

Thermo. Titr. Application Note No. H-056

Title: Standardization of Barium Acetate Solution

Scope: Standardization of barium acetate titrant used in the determination of sulfate in phosphoric acid. The same procedure is applied if barium chloride is chosen as the titrant.

Principle: The procedure is intended to mimic as far as possible that employed for the determination of sulfate in phosphoric acid (Application Note H-003). This is to reduce any differences due to possible matrix effects.

Reagents: 1 mol/L Ba(OAc)₂ (barium acetate) solution
Saturated boric acid solution
Standard 0.4 mol/L sulfate solution, prepared from anhydrous A.R. sodium sulfate.

Method: Basic Experimental Parameters:
Titrant delivery rate (mL/min.) 6
No. of endothermic endpoints 1
Data smoothing factor 65
Procedure:
Prepare two Dosinos, one containing the 1 mol/L barium acetate solution to be standardized, the other the standard 0.4 mol/L sodium sulfate solution.
Set up a titration program so that:
- Vessel contents are being stirred prior to the introduction of the sodium sulfate solution
- 0.2 mL of the barium acetate titrant is pre-dosed to form BaSO₄ seed prior to the commencement of the actual titration.
- The titration commences 20 seconds after the introduction of the pre-dosed titrant.

Suggested dosing of standard 0.4 mol/L sodium sulfate solution is as follows:

<i>mL 0.4 mol/L Na₂SO₄</i>	<i>mmole SO₄²⁻</i>
10	4
8	3.2
6	2.4
4	1.6
2	0.8

Prepare a series of titration vessels containing 5 mL saturated boric acid solution and a volume of DI water such that the total volume of fluid in the titration vessel after dosing of the standard sulfate solution aliquot comprises approximately 50 mL.

If a second Dosino is unavailable, the standard sodium sulfate solution may be made to 0.2 mol/L and dispensed by volumetric bulb pipettes. In this case, suggested aliquots are as follows:

<i>mL 0.2 mol/L Na₂SO₄</i>	<i>mmole SO₄²⁻</i>
25	5
20	4
15	3
10	2
5	1

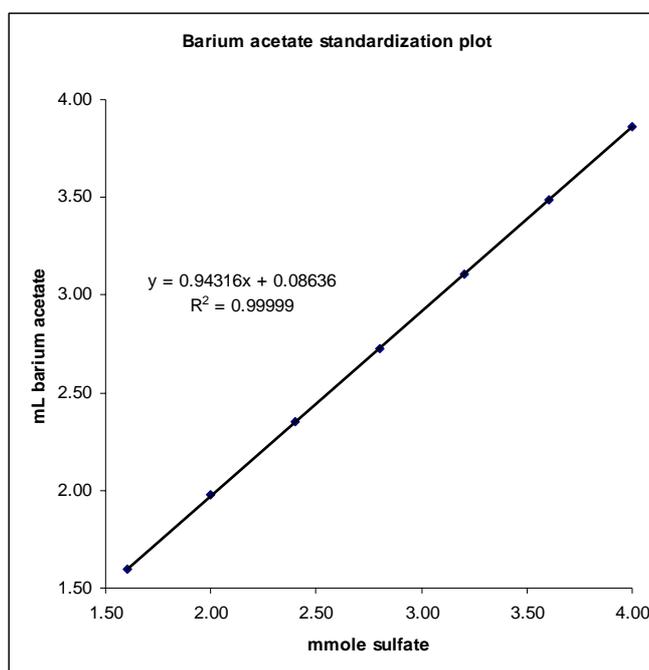
The amount of diluent water in each vessel should be adjusted so that the total volume prior to titration is approximately 50 mL.

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. Accretions of barium sulfate will occur around the diffusion tip, and should be removed by gentle brushing with a soft toothbrush after each titration run. A soda-lime guard tube should be fitted to the Dosino dispensing barium acetate to prevent contamination by CO₂.

After the standard titration set has been completed, plot mL of barium acetate against mmole SO₄²⁻. The reciprocal of the gradient of the linear regression curve represents the strength in mol/L of the barium acetate titrant, while the y-intercept represents the titration blank for this particular set of titration parameters.

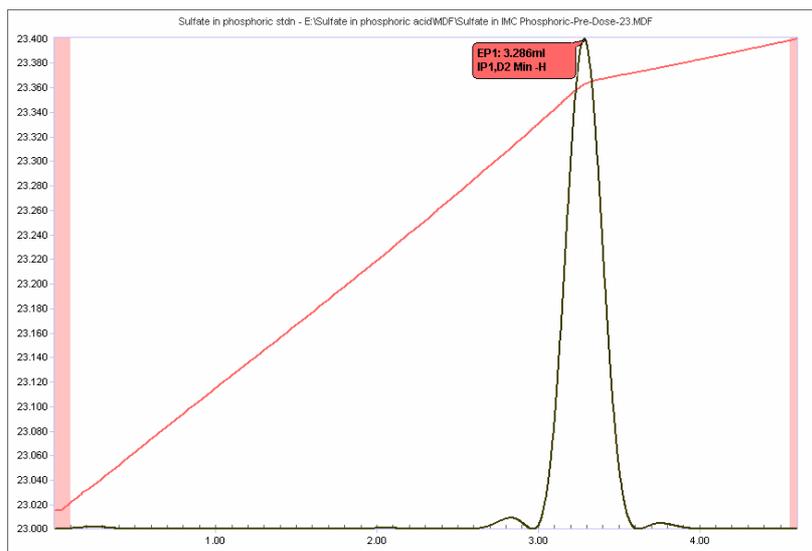
Results (Example):

	mL 0.40025 mol/L Na ₂ SO ₄ soln.	mmole SO ₄ ²⁻	Titre, mL Ba(OAc) ₂
	10	4.0025	3.660
	9	3.60225	3.286
	8	3.2020	2.908
	7	2.80175	2.526
	6	2.4015	2.150
	5	2.00125	1.776
	4	1.6010	1.396

Regression Plot:

Calculations:

1. **molarity** = 1/gradient = 1/0.94316 = 1.0603 mol/L
2. **blank** = y-intercept = 0.0864 mL

Thermometric Titration Plot:



Legend:

Red = solution temperature curve

Black = second derivative curve