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# Fertilizers and soil conditioners — Analytical methods for Sulfur Coated Urea (SCU)

*Matières fertilisantes — Méthodes analytiques pour l'urée enrobée de soufre (SCU)*

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# Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

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The committee responsible for this document is ISO/TC 134, *Fertilizers and soil conditioners*.

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# Introduction

Sulfur Coated Urea (SCU) is a coated, slow release fertilizer consisting of urea particles coated with sulfur, which was first developed by the Tennessee Valley Authority's National Fertilizer Development Center (TVA/NFDC), Alabama in 1961, and produced commercially in 1967. SCU is made by coating urea with sulfur and sealant. It contains 30 % to 40 % nitrogen and 10 % to 30 % sulfur. The main coating material of SCU is sulfur. Sulfur is insoluble in water and its chemical properties are stable, thus, it is an impermeable coating material. In addition, sulfur itself is a secondary nutrient and it does not pollute the soil.

This International Standard specifies analytical methods, including mass fraction of total nitrogen, one-day dissolution rate (1DDR), seven-day dissolution rate (7DDR), mass fraction of sulfur, mass fraction of biuret, mass fraction of water ($H_2O$), and SGN and UI of SCU. There are two methods for determining of one-day dissolution rate (1DDR) and seven-day dissolution rate (7DDR): one is titrimetric method after distillation, the other is refractometer method which is a fast analytical method.

NOTE Some countries or regions might have published other standards covering analytical methods for SCU.

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# Fertilizers and soil conditioners — Analytical methods for Sulfur Coated Urea (SCU)

## 1 Scope

This International Standard specifies analytical methods for the determination of mass fraction of total nitrogen, one-day dissolution rate (1DDR), seven-day dissolution rate (7DDR), mass fraction of sulfur, mass fraction of biuret, mass fraction of water ($H_2O$), and particle size of SCU.

These methods are applicable to SCU.

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 760, *Determination of water — Karl Fischer method (General method)*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

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ISO 5315, *Fertilizers — Determination of total nitrogen content — Titrimetric method after distillation*

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ISO 17323, *Fertilizers and soil conditioners — Sulfur Coated Urea — General requirements*

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## 3 Sampling and sample preparation

Sampling and sample preparation shall be carried out in accordance with ISO 17323.

## 4 Determination of the appearance

It shall be determined by visual method.

## 5 Determination of the mass fraction of total nitrogen

It shall be determined in accordance with ISO 5315.

## 6 Determination of 1DDR and 7DDR

### 6.1 Titrimetric method after distillation

#### 6.1.1 Principle

Digest the testing sample in static water at a constant temperature (38,0 ± 0,5) °C. Within a certain period, the nitrogen component in the testing sample will dissolve into the water through the coatings, and then the released nitrogen can be determined by titrimetric method after distillation. The percentage of released nitrogen to the total nitrogen is defined as 1DDR or 7DDR.

#### 6.1.2 Reagents

See ISO 5315.

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### 6.1.3 Apparatus

**6.1.3.1 Common laboratory apparatus**.

**6.1.3.2 The apparatus listed in** ISO 5315.

**6.1.3.3 Balance**, capable of weighing to the nearest 0, 01 g.

**6.1.3.4 Constant temperature incubator**, capable of being controlled at (38,0 ± 0,5) °C.

### 6.1.4 Procedure

The replicate experiments shall be done for the determination.

**6.1.4.1** Place 20 g uncrushed test sample (accurate to 0,01 g) into a small bag made of 100 meshes nylon yarn nets. Then, seal the bag and place it into a 250 ml Erlenmeyer flask with a plug.

**6.1.4.2** Add 200 ml of water into the flask precisely before the flask sealed with the plug.

**6.1.4.3** Shake the glass flask gently to disperse the particle of test portion. Then, place the glass flask into a constant temperature incubator with temperature set at (38,0 ± 0,5) °C and keep for 24 h and 7 d, respectively.

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**6.1.4.4** After a set period, take out the flask from the incubator and reverse it gently three times to ensure the uniformity of solution concentration throughout the flask.

