BS ISO 18611-2:2014

BSI Standards Publication

Ships and marine technology — Marine NOx reduction agent AUS 40

Part 2: Test methods

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National foreword

This British Standard is the UK implementation of ISO 18611-2:2014.

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Part 2:
Test methods

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Partie 2: Méthodes d’essai

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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.

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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

The committee responsible for this document is ISO/TC 8, *Ships and marine technology*, Subcommittee SC 2, *Marine environment protection*.

ISO 18611 consists of the following parts, under the general title *Ships and marine technology — Marine NOx reduction agent AUS 40*:

- *Part 1: Quality requirements*
- *Part 2: Test methods*
- *Part 3: Handling, transportation and storage*

Introduction

In order to protect the environment and to enhance air quality, exhaust emissions regulations around the world are continuously strengthened. For ships with large combustion engines, particulate matter (PM), nitrogen oxide (NOx) emissions, and sulfur dioxide emissions are the main concern, and efforts have been focused on the development of technology that can reduce them effectively with minimum fuel economy penalty. Selective catalytic reduction (SCR) converters using a urea solution as the reducing agent is considered to be a key technology for reducing NOx emissions. The quality of the urea solution used for that technology needs to be specified to ensure reliable and stable operation of the SCR converter systems. The ISO 18611 series provides the specifications for quality characteristics, for handling, transportation, and storage, as well as the test methods needed by manufacturers of SCR converters, by engine producers, by producers, distributors of the urea solution, and by fleet operators/ship owners.

Efficient expanding of the use of urea SCR technology requires a consolidated framework that can be followed by producers, end users, OEMs, and catalyst suppliers.

INTERNATIONAL STANDARD **ISO 18611-2:2014(E)**

Ships and marine technology — Marine NOx reduction agent AUS 40 —

Part 2:
Test methods

1 Scope

This part of ISO 18611 specifies test methods required for the determination of the quality characteristics of the NOx reduction agent AUS 40 (aqueous urea solution) specified in ISO 18611-1. In the remaining parts of ISO 18611, the term “NOx reduction agent AUS 40” will be abbreviated to “AUS 40”.

This International Standard is covering quality requirements and guidelines for AUS 40 for marine applications, irrespective of manufacturing method or technique.

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 3675, *Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 4259, *Petroleum products — Determination and application of precision data in relation to methods of test*

ISO 5661, *Petroleum products — Hydrocarbon liquids — Determination of refractive index*

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO 12185, *Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method*

3 Specifications

Compliance with the limits specified in ISO 18611-1, Table 1 shall be determined by the test methods specified in Annexes B through J of this part of ISO 18611. Determination of the density shall be conducted in accordance with ISO 3675 or ISO 12185.

NOTE For the purposes of this International Standard, the terms “%(*m*/*m*)” and “%(V/V)” are used to represent the mass fraction and the volume fraction of a material respectively.

4 Sampling

Samples shall be taken in accordance with Annex A.

5 Precision and dispute

5.1 General

All test methods referred to in this part of ISO 18611 include a precision statement according to ISO 4259. In cases of dispute, the procedures described in ISO 4259 shall be used for resolving the dispute, and interpretation of the results based on the test method precision shall be used. The test methods described in this International Standard has been adopted from ISO 22241-2. Some minor changes have been made to the ISO 22241 test methods in order to adapt it to the specifications as described in ISO 18611-1, Table 1.

The precision of the test methods has been adopted from ISO 22241-2. In ISO 22241-2, the precision was determined by statistical examination in accordance with ISO 4259 with the exception of the methdods for determination of density that was taken from ISO 3675 and ISO 12185. The precision of the test methods is specified in each annex. Additionally, this information is summarized in Annex K for all test methods for the convenience of the user of this part of ISO 18611.

The statistical significance of the precision quoted in this part of ISO 18611 is generically defined in 5.2 and 5.3, in which the “xx (unit)” stands for the repeatability and reproducibility in question.

5.2 Repeatability, *r*

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material should, in the long run, in the normal and correct operation of the test method, exceed xx (unit) in only one case in 20.

5.3 Reproducibility, *R*

The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed xx (unit) in only one case in 20.

Annex A
(normative)

Sampling

A.1 General

The sampling method specified in this annex is valid for each sampling of AUS 40 throughout the supply chain after the shipment from the manufacturer's site to the AUS 40 containers on board the vessels.

A.2 Principle

The limits for the quality characteristics of AUS 40, which are specified in ISO 18611-1, are the representative analytical results that can only be obtained when the sample is protected from any contamination before the analysis.

Therefore, suitable bottles shall be used for sampling, which do not contaminate the sample, especially regarding the trace elements, and which minimize the risk of algae or bacteria growth.

NOTE The sampling method specified in this annex is based on ISO 5667-2 and ISO 5667-3.

A.3 Possible contaminants

During the sampling process, foreign matter may lead to contamination of the sample. Under realistic conditions, the following sources of contamination will pose a major hazard:

- residues of process aids used for the production of the sampling bottles;
- contaminants which have been deposited in the empty bottles during the time they are stored empty;
- contaminants from the air, i.e. dust or any foreign matter from the surrounding, during the sampling;
- residues of cleaning agents, which have been used for cleaning the sampling equipment and the bottles as well;
- fuel.

A.4 Apparatus

A.4.1 Sampling bottles

1000-ml wide neck bottles shall be used. Suitable materials for these bottles are HD-polyethylene, HD-polypropylene, polyfluorethylene, polyvinylidenedifluoride, and tetrafluoroethylene-perfluoroalky vinyl ether copolymer (PFA). In case of dispute, PFA bottles should be used.

Prior to the first use with AUS 40, the bottles shall be cleaned and finally rinsed with de-ionized water followed by AUS 40.

A.4.2 Labels

Each bottle shall be labelled using labels of approximately 10 cm × 5 cm. The labels and the writing on these labels shall be resistant to water and to AUS 40.

