INTERNATIONAL STANDARD

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Solid fertilizers — Determination of urea nitrogen content — Gravimetric method using xanthydrol

Engrais solides — Détermination de la teneur en azote sous forme d'urée — Méthode gravimétrique utilisant du xanthydrol

Reference number
ISO 8603:1993(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 8603 was prepared by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners*, Sub-Committee SC 4, *Chemical analysis*.

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Solid fertilizers — Determination of urea nitrogen content — Gravimetric method using xanthydrol

1 Scope

This International Standard specifies a gravimetric method for the determination of the urea nitrogen content of fertilizers. This method is not applicable to fertilizers containing cyanamide or the condensation products of urea and aldehydes (e.g. urea-formaldehyde condensate, dicyandiamide or isobutylidene diurea).

NOTE 1 If biuret is present in the fertilizer, it will be precipitated during the determination but the amount likely to be present is such that the effect on the results of the determination will be negligible.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 3310-1:1990, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth.*

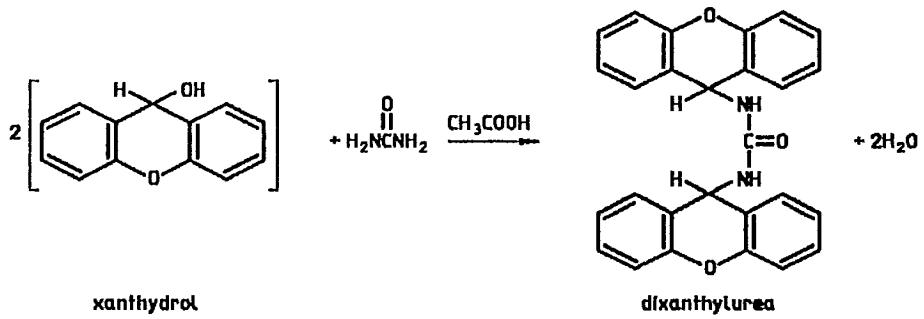
ISO 8358:1991, *Solid fertilizers — Preparation of samples for chemical and physical analysis.*

3 Principle

Precipitation of the urea with xanthydrol in acetic acid solution. Filtration of the dixanthylurea precipitate, drying and weighing.

4 Reaction

See figure 1.



xanthydrol dixanthylurea

Figure 1

5 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Acetic acid, approximately 300 g/l solution.

Dilute 285 ml of glacial acetic acid to 1 000 ml with water.

5.2 Acetic acid, glacial.

5.3 Acetic acid, 50 % (*V*/*V*) solution.

5.4 Ethanol or **methanol**.

5.5 Xanthydrol (9-hydroxyxanthene), 5 % (*m*/*m*) solution in ethanol or methanol.

NOTES

2 Xanthydrol containing a large proportion of insoluble matter should be filtered immediately before use. Commercially available solutions of analytical-grade xanthydrol in methanol [generally 10 % (*m*/*m*)] may be used after dilution with methanol to obtain a concentration of 5 % (*m*/*m*).

3 A freshly prepared solution may be kept for 3 months providing it is stored in a tightly closed bottle, away from light.

6 Apparatus

Ordinary laboratory apparatus and, in particular, the following.

6.1 Test sieve, 1,00 mm nominal size of openings, complying with ISO 3310-1.

6.2 Glass filter crucible, porosity grade P16 (pore size, 5 µm to 16 µm).

6.3 One-mark volumetric flask, 500 ml capacity, complying with class A of ISO 1042.

6.4 One-mark pipettes, 5 ml, 10 ml and 20 ml capacity, complying with class A of ISO 648.

6.5 Mechanical flask shaker, with a rotary or reciprocating action.

6.6 Drying oven, capable of being controlled at 130 °C ± 5 °C.

6.7 Centrifuge, capable of operating at a rate of 1 000 r/min to 6 000 r/min, equipped with centrifuge tubes of 100 ml capacity.

6.8 Desiccator, containing an efficient desiccant.

7 Preparation of test sample

Prepare the test sample in accordance with ISO 8358 and then follow the instructions below.

If necessary, crush the sample so that it completely passes the test sieve (6.1). Carry out the crushing (if any) and sieving operations as quickly as possible, in the absence of any moisture. Also, avoid loss of moisture by protecting the sample from any source of heat.

Mix the test sample thoroughly with a spatula to make it homogeneous and store it in a tightly closed bottle.

8 Procedure

8.1 Test portion

Weigh, to the nearest 0,001 g, about 10 g of the test sample and transfer it to the volumetric flask (6.3).

8.2 Preparation of test solution

8.2.1 Samples soluble in water

Add about 400 ml of water to the test portion (8.1) and shake the flask continuously for 30 min, using the mechanical flask shaker (6.5). Dilute to the mark with water and mix.