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**6.1.4.5** Then, cool the flask down to room temperature, and the solution should be filtrated with dry filter paper with a pore size of 30 µm to 50 µm.

**6.1.4.6** Pipette 5 ml of the as-prepared solution; the total released nitrogen during a 24 h and 7 d period should be determined in accordance with ISO 5315.

NOTE 1 Nylon yarn nets were used herein for the convenience of filtration (large undissolved particles of SCU can be discarded together with the nylon yarn net).

NOTE 2 Replicate tests during the actual operation can refer to two, three, or more tests.

### 6.1.5 Calculation

**6.1.5.1** Calculate the total released nitrogen during 24 h period, $w_1$, expressed in the mass fraction (%), according to Formula (1):

$$w_1 = \frac{w'_1}{V_0 / V} \quad (1)$$

where

$w'_1$ is the total released nitrogen of the test solution pipetted during a 24 h period calculated according to ISO 5315, in the unit of mass fraction (%);

$V_0$ is the volume value of the test solution pipetted during the test, in the unit of millilitre (ml);

$V$ is the total volume value of the test solution, in the unit of millilitre (ml).

Express the result to within two decimal places. The average value of the results of parallel tests shall be defined as the final result of the determination.

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**6.1.5.2** Calculate the 1DDR, $x_1$, as the mass fraction (%), according Formula (2)

$$x_1 = \frac{w_1}{w_0} \times 100 \qquad (2)$$

where

$w_1$ is the value of the total released nitrogen during 24 h period, expressed in the mass fraction (%);

$w_0$ is the value of the total nitrogen determined in accordance with the provision 5, expressed in the mass fraction (%).

**6.1.5.3** Calculate the total nitrogen release during 7 d, $w_2$, expressed in the mass fraction (%), with Formula (1), prescribed in 6.1.5.1.

Express the results to within two decimal places. The average value of the results of two parallel tests shall be defined as the result of the test.

**6.1.5.4** Calculate the 7DDR, $x_2$, expressed in the mass fraction (%), according to Formula (3):

$$x_2 = \frac{w_2}{w_0} \times 100 \qquad (3)$$

where

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$w_2$ is the value of the total released nitrogen during 7 d, expressed in mass fraction (%).

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## 6.2 Refractometer method

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### 6.2.1 Principle

First, determine the solid contents in the sulfur coated urea product, and then calculate the mass of urea in the sample. Based on the feature that the mass fraction of urea (%) in a solution at a certain temperature is proportional to the refractive index of the solution, calculate the urea contents (g/l) in the solution by determining the refractive index of the solution.

### 6.2.2 Reagents

**6.2.2.1 Urea solution**, 200 g/l.

Weigh 100 g urea, dissolve it in 250 ml water, and then dilute the solution to 500 ml and mix.

### 6.2.3 Apparatus

**6.2.3.1 Ordinary laboratory apparatus.**

**6.2.3.2 Balance**, capable of weighing to the nearest 0,000 1 g.

**6.2.3.3 Magnetic stirring apparatus.**

**6.2.3.4 Temperature-controlled refractometer**, readability, 0,000 01RI, Accuracy: ±0,000 05RI, temperature accuracy: ±0,05 °C at 20 °C, thermometer resolution: 0,01 °C.

**6.2.3.5 Constant temperature incubator**, capable of being controlled at (38 ± 0,5) °C.

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**6.2.3.6 Drying oven**, capable of being controlled at (100 ± 2) °C.

### 6.2.4 Procedure

#### 6.2.4.1 Preparation of calibration curve

##### 6.2.4.1.1 Preparation of the standard solution

As shown in Table 1, pipette into a series of eight 100 ml volumetric flasks, 0,00 ml (as compensation solution), 2,50 ml, 5,00 ml, 10,00 ml, 20,00 ml, 30,00 ml, 40,00 ml, and 50,00 ml of the urea standard solution. Make each flask up to the mark with water and mix thoroughly.