A.5 Sampling

The locked wide-neck bottle shall be opened; the cap shall be put down on a clean surface with the opening turned downward. After flushing the sampling pipe, the bottle shall be filled completely with AUS 40 from the container. The first filling shall be discarded, and the bottle shall immediately be re-filled with AUS 40 and closed tightly. The label shall be attached to the bottle (see A.4.2). During the filling of the sample, due care shall be taken that neither dust nor liquid pollutants get into the bottle.

The filled bottle should reach the laboratory as soon as possible. During transportation and storage, the sample should be kept at the lowest possible temperature, preferably between 1 °C and room temperature (20 °C), and kept away from daylight to prevent growth of algae. Samples to be stored for a long period of time should be stored at between 1 °C and 15 °C and should be kept away from daylight.

It is recommended to conduct the analysis within three weeks in order to take into account possible changes in the ammonia content.

A.6 Sample quantity

The minimum quantity of sample material depends on the type of analysis conducted. Whenever possible, make sure that a sufficient volume of sample material is available (recommendation: 1 litre), and at least double that which is required for complete verification of AUS 40 specifications. In case of dispute, a sufficient number of samples shall be taken according to ISO 4259.

A.7 Labelling and Chain of Custody information

Samples will be labelled and a Chain of Custody form shall be utilized in order to track the movement of the samples as they are transferred to a laboratory for analysis.

A.7.1 Labelling

The label should contain the following information:

- sample identification number;
- product name;
- address/vessel name where the sample was taken from1);
- container/location from which the sample was taken1);
- date and time of sampling1).

A.7.2 Chain of Custody information

The Chain of Custody form shall contain the following information:

- sample identification number;
- product name;
- address/vessel name where the sample was taken from1);
- container/location from which the sample was taken1);
- date and time of sampling1);
- name and signature of person who sampled
- date when the sample left the site.

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1) Mandatory only in cases of dispute.

Annex B
(normative)

Determination of urea content by total nitrogen

B.1 General

This annex specifies the procedure for determining the urea content of AUS 40.

The method is applicable for the determination of the urea content in the range from 38 % to 42 % (*m*/*m*).

B.2 Principle

The sample is combusted at high temperatures in a stream of oxygen. Following the reduction of formed nitrogen oxides to elemental nitrogen and removal of any interfering products of combustion, nitrogen is measured with a thermal-conductivity detector. The urea content is calculated from the determined total nitrogen minus the nitrogen content of biuret.

B.3 Apparatus

B.3.1 Automatic nitrogen analyser

The unit to be used is based on combustion methods.

B.3.2 Analytical balance

The accuracy of the balance is a function of the analyser used and the required weighed portions. Resolution should be 0,1 % or better of the weighed portion.

B.3.3 Auxiliary devices for sample preparation

Examples of these devices include

- tweezers with a blunt tip,
- micro-spatula with a flattened tip, and
- pipette.

The pipette is recommended for weighing in and thus does not need to be calibrated. It is important, however, to obtain a good droplet size (small droplets). Fixed-volume pipettes or pipettes with an adjustable volume in the range of 10 µl to 1 000 µl or single-trip Pasteur pipettes with a fine tip can also be used.

NOTE This is customarily chemically resistant glass.

B.4 Chemicals

B.4.1 De-ionized water, conductivity less than 0,1 mS/m, according to ISO 3696, grade 2.

B.4.2 Auxiliary combustion agent and other equipments, appropriate for use with the selected nitrogen analyser.

The following materials are merely examples. Other or similar materials can be used as required, depending on the system that is available:

— tin capsule or similar sample containers;

— auxiliary combustion agent, non-nitrogenous, such as saccharose, cellulose;

— absorbing agent for liquids, non-nitrogenous, such as magnesium oxide.

B.4.3 Standard substances for nitrogen determination, preferably with certified nitrogen content.

EXAMPLE Suitable standard substances include ethylenediamine tetraacetic acid (EDTA), nicotinic acid amide.

Low-biuret urea of adequate purity (for example, crystalline ultra pure or analytical) or other such standard substances recommended by and available from the equipment manufacturer can also be used. Certified standard substances should be preferred.

NOTE Liquid standard substances (e.g. urea solutions) are not suited for calibration purposes.

B.4.4 Oxygen, min. 99,995 % O_2.

B.4.5 Other ultrapure gases, if required to operate the nitrogen analyser, such as helium, min. 99,996 % He.

B.4.6 Other reagents or auxiliary agents, as required by the equipment.

B.5 Procedure

B.5.1 General

The sample should be fully dissolved and free from urea crystals. It can be heated to max. 40 °C as required prior to further processing.

NOTE Different types of apparatus are available on the market. The resulting various resources and modes of operation are not an object of this part of ISO 18611. Rather, activity should be based on the respective operation manuals.

B.5.2 Reference curve

Perform calibration as required for the specific type of analyser and according to the respective operation manuals (for example, after replacement of the combustion tube, reagent or similar) by performing measurements as described in B.5.4. Weigh in an appropriate amount of standard substances repeatedly as appropriate for the respective types of apparatus to obtain a reference curve.

B.5.3 Inspecting the apparatus for good working order, and the reference curve

Use an appropriate standard substance to review the good working order of the apparatus and the reference curve. Preferably, a certified urea standard solution should be used.

Frequency of inspection is a function of the analyser used.

B.5.4 Measurement

Weigh a portion of the sample in a suitable holder (such as a tin capsule) as specified for the type of nitrogen analyser used. The amount should be such that the absolute amount of nitrogen is in the middle range of the reference curve.

Use approximately the threefold amount of combustion agent (for example, non-nitrogenous cellulose) and additional binders (for example, magnesium oxide) as required.

When using liquid feeder systems, the volume used should be no less than 100 µl. The sample mass is calculated using the density that was calculated according to ISO 12185.

