accurately approximately 2, 4, 6, 8 and 10g of a typical sample of industrial phosphoric acid into titration vessels containing 30mL DI water, and titrate as above. Plot sample mass (x-axis) against endpoint volume (y-axis), and determine the y-intercept by regression analysis. This volume is the systematic error or “blank” value, to be subtracted from the endpoint volume in subsequent determinations. Record the linear correlation coefficient for this determination.

Results (Example): Analysis of sulfate in works phosphoric acid:

	As SO_4^{2-} % w/w	
Mean (n=10):	2.52±0.01	
Blank determination	Blank = 0.1522mL	R^2 = 0.9993

Calculation: $\% SO_4 = \frac{(EP\ mL - blank\ mL) * c(BaCl_2) * 96.0626 * 0.1)}{Sample\ size,\ g}$

Thermometric Titration Plot:



Legend:

Blue = solution temperature curve

Black = second derivative curve