**Table 1 — Amount of urea content per standard solution**

| Volumes of urea standard solution/ml | The corresponding urea contents/g/l |
|---|---|
| 0,00 | 0,00 |
| 2,50 | 5,00 |
| 5,00 | 10,0 |
| 10,00 | 20,0 |
| 20,00 | 40,0 |
| 30,00 | 60,0 |
| 40,00 | 80,0 |
| 50, 00 | 100,0 |

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##### 6.2.4.1.2 Preparation of the calibration curve

Prior to the test, set the optimum parameters for the refractometer, following the instruction of the guidebook.

Pipette 2 to 3 drops of the as-prepared urea standard solution and directly drop on the measuring disk of the refractometer, then wait for 3 min to 4 min until the temperature is stable at (30 ± 0,1) °C. Then, the refractive index of standard solutions with different concentrations can be measured and recorded.

With the refractive indexes of the urea standard solutions as the ordinate, and the urea contents (g/l) in the corresponding standard solution as the abscissa, the calibration curve can be plotted, and determine the equation of linear regression.

#### 6.2.4.2 Determination of solid contents in samples

The replicate experiments shall be done for the determination.

Weigh 2 g (accurate to 0,000 2 g) of the as-prepared test sample (crushed) into a tall-type beaker, and add 100 ml of water; the system should be mixed up on a magnetic stirrer at least 2 min to form a slurry solution. Make sure that all the granules are completely crushed and the urea is dissolved completely.

Place a piece of weighted filter paper into a Buchner funnel, the paper should be soaked with water and fitted to the shape of the Buchner funnel. Pour the sample containing slurry solution onto the filter paper in the Buchner funnel; the residue on the stirrer should be washed onto the filter paper.

Place the insoluble substances into a drying oven at 103 °C to 105 °C and hold for 45 min, and then cool down to room temperature in a dryer for 30 min. The mass of the insoluble substances together with the filter paper should be weighed and recorded ($m_2$).

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The solid content, $w$, can be calculated, expressed in the mass fraction (%), according to Formula (4):

$$w = \frac{m_2 - m_3}{m_4} \times 100 \tag{4}$$

where

$m_2$ is the mass of the insoluble substances and filter paper, in the unit of gram (g);

$m_3$ is the mass of the filter paper, in the unit of gram (g);

$m_4$ is the mass of the test portion, in the unit of gram (g).

The average value of the results of two parallel experiments shall be defined as the result of the test.

NOTE Replicate tests during the actual operation can refer to two, three, or more tests.

#### 6.2.4.3 Determination of the urea contents in solution

Prepare the sample as set out in 6.1.4.1 to 6.1.4.5.

Pipette 2 or 3 drops of the filtered solution and directly drop on the measuring disk of the refractometer, wait for 3 min to 4 min until the temperature of the solution stabilize at (30 ± 0,1) °C, and then the refractive index should be measured by the refractometer and recorded.

### 6.2.5 Calculation

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**6.2.5.1** Calculate the mass of urea in the sample, $m_5$, in the unit of gram (g), according to Formula (5):

$$m_5 = \frac{(100 - M) \times m_1}{100} \tag{5}$$

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where

$m_1$ is the mass of the uncrushed test portion, in the unit of gram (g).

**6.2.5.2** Calculate 1DDR or 7DDR, $X$, expressed in the mass fraction (%), according to Formula (6):

$$X = \frac{(n - n_0 \times V)}{m_5 \times 1\ 000} \times 100 \tag{6}$$

where

$N$ is the urea concentration of test solution prepared in a period of 24 h and 7 d, determined directly from the calibration curve or calculated by the linear regression equation, corresponding to the refractive indexes, in the unit of gram (g/l);

$n_0$ is the urea concentration value corresponding to the blank refractive index, determined directly from the calibration curve or calculated by the linear regression equation, in the unit of gram(g/l);

$V$ is the total volume value of the test solution, in the unit of millilitre (ml).

The average value of the results of two parallel experiments shall be defined as the result of the test.

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## 7 Determination of the mass fraction of sulfur

### 7.1 Principle

Use water and sulfur-saturated acetone solution to extract water-soluble and acetone-soluble substances, according to the sulfur's behaviour of solubility. Then, extract all the sulfur by carbon disulfide. Calculate the content of sulfur by the subtraction method.