Enter the required data (weighed portion, sample identification) into the analyser (or a control computer), depending on the type of apparatus. Feed the weighed-in sample to the analyser, and start combustion.

Perform at least three (3) single determinations.

B.6 Results

B.6.1 Calculation

Prior to calculating the reference curve, drift of the baseline, or samples, determine the blank reading value by means of blank samples, and use this value to correct the respective analytical sequences.

Use the apparatus-specific programme to calculate the reference curve or the drift correction for the samples.

Calculate the mean value for the samples. If there is a strong dispersion of single values (relative standard deviation RSD > 1,0 %), repeat the affected sample. After that, determine the mean value for this sample from all single values.

Determine the urea content from the mean value from at least three nitrogen determinations:

$$w_{\mathrm{U}} = 2{,}1438 \times \left(w_{\mathrm{N}} - F_1 \times w_{\mathrm{Bi}} - F_2 \times w_{\mathrm{NH3}} \right) \tag{B.1}$$

where

w_{U} is the urea content [% (*m*/*m*)];

w_{N} is the mean value of the nitrogen content [% (*m*/*m*)] (to the nearest 0,01 %);

w_{Bi} is the mean value of the biuret content (%), determined according to Annex E;

w_{NH3} is the mean value of the ammonia content (%), determined according to Annex D;

F_1 is the factor for converting the biuret content to nitrogen (0,407 6);

F_2 is the factor for converting the ammonia content to nitrogen (0,822 5).

B.6.2 Expression of results

The result is the arithmetic mean value from at least three (3) single determinations (nitrogen determinations).

Round off the result of the urea content calculation to the nearest 0,1 %.

B.7 Precision

See 5.2, 5.3, and Table B.1.

Table B.1 — Precision

Urea content w_U % (m/m)	**Repeatability** r % (m/m)	**Reproducibility** R % (m/m)
38 to 42	0,4	1,0

B.8 Test report

The report shall include the following data as a minimum requirement:

a) type and description of tested product;

b) reference to this part of ISO 18611 (i.e. 18611-2);

c) sampling method used;

d) test result (see B.6);

e) deviations from the specified mode of operation, if any;

f) test date.

Annex C
(normative)

Refractive index and determination of urea content by refractive index

C.1 General

This annex specifies the procedure for the determination of the refractive index of AUS 40. The test method is applicable to liquids having refractive indices in the range of 1,33 to 1,41 and at temperatures of 20 °C to 30 °C.

Based on the measurement of refractive index, the method shall be used for determining the content of urea in the range of 38 % to 42 % (*m*/*m*).

C.2 Principle

Measurement is based on the dependence of the refractive index on the concentration of urea in an aqueous solution at a definite temperature.

The content is determined by means of a reference curve.

NOTE The method specified in this annex is based on ISO 5661.

C.3 Apparatus

C.3.1 Refractometer, measuring range 1,330 00 to 1,410 00, resolution 0,000 01.

C.3.2 Analytical balance, resolution 0,1 mg or better.

C.3.3 Thermostat, temperature-control precision 0,02 °C.

C.3.4 Drying oven.

C.3.5 150 ml beaker, tall form.

C.3.6 Typical laboratory glass.

C.4 Chemicals

C.4.1 De-ionized water, conductivity less than 0,5 mS/m, according to ISO 3696, grade 3.

C.4.2 Urea, crystalline, with biuret content less than 0,1 % (*m*/*m*).

Prior to weighing the urea to draw the reference curve, it shall be dried for 2 h at 105 °C.

C.4.3 **Urea test solution**, 40 % (*m/m*).

The test solution shall be made by exactly weighing urea and water. The desired value and the permissible dispersion shall be established through 10 measurements.

The solution shall be kept air-tight in the refrigerator and should be used within 12 weeks maximum.

C.5 Procedure

C.5.1 General

The sample should be fully dissolved and free from urea crystals. It can be heated to ≤40 °C as required prior to further processing.

Different types of apparatus are available on the market. The resulting various resources and modes of operation are not an object of this part of ISO 18611. Rather, operation should be based on the respective operation manuals.

C.5.2 Drawing the reference curve and determining the evaluation factor

The following urea solutions shall be prepared by weighing urea in glass beakers and then adding the corresponding quantity of de-ionized water: 38,0 % (*m/m*) / 39 % (*m/m*) / 40 % (*m/m*) / 41 % (*m/m*) / 43,0 % (*m/m*).

The refractive index of these solutions shall be determined at 20 °C ± 0,02 °C.

The diagram shall show a stringent linear relationship between the refractive index and concentration. An evaluation factor shall be calculated from the urea concentrations and the refractive indices:

$$F = \frac{\sum_{i=1}^{5} w_{\mathrm{U},i}}{\sum_{i=1}^{5} \left(n_{\mathrm{U},i} - n_{\mathrm{W}} \right)} \tag{C.1}$$

where

F is the evaluation factor (%);

$w_{\mathrm{U,i}}$ is the urea content of the *i*-th reference solution [%(*m/m*)];

$n_{\mathrm{U,i}}$ is the refractive index of the *i*-th reference solution;

n_{W} is the refractive index of water and is 1,332 96 when measured with a refractometer of five-decimal resolution.

C.5.3 Checking the instrument function and the reference curve

The instrument function shall be checked weekly using water or a reference standard. If a deviation greater than 0,000 02 from the desired value occurs, adjust the instrument according to the instructions provided by the manufacturer. If afterwards the desired value is not attained, then the instrument shall be disabled for further measurements and the manufacturer’s service should be called for.

Adjust the thermostat to the desired temperature, reading this temperature on the refractometer thermometer on the discharge side. Maintain the flow of water so that the desired temperature shall be reached and maintained within ±0,02 °C.

