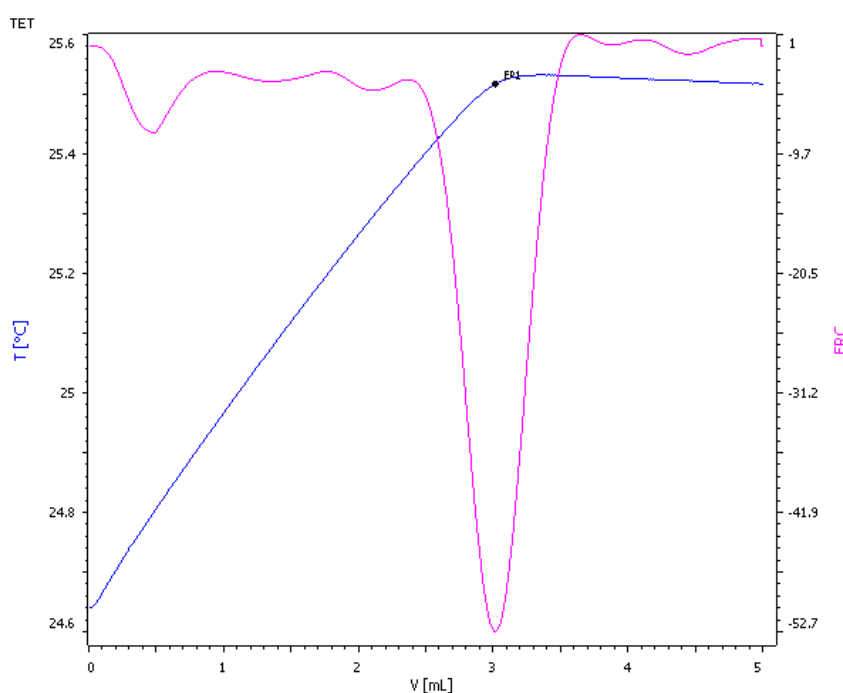


Sulfate in fertilizers

Rapid and reliable determination by thermometric titration



Sulfur is a secondary macronutrient for plants and is essential for chloroplast growth and function. In fertilizers, sulfur is usually provided in the form of sulfate. Traditionally the sulfate content is determined gravimetrically by precipitation with barium. The drawback of this method is that it requires numerous time consuming and laborious analysis steps.

In this Application Note, an alternative method is presented, where sulfate is determined by a precipitation titration with barium chloride. Various solid and liquid NPK fertilizers with sulfur contents between 1 and 8% were analyzed. The analysis of sulfate in fertilizers by thermometric titration requires no sample preparation at all for liquid NPK fertilizers, and only minimal sample preparation for solid NPK fertilizers. One determination takes about 3 minutes only. To increase the sensitivity of the method, the samples are spiked with a standard sulfuric acid solution, which is then considered when calculating the result.

Method description

Sample

Two liquid NPK fertilizers

Four solid NPK fertilizers

Sample preparation

Liquid fertilizers

No sample preparation is required for liquid fertilizers.

Solid fertilizers

Approximately 5 g sample is weighed into a 100 mL Erlenmeyer flask and dissolved in about 75 mL deionized water. The flask is placed into a heated ultrasonic bath at about 40 °C for 30 min. The solution is then filtrated into a 100 mL volumetric flask and the filter paper is rinsed with deionized water. The flask is then filled to the mark with deionized water.

Configuration

859 Titrotherm	2.859.1010
800 Dosino, 2x	2.800.0010
Dosing unit 50 mL	6.3032.250
Dosing unit 20 mL	6.3032.220
Thermoprobe HF	6.9011.040

Solutions

Titration $c(\text{BaCl}_2) = 1 \text{ mol/L}$	246.7 g BaCl_2 is weighed into a 1 L volumetric flask and dissolved in approximately 500 mL deionized water. The flask is then filled to the mark with deionized water.
$c(\text{HCl}) = 0.5 \text{ mol/L}$	164 mL conc. HCl is pipetted into a 1 L volumetric flask and filled to the mark with deionized water.
Standard addition solution $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$	5.55 mL conc. sulfuric acid is pipetted into a 1 L volumetric flask and filled to the mark with $c(\text{HCl}) = 0.5 \text{ mol/L}$.

Analysis

Liquid fertilizers

0.5 to 5 g liquid NPK fertilizer is weighed directly into the titration vessels. Prior to the titration, 25 mL of the sulfuric acid solution is dosed to the sample, which is then made up to a total volume of approximately 30 mL with deionized water. Then the sample is titrated with $c(\text{BaCl}_2) = 1 \text{ mol/L}$ until after the exothermic endpoint.

Solid fertilizers

5 to 20 mL of the prepared solid NPK fertilizer solution is pipetted into the titration vessels. Prior to the titration, 25 mL of the sulfuric acid solution is dosed to the sample, which is then made up to a total volume of approximately 50 mL with deionized water. Then the sample is titrated with $c(\text{BaCl}_2) = 1 \text{ mol/L}$ until after the exothermic endpoint.

Parameters

Mode	TET
Start volume	0 mL
Pause	10 s
Stirrer	15
Dosing rate	2 mL/min
Filter factor	50
Damping until	0.5 mL
Stop volume	5 mL
Evaluation start	0.5 mL
Reaction type	Exothermic
EP criterion	-25

Result

Sample no. (n = 5)	S / %	s(abs) / %
Liquid NPK fertilizer 1	1.91	0.01
Liquid NPK fertilizer 2	0.99	0.02
Solid NPK fertilizer 1	3.63	0.02
Solid NPK fertilizer 2	7.71	0.02
Solid NPK fertilizer 3	6.34	0.03
Solid NPK fertilizer 4	7.35	0.04

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