### 7.2 Reagents

**7.2.1 Acetone**

**7.2.2 Sulfur**, solid.

**7.2.3 Carbon disulfide**.

**7.2.4 Sulfur-saturated acetone solution.**

Add a certain amount of sulfur into acetone, and stir continuously. Some more sulfur should be added in the acetone as long as the former can be dissolved thoroughly, until sulfur precipitate from acetone.

### 7.3 Apparatus

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**7.3.1 Ordinary laboratory apparatus.**

**7.3.2 Balance**, capable of weighing to the nearest 0,000 1 g.

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**7.3.3 Glass crucible filter**, No. 4, volume of 30 ml.

**7.3.4 Drying oven**, capable of being controlled at (100 ± 2) °C.

### 7.4 Procedure

**7.4.1 Determination of the sulfur content**

**Warning — This method of analysis involves the use of carbon disulfide ($CS_2$). Special safety measures shall therefore be taken, in particular with regard to the following:**

- **the storage of $CS_2$;**
- **protective equipment for staff;**
- **occupational hygiene;**
- **prevention of fire and explosions;**
- **disposal of the reagent.**

**Warning — This method requires a highly skilled staff and a suitable equipped laboratory.**

The replication experiments shall be done for the determination.

Weigh a certain amount of (with 200 mg to 300 mg sulfur contained) as-prepared test sample (crushed) into a 125 ml Erlenmeyer flask with a plug. Add 50 ml of water into the flask precisely before the flask sealed with the plug. Shake the flask vigorously to dissolve the urea content thoroughly. Remove all



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contents from the triangular flask into a glass crucible filter (7.3.2) which has been dried to a constant weight at (100 ± 2) °C, and then wash the flask five to six times with water.

Wash the glass crucible filter and its contents with 10 ml sulfur-saturated acetone solution (7.2.4), the content should be dried up by a vacuum pump, repeat this operation four times. Then, dehydrate the sample in the drying oven at (100 ± 2) °C for 1 h; after drying, remove the sample from the dryer and cool it down to room temperature and weigh.

Pipette another 10 ml carbon disulfide to wash the test portion, then the content should be dried up by a vacuum pump, repeat this operation 3 to 5 times, until all the sulfur content within the test portion has been rinsed out.

Then, dehydrate the test portion in the drying oven at (100 ± 2) °C for 1 h; after drying, remove the sample from the dryer and cool it down to room temperature and weigh.

The mass difference between the above two weights should be the mass of sulfur content.

#### 7.4.2 Blank test

Replace the test portion with other inert material free of sulfur (ordinary urea, for example), and carry out the blank test in parallel with the determination using the same procedure and the same quantities of all reagents.

### 7.5 Calculation

Calculate the sulfur content (represented by the fraction of S element), $w_3$, expressed in the mass fraction (%), according to Formula (7):

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$$w_3 = \frac{m_7 - m_8 - m_9}{m_6} \times 100 \quad (7)$$

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where

$m_6$ is the mass of the test portion, in the unit of gram (g);

$m_7$ is the mass of the test portion after washing by sulfur-saturated acetone, in the unit of gram (g);

$m_8$ is the mass of the test portion after washing by carbon disulfide, in the unit of gram (g);

$m_9$ is the mass of sulfur in the blank test, in the unit of gram (g).

Express the result to within two decimal places. The average value of the results of parallel experiments shall be defined as the result of the test.

## 8 Determination of the mass fraction of biuret

### 8.1 Principle

Under alkaline conditions in the presence of potassium sodium tartrate, biuret forms a purple complex with copper salts. The absorbance of the solution is measured at a wavelength of 550 nm.

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## 8.2 Reagents

### 8.2.1 Alkaline solution of potassium sodium tartrate.

In a 1 L volumetric flask, dissolve 40 g of sodium hydroxide in 500 ml water and leave it to cool. Add 50 g of potassium sodium tartrate ($NaKC_4H_4O_6$ $4H_2O$). Make the flask up to the mark with water and leave to stand for 24 h before use.

### 8.2.2 Copper sulfate solution.

In a 1 L volumetric flask, dissolve 15 g of copper sulfate pentahydrate ($CuSO_4$ $5H_2O$) in 500 ml water. Make the flask up to the mark with water.