Furthermore, the reference curve shall be verified weekly with urea solution [32,5 % (*m/m*)]. In the process, the refractive index shall be determined and the concentration shall be calculated with the help of the factor according to C.6. If the concentration determined deviates from the desired value by more than 0,1 % (*m*/*m*), a new test solution shall be used. If the deviation persists, the reference curve shall be created anew.

C.5.4 Sample preparation and measuring

The original sample shall be measured at 20 °C ± 0,02 °C without further preparation.

Measure the urea content two times with different test portions. Should the difference between the separate values be more than 0,000 05, the measurements shall be repeated.

C.6 Results

C.6.1 Calculation

Urea content shall be calculated according to Formula (C.2):

$$w_{\mathrm{U}} = \left(n_{\mathrm{P}} - n_{\mathrm{W}}\right) \times F - w_{\mathrm{Bi}} - W_{\mathrm{NH3}} \tag{C.2}$$

where

w_{U} is the urea content [% (*m*/*m*)];

n_{P} is the refractive index of the sample (with five decimals);

n_{W} is the refractive index of water (with five decimals);

F is the evaluation factor (%);

w_{Bi} is the biuret content of the solution [% (*m*/*m*)]

(determined according to Annex E; biuret has the same refractive index as urea);

$\mathrm{w}_{\mathrm{NH3}}$ is the ammonia content of the sample determined according to Annex D [ammonia dissolved in water can be assumed to have the same refractive index as water if concentration is less than 0,5 % (m/m)].

C.6.2 Expression of results

The result is defined as the arithmetic mean of two single measurements. Round off the result of the refractive index to four decimals. Round off the result of the urea content calculation to the nearest 0,1 % (*m*/*m*).

C.7 Precision

See 5.2, 5.3, and Table C.1.

Table C.1 — Precision

Characteristics	Repeatability *r*	Reproducibility *R*
Refractive index n_P 1,33 to 1,41	0,000 1	0,001 0
Urea content w_U 38 % to 42 % (*m*/*m*)	0,1 % (*m*/*m*)	1,0 % (*m*/*m*)

C.8 Test report

The report shall include the following data as a minimum requirement:

a) type and description of tested product;

b) reference to this part of ISO 18611 (i.e. 18611-2);

c) sampling method used;

d) test result (see C.6);

e) deviations from the specified mode of operation, if any;

f) test date.

Annex D
(normative)

Determination of alkalinity

D.1 General

This annex specifies the procedure for the determination of the alkalinity of AUS 40, calculated as ammonia, in the range 0,1 % to 0,5 % (*m/m*).

D.2 Principle

The measurement is based on potentiometric titration of free ammonia of a test portion with a standard volumetric hydrochloric acid solution to the end point at pH = 4.5

D.3 Apparatus

D.3.1 Analytical balance, resolution 0,1 mg or better.

D.3.2 Automatic burette.

D.3.3 Potentiometer, capable of measuring with a precision of 0,01 pH units, equipped with glass combined pH-electrode.

D.3.4 Magnetic stirrer.

D.3.5 Beaker, 150 ml, tall shaped.

D.3.6 Measuring cylinder, 100 ml.

D.4 Chemicals

D.4.1 General

During the analysis, use only reagents of recognized analytical grade and only distilled or de-ionized water of an electric conductivity lower than 0,5 mS/m, according to ISO 3696, grade 3.

D.4.2 Hydrochloric acid

This is 0,01 mol/l standard solution.

D.4.3 Buffer solutions

The following standard buffer solutions shall be used for the determination of alkalinity:

- standard buffer solution, pH = 4,008;
- standard buffer solution, pH = 9,184;
- standard buffer solution, pH = 8,00.

NOTE Such solutions are commercially available.

D.5 Procedure

D.5.1 Interferences

The samples of AUS 40 taken shall be stored and shipped at a temperature not higher than 25 °C in order to avoid ammonia formation.

The containers shall be closed tightly and the analysis time shall not be protracted in order to avoid evaporation of ammonia.

D.5.2 Check of potentiometric system

The correct function of the potentiometric system shall be checked by use of the standard buffer solutions at pH = 4,008 and pH = 9,180.

The standard buffer solution at pH = 8,00 shall be used for daily check of the potentiometric system.

D.5.3 Preliminary test

Weigh about 1 g of the homogenous sample ±0,05 g (sample mass m_S) and put it into a 150 ml beaker filled with about 100 ml distilled or de-ionized water.

Titrate with the hydrochloric acid solution (0,01 mol/l) under stirring to the end point at pH = 4,5.

Calculate the content of ammonia.

Depending on the content of alkalinity found, weigh the following sample portions for the determination:

- alkalinity content found by the preliminary test [% (*m*/*m*)]: 0,02 0,05 0,1 0,2 to 0,5
- mass of test portion for the determination (g): 10 5 2 1
- see D.6.1 for example.

D.5.4 Determination

Weigh the mass of the homogenous sample ±0,05 g found by the preliminary test (sample mass m_S) and put it into a 150 ml beaker filled with about 100 ml distilled or de-ionized water.

Titrate with the hydrochloric acid solution (0,1 mol/l) under stirring at first to pH = 7,5 with normal speed, then titrate to the end point at pH = 4,5 with reduced speed.

Perform two measurements.

D.6 Results

D.6.1 Calculation

The alkalinity, expressed as a percentage by mass of ammonia (NH_3), is given by Formula (D.1):

$$w_{NH3} = (V \times 0{,}017) / m_S \tag{D.1}$$

where

w_{NH3} is the alkalinity, calculated as ammonia [% (m/m)];

V is the volume of the hydrochloric acid solution used for the titration (ml);

m_S is the mass of the test portion (g).

D.6.2 Expression of results

Calculate the mean value of the two measurements. Express the result to the nearest 0,01 % (m/m).

D.7 Precision

See 5.2, 5.3, and Table D.1.

