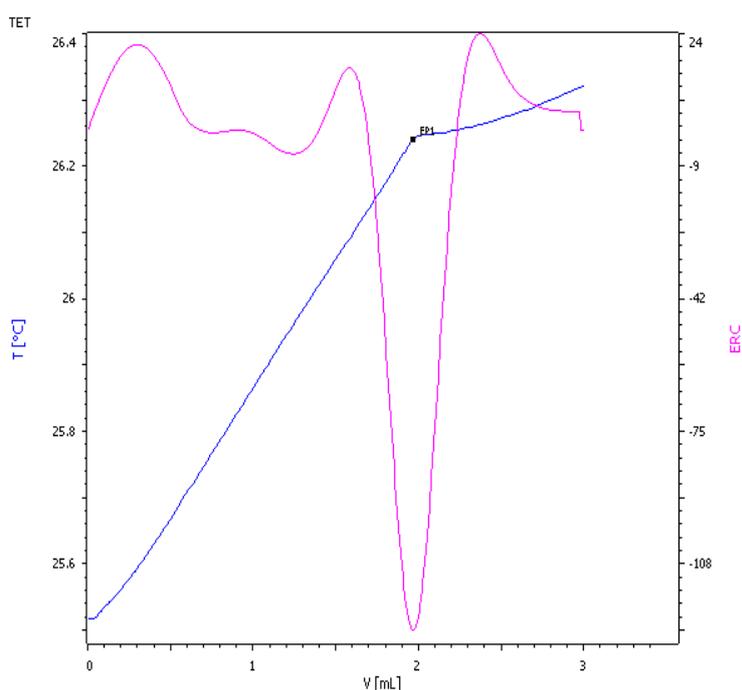


# Ammonium in fertilizers

Rapid and reliable determination by thermometric titration



Nitrogen is a primary macronutrient for plants and is a constituent of amino acids and thus proteins. In fertilizers, nitrogen is provided in the form of nitrate or ammonium, as plants are only able to take up these two nitrogen ions. As amino acids can only be built from ammonium, a distinction between ammonium nitrogen and nitrate nitrogen in fertilizers helps to select the right fertilizer for the plants.

Traditionally the ammoniacal nitrogen is determined Kjeldahl digestion followed by titration. In this Application Note, an alternative method is presented, where the ammoniacal nitrogen is determined by a redox titration with sodium hypochlorite. Various solid and liquid NPK fertilizers with ammoniacal nitrogen contents between 1.5 and 13.5% were analyzed. The analysis by thermometric titration is highly robust, requires no sample preparation at all for liquid NPK fertilizers and only minimal sample preparation for solid NPK fertilizers. One determination takes only about 2 minutes.

# Method description

## Sample

Two liquid NPK fertilizers

Five 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 10 mL	6.3032.210
Dosing unit 50 mL	6.3032.250
Thermoprobe	6.9011.020

## Solutions

Titrant $c(\text{NaOCl})=0.25 \text{ mol/L}$	154.3 mL NaOCl is pipetted into a 1 L volumetric flask and filled to the mark with deionized water.
Auxiliary solution $\text{Br}^-/\text{HCO}_3^-$	200 g KBr and 120 g $\text{KHCO}_3$ are 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.

## Analysis

### Liquid fertilizers

0.25 to 5 g liquid sample is weighed directly into the titration vessels. Prior to the titration, 10 mL of auxiliary solution is dosed to the solution, which is then made up to a total volume of approximately 30 mL with deionized water. Then the sample is titrated with  $c(\text{NaOCl})=0.25 \text{ mol/L}$  until after the exothermic endpoint.

### Solid fertilizers

0.5 to 12.5 mL of the solid sample solution is pipetted into the titration vessels. Prior to the titration, 10 mL of auxiliary solution is dosed to the solution, which is then made up to a total volume of approximately 30 mL with deionized water. Then the sample is titrated with  $c(\text{NaOCl})=0.25 \text{ mol/L}$  until after the exothermic endpoint.

## Parameters

Mode	TET
Start volume	0 mL
Pause	0 s
Stirrer	15
Dosing rate	4 mL/min
Filter factor	60
Damping until	0 mL
Stop volume	10 mL
Evaluation start	0 mL
Reaction type	Exothermic
EP criterion	-15

## Result

Sample no. (n = 5)	Ammonical nitrogen / %	s(abs) / %
Liquid NPK fertilizer 1	1.89	0.010
Liquid NPK fertilizer 2	1.64	0.003
Solid NPK fertilizer 1	9.88	0.084
Solid NPK fertilizer 2	3.76	0.023
Solid NPK fertilizer 3	4.39	0.023
Solid NPK fertilizer 4	13.62	0.040
Solid NPK fertilizer 5	2.72	0.009

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