
Nitrate and Ammonia Determination in Fertilizers according to the Devarda Method

Reference: UNI EN ISO 15476:2009, AOAC 892.01

Tested with VELP Scientifica UDK 149 Kjeldahl Distillation Unit (Code F30200140)



Introduction

Healthy plants often contain 3 to 4 percent nitrogen in their above-ground tissues. This is a much higher concentration compared to other nutrients.

Nitrogen is so vital because it is a major component of chlorophyll, the compound by which plants use sunlight energy to produce sugars from water and carbon dioxide (i.e., photosynthesis).

Plants thus have the ability to take up several chemical forms of nitrogen from the soil. The most common are: ammonium (NH_4^+), nitrate (NO_3^-), and urea, $((\text{NH}_2)_2\text{CO})$. Many commercial fertilizer mixes contain a combination of all three nitrogen forms.

Determination of nitric and ammonia nitrogen in fertilizer samples

This method describes the procedure for the determination of nitrate and ammoniacal nitrogen with reduction using Devarda's alloy.

When a solution of nitrate ions and Devarda's alloy is mixed with aqueous sodium hydroxide, the mixture gently liberates ammonia gas. This conversion under the form of ammonia, requires some minutes.

The method is applicable to all nitrogenous fertilizers, including compound fertilizers, in which nitrogen is found exclusively in nitrate form or in ammoniacal and nitrate form.

Samples

Two samples of ammonium nitrate fertilizer have been tested:

Fertilizer 1 expected value: 34.69 %N

Fertilizer 2 expected value: 26.79 %N

Reagents and equipment

- Magnetic stirrer
- Burette 50 ml, precision 0.1 ml
- Sodium Hydroxide 32 %
- Devarda Alloy, powder
- Sodium hydroxide 0.1 N
- Sulfuric acid 0.2 N
- Methyl red in ethanol, as indicator

Sample Preparation

300-350 mg of sample have been weighed in a beaker and transferred to the test tube. The beaker is flushed several times with distilled water, then poured to the test tube.

Just before starting the distillation Devarda's alloy powder, about 1.5 g, is added manually to the test tube.

Distillation and titration

Distill the samples with the following parameters (set a customizable method):

- H₂O: 10 ml
- NaOH: 50 ml
- Receiving solution: 0 ml
- Distillation time: 5 min
- Steam power: 80 %
- Pause: 12 minutes

Set *No automatic distillation residues discharge* at the end of the distillation.

As receiving solution in a 250 ml Erlenmeyer flask, 50.0 ml of sulphuric acid 0.2 N (measured with a marked volumetric flask) and 4-5 drops of methyl red indicator solution are added manually.

Titrant solution: NaOH 0.1 N, dosed manually using the 50 ml burette, up to the colour change.

Typical Results on Fertilizer samples

From the ml of titration solution, %N have been calculated by the following formula:

$$\%N = \frac{(V1 \cdot N1 - V2 \cdot N2) - (V1_B \cdot N1 - V2_B \cdot N2) \cdot M_N}{m_{\text{sample}}} \cdot 100$$

where:

V1: ml of the receiving solution (50 ml)
 N1: Normality of the receiving solution (0.2 N)
 V2: ml of NaOH dosed for titration of the sample
 N2: Normality of the NaOH solution (0.1 N)
 V1_B : ml of the receiving solution for the blank (10 ml)
 V2_B : ml of NaOH dosed for titration of the blank
 M_N: Nitrogen molecular weight (14.007 g/mol)
 m_{sample}: sample quantity (mg)

The following table shows the obtained results:

Sample	Sample quantity (mg)	V2 (ml NaOH)	%N
Fertilizer 1	324.7	19.6	34.55
Fertilizer 1	330.0	17.5	34.89
Fertilizer 1	395.5	1.30	34.85
Average ± SD%			34.76 ± 0.18
RSD% *			0.53
Recovery %			100.2
Fertilizer 2	336.2	35.2	26.87
Fertilizer 2	301.2	42.0	26.83
Fertilizer 2	342.9	34.3	26.72
Average ± SD%			26.81 ± 0.08
RSD% *			0.31
Recovery %			100.1

Fertilizer 1: expected value: 34.69 %N

Fertilizer 2: expected value: 26.79 %N

The blank value (V2_B) is 19.7 ml of NaOH.

* RSD% = (Standard Deviation * 100) / Average

The distillation process was verified by using ammonium chloride as standard solution (1000 ppm N), as reference substance.

Conclusions

The determination of nitrate and ammoniacal nitrogen in fertilizer according to Devarda Method by using VELP UDK 149 Distillation Unit has proven to guarantee accurate and precise results corresponding well to specifications.

VELP UDK Distillation Unit is the perfect response for all laboratories looking for reliability, rapidity and ease-of use, producing results that corresponds to the expected values.