Table D.1 — Precision

Alkalinity content $w(NH_3)$ % (m/m)	**Repeatability** r % (m/m)	**Reproducibility** R % (m/m)
0,1 to 0,5	0,01	0,2 × x
NOTE x is mean value.		

D.8 Test report

The report shall include the following data as a minimum requirement:

a) type and description of tested product;

b) reference to this part of ISO 18611 (i.e. 18611-2);

c) sampling test method;

d) test result (see D.6);

e) deviations from the specified mode of operation, if any;

f) test date.

Annex E
(normative)

Determination of biuret content

E.1 General

This annex specifies the procedure for the determination of the biuret content of AUS 40 with contents of biuret from 0,1 % to 0,8 % (*m/m*) by photometric method. The method is also applicable to contents up to 1,5 % (*m/m*); however, precision data have not been determined.

E.2 Principle

Biuret forms in alkaline solution in the presence of sodium-potassium-tartrate with bivalent copper a violet-coloured complex with an absorption maximum at 550 nm. The colour complex is read spectrophotometrically at 550 nm and the biuret concentration is determined by reference to a calibration curve prepared from standard biuret solutions.

E.3 Apparatus

E.3.1 Laboratory balance, resolution in reading 0,001 g.

E.3.2 Vacuum filtration unit, applicable for filter with 0,45 µm pore size.

E.3.3 Spectrophotometer, for use at 550 nm with 50-mm-cell.

E.3.4 Volumetric flasks, 1 000 ml, 250 ml, 100 ml, 50 ml.

E.3.5 Pipettes.

E.3.6 Rotary evaporator.

E.3.7 Constant-temperature bath, capable of maintaining a temperature of 30 °C ± 1 °C.

E.4 Chemicals

E.4.1 Chemicals of analytical grade

These shall be used in all tests. The water shall be de-ionized and boiled out to remove carbon dioxide before use.

E.4.2 Saturated potassium carbonate solution

E.4.3 Copper sulfate solution

Dissolve 15 g copper sulfate ($CuSO_4 \cdot 5H_2O$) in CO_2-free water and dilute to 1 000 ml.

E.4.4 Alkaline potassium sodium tartrate solution

Dissolve 40 g sodium hydroxide in 500 ml water in a 1 000 mI volumetric flask. After cooling, add 50 g potassium sodium tartrate ($KNaC_4H_4O_6 \cdot 4H_2O$) and fill up the flask with water to the mark. Let the flasks stand 1 day before use.

E.4.5 Biuret standard solution, of 0,8 mg biuret/ml

Dissolve 800 mg pure biuret in CO_2-free water and dilute to 1 000 ml. Dry the biuret for 3 h at 105 °C before use.

Biuret can be purified as follows:

- add 50 g biuret to 500 ml ammonia solution of 25 % (*m/m*) concentration and stir for 15 min;
- filter, rinse with ammonia-free water, and dry the biuret;
- dissolve in ethanol (10g/1 litre), filter, and concentrate by gentle heating to one-fourth the volume;
- cool to 5 °C and filter;
- dry the biuret in vacuum oven at 80 °C;
- check the purity by photometrical measurements according to E.5.5.

The step of re-crystallizing from ethanol shall be repeated until there is no more noticeable improvement of purity.

E.5 Procedure

E.5.1 Interferences

Spectrophotometric measuring is only allowed with clear solutions. Pass the sample through a 0,45 µm filter to get a clear solution.

Ammonia forms with bivalent copper a coloured complex, which absorbs light energy at 550 nm. The method is applicable only if the ammonia-content of the sample is less than 500 mg/kg.

To remove ammonia-contents greater than 500 mg/kg, put 50,0 g of the sample in a 1 l flask of a rotary evaporator, add 15 ml potassium carbonate solution and evaporate it for 1 h at 40 °C with a rotating speed of 60 r/min and under a vacuum of 2 kPa to 3 kPa to an end volume of approximately 20 ml. Transfer this volume into a 250 ml volumetric flask.

E.5.2 Preparation of the calibration curve

Into a series of six 50 ml volumetric flasks, transfer 2 ml, 5 ml, 10 ml, 15 ml, 20 ml, and 25 ml of the biuret standard solution and add water (to each of the six flasks) to a total of mixture volume of approximately 25 ml. Add, while stirring after each addition, 10 ml of the alkaline potassium sodium tartrate solution and 10 ml of the copper sulfate solution. Immerse the flasks in the constant-temperature bath, regulated at 30 °C ± 1 °C and leave them there for about 15 min.

Carry out a blank test in parallel with the determination; following the same procedure and using the same quantities of all the reagents used for the measurement (see E.5.5).

After cooling to room temperature, fill up the flasks with water to the mark and mix well. Carry out the photometric measurements with the spectrophotometer at a wavelength of about 550 nm using a 50 mm cell against water as the reference.

Subtract the extinction of the blank test from the extinction of the measured values and set up the calibration curve. In the concentration range, the curve shall be strictly linear.

E.5.3 Calculation of the calibration factor

Calculate the calibration factor according to Formula (E.1):

$$F = \frac{\sum_{i=1}^{6} m_{\mathrm{Bi},i}}{\sum_{i=1}^{6} \left(E_{1,i} - E_2\right)} = \frac{61{,}6}{\sum_{i=1}^{6} \left(E_{1,i} - E_2\right)} \tag{E.1}$$

where

F is the calibration factor (mg);

$m_{\mathrm{Bi},i}$ is the mass of biuret of the *i*-th sample (mg);

$E_{1,i}$ is the extinction of the *i*-th sample;

E_2 is the extinction of the blank test.

The determination of the calibration curve and the calibration factor shall be repeated on a yearly basis and shall be documented.

E.5.4 Day-factor

The day-factor shall be determined on a weekly basis.

Perform a measurement of 10 ml of the biuret standard solution (8 mg biuret) as described in E.5.5.