8.2.2 Samples containing water-insoluble material likely to retain urea

Add 50 ml of water and 50 ml of the acetic acid solution (5.1) to the test portion (8.1). Mix the contents of the flask and allow to stand undisturbed until the liberation of carbon dioxide has ceased. Add about 300 ml of water and shake the flask continuously for 30 min, using the mechanical flask shaker (6.5). Dilute to the mark with water and mix.

8.3 Determination

8.3.1 Filter the test solution (8.2) through a dry, medium-speed, low-ash grade filter paper into a clean, dry conical flask. Discard the first 50 ml of the filtrate and transfer one aliquot portion of the remainder, containing not more than 20 mg of ureic nitrogen, into a centrifuge tube (6.7), using one of the one-mark pipettes (6.4). The volume of the aliquot portion of the filtered test solution shall be as given in table 1.

Table 1 — Volume of filtered test solution used for each determination

Expected ureic nitrogen content of the sample, % (*m*/*m*)	< 3	2 to 7	7 to 15[1)]
Volume of filtered test solution, ml	20	10	5

1) For higher contents of urea nitrogen, reduce the mass of the test portion.

8.3.2 Add, to the aliquot portion (8.3.1), 40 ml of the glacial acetic acid (5.2) and stir with a glass rod for 1 min. Then proceed as in a) or b) as appropriate.

a) If no precipitate is formed, add 10 ml of the acetic acid solution (5.3).

b) If a precipitate is formed, allow it to settle for 5 min and then centrifuge: usually 2 min to 3 min at a rotational frequency of 3 000 r/min is sufficient to obtain a clear solution. Decant the clear solution quantitatively into a 100 ml beaker. Add 5 ml of the dilute acetic acid solution (5.3) to the centrifuge tube, stir the precipitate with the same glass rod and centrifuge again. Decant the clear solution into a beaker containing the main solution. Repeat twice the process of washing the precipitate.

Add, drop by drop, 10 ml of the xanthydrol solution (5.5), stirring continuously with a glass rod. Allow to stand until the precipitate appears, then stir again for 1 min to 2 min and allow to stand for 1,5 h.

Collect the precipitate quantitatively, using suction, in the glass filter crucible (6.2) which has been previously dried for 1 h in the oven (6.6), set at 130 °C, cooled in the desiccator (6.8) to room temperature and weighed.

Wash the precipitate three times with 5 ml portions of the ethanol or methanol (5.4) and place the filter crucible in the oven (6.6), set at 130 °C, for 1 h. Allow the filter crucible to cool in the desiccator (6.8) and weigh. Repeat the operations of heating, cooling and weighing until two successive weighings do not differ by more than 0,001 g.

8.4 Blank test

Carry out a blank test at the same time as the determination, following the procedure specified in 8.3.2, but replacing the aliquot portions of the test solution with water or, if the acetic acid solution (5.1) is used to dissolve the test portion (see 8.2.2), with a solution of the same acetic acid concentration.

If the mass of the precipitate obtained in the blank test exceeds 0,001 g, repeat the entire test using a fresh xanthydrol solution (5.5).

9 Expression of results

The urea nitrogen content, expressed as a percentage by mass of nitrogen, is given by the equation

$$N = \frac{0{,}066\ 7 \times (m_1 - m_2) \times 500 \times 100}{m_0 \times V}$$

i.e.

$$N = \frac{3\ 335 \times (m_1 - m_2)}{m_0 \times V}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the precipitate obtained in the determination;

m_2 is the mass, in grams, of the precipitate obtained in the blank test;

V is the volume, in millilitres, of the aliquot portion of the test solution taken as described in 8.3.1;

0,066 7 is the mass, in grams, of nitrogen in 1 g of dixanthylurea.

10 Precision

Table 2 — Repeatability limit (r) and reproducibility limit (R)

Ureic nitrogen content, % (m/m)	r, % (m/m) nitrogen	R, % (m/m) nitrogen
6,1	0,14	0,48
9,4	0,24	0,55
20,1	0,45	1,14

10.1 General

The precision data were determined from an experiment conducted in 1981 involving 24 laboratories and 3 levels.

10.2 Repeatability

The difference between two single test results obtained from identical test material by one analyst using the same apparatus within a short time-interval shall exceed the repeatability limit, r, given in table 2, on average not more than once in 20 cases in the normal and correct operation of the method.

10.3 Reproducibility

The difference between two single and independent test results found by two analysts working in different laboratories using identical test material shall exceed the reproducibility limit, R, given in table 2, on average not more than once in 20 cases in the normal and correct operation of the method.

11 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) the results and the method of expression used;

c) all information necessary for the complete identification of the sample;

d) any unusual features noted during the determination;

e) any operation not included in an International Standard or in an International Standard to which reference is made, or regarded as optional.

UDC 631.8:543.21:546.17

Descriptors: fertilizers, urea, chemical analysis, determination of content, nitrogen, gravimetric analysis.

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