### 8.2.3 Freshly prepared biuret standard solution, corresponding to 0,002 g biuret per millilitre.

Biuret—to recrystallize, weigh 15 g reagent grade biuret (chemically pure), transfer to 1 L beaker, add 500 ml 95 % alcohol(analytical grade), and dissolve. Concentrate by gentle heating to 250 ml. Cool at 5 °C and filter through fritted glass funnel. Repeat crystallization and dry final product for 1 h at 105°in oven. Remove from oven, place in desiccator, and cool to room temperature.

In a 250 ml volumetric flask, dissolve 0,500 0 g of recrystallized biuret in water, make the flask up to the mark with water.

### 8.2.4 Hydrochloric acid solution, $c$ = 1mol/l.

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## 8.3 Apparatus

### 8.3.1 Common used laboratory apparatus.

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### 8.3.2 Balance, capable of weighing to the nearest 0,000 1 g.

### 8.3.3 Spectrophotometer.

### 8.3.4 Water bath, capable of being controlled at (30 ± 5) °C.

## 8.4 Procedure

### 8.4.1 Preparation of the calibration curve.

As shown in Table 2, pipette into a series of six 100 ml volumetric flasks, 0,00 ml, 2,50 ml, 5,00 ml, 10,00 ml, 20,00 ml, and 30,00 ml of the biuret standard solution. Dilute them to 50 ml with water. Add 20,0 ml of the alkaline potassium sodium tartrate solution (8.2.1) and 20,0 ml of the copper sulfate solution (8.2.2) into the volumetric flasks successively, make up to the mark with water, leave to stand for 20 min in a water bath controlled at (30 ± 5)°C and shake again.



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**Table 2 — Amount of biuret content per standard solution**

| Volumes of biuret standard solution/ml | The corresponding biuret contents/mg |
|---|---|
| 0,00 | 0,00 |
| 2,50 | 5,00 |
| 5,00 | 10,0 |
| 10,00 | 20,0 |
| 20,00 | 40,0 |
| 30,00 | 60,0 |

Transfer the solutions to spectrophotometer cells and measure their absorbance at the wavelength of 550 nm using the spectrophotometer, against the compensation solution containing 0 ml of biuret standard solution, 200 ml of the alkaline tartrate solution (8.2.1) and 200 ml of the copper sulfate solution (8.2.2).

Plot the calibration curve with the absorbance value on the ordinate and the corresponding quantities of biuret (in milligrams) on the abscissa. Deduce the equation of regression from the data obtained.

#### 8.4.2 Preparation of the solution to be analysed

Weigh 3 g (accurate to 0,000 2 g) of the as-prepared test sample (crushed) into a 50 ml beaker and add 20 ml of water; the system should be stirred by a glass rod until the urea dissolved. Then, the solution should be filtered into a 100 ml volumetric flask. Add 0,3 ml of hydrochloric acid solution (8.2.4) into the 100 ml volumetric flask and vigorously shake the flask, for the solution might be a little turbid.

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Pipette 20,0 ml of the alkaline potassium sodium tartrate solution (8.2.1) and 20,0 ml of the copper sulfate solution (8.2.2) into the volumetric flasks successively. Then make up to the mark with water, leave to stand for 20 min in a water bath controlled at (30 ± 5) °C and shake again.

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Carry out a blank test in parallel with the determination using the same procedure and the same quantities of all reagents but omitting the test portion.

Measure the absorbance of both the test portion and the blank, then, determine the mass of the biuret from the corresponding calibration curve.

### 8.5 Calculation

Calculate the mass concentration, $w_4$ (%), of the biuret within the sample according to Formula (8):

$$w_4 = \frac{(m_{11} - m_{12}) \times 10^{-3}}{m_{10}} \times 100 = \frac{m_{11} - m_{12}}{m_{10} \times 10} \tag{8}$$

where

$m_{11}$ is the mass of biuret, in the unit of milligrams;

$m_{12}$ is the mass of biuret in blank test, in the unit of milligrams;

$m_{10}$ is the mass of test portion, in the unit of grams.

Express the result to within two decimal places. The average value of the results of parallel experiments shall be defined as the result of the test.

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