Calculated in Formula (E.2):

$$F_{\mathrm{D}} = \frac{8}{\left(E_1 - E_2\right)} \tag{E.2}$$

where

F_{D} is the day-factor (mg);

E_1 is the extinction of the standard solution (average from two measures);

E_2 is the extinction of the blank test.

The deviation of the day-factor shall be within ±5 % to the calibration factor. For measuring of samples, the day-factor shall be used.

E.5.5 Measurement

Weigh, to the nearest 0,01 g, 100 g of the test sample in a 250 mI volumetric flask. Fill the flask to the mark with water and mix well.

Transfer an aliquot of 10 ml from the test solution into a 50 ml volumetric flask and add water to approximately 25 ml. Add, with stirring after each addition, 10 ml of the alkaline potassium sodium tartrate solution and 10 ml of the copper sulfate solution. Immerse the flask in the constant-temperature bath, regulated at 30 °C ± 1 °C and leave it there for about 15 min.

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all the reagents used for the determination.

After cooling to room temperature, fill up the flask with water to the mark and mix well. Carry out the photometric measurements with the spectrophotometer at a wavelength of about 550 nm using a 50 mm cell against water as the reference.

To determine non-specific absorptions, put another 10 ml of the test solution into a 50 ml volumetric flask, fill the flask up to the mark with water, and measure the absorption in the same order.

Duplicate determinations shall be carried out.

E.6 Results

E.6.1 Calculation

The biuret content is given, as a percentage by mass, by Formula (E.3):

$$w_A = \frac{(E_S - E_B) \times F_D \times 250}{m_S \times 10 \times 1\,000} \qquad \text{(E.3)}$$

where

w_A is the content of biuret [% (*m*/*m*)];

E_S is the extinction of the sample;

E_B is the extinction of the blank test (reagent blank + sample blank);

m_S is the mass of sample used to prepare test solution (g);

F_D is the calibration factor (µg).

E.6.2 Expression of results

Express the result to the nearest 0,01 % (*m*/*m*).

E.7 Precision

See 5.2, 5.3, and Table E.1.

Table E.1 — Precision

Biuret content w_{Bi} % (*m*/*m*)	**Repeatability** *r* % (*m*/*m*)	**Reproducibility** *R* % (*m*/*m*)
0,1 to 0,8	0,01	0,04

E.8 Test report

The report shall include the following data as a minimum requirement:

a) type and description of tested product;

b) reference to this part of ISO 18611 (i.e. 18611-2);

c) sampling method used;

d) test result (see E.6);

e) deviations from the specified mode of operation, if any;

f) test date.

Annex F
(normative)

Determination of aldehyde content

F.1 General

This annex specifies the procedure for the determination of the content of free and bound aldehyde, calculated as formaldehyde, of AUS 40 with contents of aldehyde from 5 mg/kg to 100 mg/kg.

F.2 Principle

Formaldehyde forms in strong sulphuric acid solution with chromotropic acid a purple colour with absorption maximum at 565 nm. The colour complex is read spectrophotometrically at 565 nm and the aldehyde concentration is determined by reference to a calibration curve prepared from standard formaldehyde solutions.

NOTE The method specified in this Annex is based on ISO 22241-2.

F.3 Apparatus

F.3.1 Laboratory balance, resolution in reading 0,000 1 g.

F.3.2 Spectrophotometer, for use at 565 nm with 10 mm cell.

F.3.3 Volumetric flasks.

F.3.4 Pipettes.

F.4 Chemicals

F.4.1 Chemicals of analytical grade, shall be used in all tests.

F.4.2 Sulphuric acid, 96 % (*m/m*).

F.4.3 Chromotropic acid (4,5-dihydroxynaphthalene-2,7-disulphonic acid sodium salt or 4,5-dihydroxy-naphthalene-2,7-disulphonic acid disodium salt dihydrate), 3 % (*m/m*) in sulphuric acid of 15 % (*m/m*).

In order to make this solution, add 41 ml sulphuric acid to 410 ml of water while cooling the mixture and then add 15 g of chromotropic acid and mix them well.

NOTE If stored in a brown glass bottle, this solution is usable for at least 3 months.

F.4.4 Formaldehyde standard solution, prepared as follows:

— put 6,5 g to 7 g of formaldehyde solution having a concentration of 37 % (*m/m*) into a 500 ml volumetric flask, fill up the flask with water, and mix it well;

- — determine the formaldehyde content of the solution, for example, in accordance with ISO 9020 method;
- — dilute the solution to 1:1 000. Mark the exact value of the formaldehyde content to the flask (the formaldehyde content as determined in the previous step divided by 1 000).

F.5 Procedure

F.5.1 Preparation of the calibration curve

Into a series of six 50 ml volumetric flasks, transfer 0,2 ml, 0,5 ml, 1 ml, 2 ml, 5 ml, and 10 ml of the formaldehyde standard solution and add water to a total mixture volume of approx. 10 ml. Add, while stirring, 1 ml of chromotropic acid solution followed by gradual addition of 20 ml sulphuric acid during the course of 5 min. The temperature rise during the addition of sulphuric acid shall exceed 100 °C, which is necessary for the reaction to come to completion. Let the flask stand at ambient air for 15 min without any further cooling.

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all the reagents used for the measurement (see F.5.4).

After cooling to room temperature, fill the flask with water up to the mark and mix well. Carry out the photometric measurements with the spectrophotometer at a wavelength of about 565 nm using a 10 mm cell against water as the reference.

Subtract the extinction of the blank test from the extinction of the measured values and set up the calibration curve. In the concentration range, the curve shall be strictly linear.

F.5.2 Calculation of the calibration factor

Calculate the calibration factor according to Formula (F.1):

$$F = \frac{\sum_{i=1}^{6} m_{\mathrm{HCHO},i}}{\sum_{i=1}^{6} \left(E_{1,i} - E_2 \right)} \tag{F.1}$$

where

F is the calibration factor (ug);

$m_{\mathrm{HCHO},i}$ is the mass of the formaldehyde of the i-th sample (ug);

$E_{1,i}$ is the extinction of the i-th sample;

E_2 is the extinction of the blank test.

The determination of the calibration curve and the calibration factor shall be repeated on a yearly basis and shall be documented.

F.5.3 Check of the method

Every three months, the method shall be checked as follows.

Into a series of three 50 ml volumetric flasks, transfer 2 ml of the formaldehyde standard solution and add water to a total volume of approx. 10 ml. Follow the procedure described in F.5.4 and calculate the aldehyde content as shown in F.6.

Compare the findings with the content of the standard solution. If the deviation is less than or equal to 2 %, the method is ready to use. If the deviation is more than 2 %, repeat the check. If the deviation is more than 2 % again, the method shall not be used unless a new calibration curve is prepared.

F.5.4 Measuring of samples

Weigh, to the nearest 0,001 g, 0,5 g to 1,0 g of the test sample in a 50 mI volumetric flask and dilute it with water to a total mixture volume of approximately 10 ml. Add, with stirring, 1 ml of chromotropic acid solution followed by gradual addition of 20 ml sulphuric acid during the course of 5 min. The temperature rise during the addition of sulphuric acid shall exceed 100 °C, which is necessary for the reaction to come to completion. Let the flask stand at ambient air for 15 min without any further cooling.

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all the reagents used for the determination.

After cooling to room temperature, fill the flask with water up to the mark and mix well. Carry out the photometric measurements with the spectrophotometer at a wavelength of about 565 nm using a 10 mm cell against water as the reference.

F.6 Results

F.6.1 Calculation

The aldehyde content is given by Formula (F.2)

$$w_A = \frac{(E_S - E_B) \times F}{m_S} \tag{F.2}$$

where

w_A is the content of aldehyde (mg/kg);

E_S is the extinction of the sample;

E_B is the extinction of the blank test (reagent blank + sample blank);

m_S is the mass of sample used (g);

F is the calibration factor (ug).

F.6.2 Expression of results

Express the result to the nearest 1 mg/kg.

F.7 Precision

See 5.2, 5.3, and Table F.1.

Table F.1 — Precision

Content of aldehyde w_A mg/kg	**Repeatability** r mg/kg	**Reproducibility** R mg/kg
5 to 100	0,14	0,5x x
NOTE x is mean value.		

F.8 Test report

The report shall include the following data as a minimum requirement:

a) type and description of tested product;

b) reference to this part of ISO 18611 (i.e. 18611-2);

c) sampling method used;

d) test result (see F.6);

e) deviations from the specified mode of operation, if any;

f) test date.

Annex G
(normative)

Determination of insoluble matter content by gravimetric method

G.1 General

This annex specifies the procedure for determining the insoluble matter content in AUS 40 above 1 mg/kg.

G.2 Principle

The sample is filtered and the mass of the residue is determined by gravimetric analysis.

G.3 Apparatus

G.3.1 Filtration equipment for vacuum filtration, suitable for 47 mm or 50 mm diameter membrane filters.

G.3.2 Membrane filter, pore size 0,8 µm, cellulose mixed ester.

G.3.3 Petri dish with cover, suited to the fitting of membrane filters (e.g. 80 mm × 15 mm).

G.3.4 Flat-tipped tweezers.

G.3.5 Analytical balance, resolution 0,01 mg or better.

G.3.6 Balance, resolution 0,01 g or better.

G.3.7 Glass beaker, nominal volume 400 ml (preferably high shaped with volumetric separation).

G.3.8 Drying oven, capable of maintaining a temperature of 105 °C with a precision of ±2 °C.

G.3.9 Desiccator filled with a drying agent.

NOTE Sulphuric acid or calcium chloride are not suitable as drying agents.

G.3.10 Standard laboratory glass.

G.4 Chemicals

G.4.1 De-ionized water, conductivity less than 0,1 mS/m, according to ISO 3696, grade 2.

G.5 Procedure

The sample shall be completely dissolved and free from any urea crystals. If required, the sample shall be warmed before being further processed to ≤40 °C.

The filter for use in the test shall be washed with water in advance. To do this, a filter shall be moistened in approximately 100 ml water in the vacuum filtration unit and the water shall be sucked through the filter. The filter shall then be dried to a mass consistency in the drying chamber and shall be stored in a Petri dish (one filter per Petri dish) in the desiccator. The membrane filters shall be weighed to 0,01 mg precision immediately before an analysis is made.

The filters shall always stay in the Petri dish for weighing.

The sample shall be shaken thoroughly to be homogeneous. Immediately after this, about 100 ml to 150 ml of the sample shall be placed in a dry, calibrated 400 ml glass beaker, weighed to 0,01 g precision, and 200 ml of water added. The sample shall not be pipetted for weighing.

The filtration equipment shall be set up using the prepared membrane filter. The filter shall be moistened with a little water (1 ml to 2 ml) without applying vacuum. The prepared sample shall be placed in the filtration vessel and the vacuum shall be arranged so that the sample is drawn swiftly through the filter.

The glass beaker shall be rinsed with five portions of water each of approximately 30 ml to 50 ml. The rinsing solution shall also be passed through the filter (sample vessel of the filtration equipment shall also be rinsed). The sample shall have completely run through the filter before the start of the first rinse (allow the filter to dry briefly).

The filtering equipment shall be dismantled and the filter dried at 105 °C until constancy of the mass is achieved. After cooling to room temperature in a desiccator, the filter shall be weighed to 0,01 mg resolution.

It shall be ensured that the filter is washed completely clean of all urea remnants. If it is found that filters are still stuck to the glass floor of the Petri dish, this indicates that the washing has not been adequate. These filters shall be disposed of and the analysis shall be repeated.

G.6 Results

G.6.1 Calculation

$$w_{\text{ins}} = \frac{(m_{\text{FR}} - m_{\text{FL}})}{m_{\text{S}}} \times 1\,000 \qquad \text{(G.1)}$$

where

w_{ins} is the content of insoluble matters (mg/kg);

m_{FL} is the mass of the dried empty filter (mg);

m_{FR} is the mass of dried filter with sample deposit (mg);

m_{S} is the sample mass (g).

G.6.2 Expression of results

The mean value of the calculation is valid as the result. Should the difference between the separate values be more than 25 % of the higher value, the determination shall be repeated. The result shall be rounded off as follows:

- <10 mg/kg to the nearest 0,1 mg/kg;
- ≥10 mg/kg to the nearest 1 mg/kg.

G.7 Precision

See 5.2, 5.3, and Table G.1.

Table G.1 — Precision

Insoluble matter content w_{ins} mg/kg	**Repeatability** r mg/kg	**Reproducibility** R mg/kg
>1	0,23 × x	0,38 × x
NOTE x is mean value.		

G.8 Test report

The test report shall contain the following data as a minimum:

a) type and distinguishing characteristics of the tested products;

b) reference to this part of ISO 18611 (i.e. 18611-2);

c) the sampling process applied;

d) the test result (see G.6);

e) deviation from the specified mode of operation, if any;

f) test date.

Annex H
(normative)

Determination of phosphate content by photometric method

H.1 General

This annex specifies the procedure for the determination of the content of total phosphorus as phosphate in AUS 40 with contents from 0,05 mg/kg to 10 mg/kg. Expansion of the measurement range is possible by varying the sample amount.

H.2 Principle

The sample is evaporated and incinerated with calcium carbonate to mineralize the phosphorus compounds.

After this treatment, the sample is transformed from phosphate to orthophosphate by means of hydrochloric acid.

Orthophosphate-ions react in acid solvent with molybdate and antimony-ions to an antimony-phosphomolybdate complex.

The reduction of this complex with ascorbic acid leads to an intensive coloured molybden blue-complex. The intensity of the colour indicates the concentration of orthophosphate-ions.

H.3 Apparatus

H.3.1 **Analytical balance**, resolution 0,01 g or better.

H.3.2 **Incineration dish** (platinum or quartz glass).

H.3.3 **Heating plate** or **sand bath**.

H.3.4 **Muffle furnace** (700 °C).

H.3.5 **Spectrophotometer** (for use at 800 nm with a 1 cm cuvette).

H.3.6 **Cells**, made of optical glass, 1 cm.

H.3.7 **Graduated flasks**.

H.3.8 **Bulb pipettes**.

H.4 Chemicals

H.4.1 **De-ionized water**, conductivity less than 0,1 mS/m, according to ISO 3696, grade 2.

H.4.2 **Calcium carbonate**, analytical grade.

H.4.3 Hydrochloric acid, having a concentration of 25 % (*m*/*m*).

H.4.4 Sulphuric acid, having a concentration of 96 % (*m*/*m*).

H.4.5 Ascorbic acid, analytical grade.

H.4.6 Ammonium heptamolybdate tetrahydrate, analytical grade.

H.4.7 Potassium antimony(III) oxytartrate hemihydrate.

H.4.8 Ascorbic acid solution, having a concentration of 100 g/l.

Dissolve 10 g ascorbic acid (see H.4.5) in 100 ml water (see H.4.1).

NOTE The solution is durable for two weeks if it is placed in the refrigerator. The solution can be used as long as it is colourless.

H.4.9 Molybdate solution.

Dissolve 13 g ammonium heptamolybdate tetrahydrate $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ (see H.4.6) in 250 ml water (see H.4.1). Put 150 ml sulphuric acid (see H.4.4) while cooling and stirring into the first solution.

After this, dissolve 0,35 g potassium antimony(III) oxytartrate hemihydrate $K(SbO)\ C_4H_4O_6\cdot 1/2H_2O$ (see H.4.7) in 100 ml water (see H.4.1) and mix it into the sulphuric acid molybdate solution.

NOTE The whole solution is durable for two months if stored in a brown glass bottle.

H.4.10 Potassium hydrogen phosphate, KH_2PO_4, analytical grade, dried at 105 °C.

H.4.11 Phosphate stock solution, having a concentration of 200 mg/l.

Weigh in 286,6 mg potassium hydrogen phosphate (H.4.10) and put it into a 1 l graduated flask (H.3.7) and dissolve it with water (see H.4.1). Add 2 ml sulphuric acid (see H.4.4), fill the flask with water (see H.4.1) up to the calibrating mark and homogenize.

NOTE The solution is durable in a closed glass bottle for a minimum of three months.

H.4.12 Phosphate stock solution, having a concentration of 2 mg/l.

From the stock solution (see H.4.11), generate a second stock solution of 2 mg/l by dilution with water (see H.4.1) to 1:100.

H.5 Procedure

H.5.1 Preparation of the calibration curve

Put 1 ml, 2 ml, 5 ml, and 10 ml stock solutions (see H.4.12) (corresponding 2 µg, 4 µg, 10 µg, and 20 µg phosphate) separately into a 50 ml graduated flask (see H.3.7) and dilute them with water (see H.4.1) to 40 ml. Every concentration shall be measured 10 times in accordance with H.5.7.

H.5.2 Calculation of the calibration factor

Calculate the calibration factor according to Formula (H.1):

$$C = \frac{\sum_{i=1}^{4} m_{\text{phosphate},\, i}}{\sum_{i=1}^{4} \left(E_{1,i} - E_2 \right)} \tag{H.1}$$

where

C is the calibration factor (µg);

$m_{\text{phosphate},\, i}$ is the mass of phosphate of the *i*-th sample (µg);

$E_{1,i}$ is the extinction of the *i*-th sample;

E_2 is the extinction of the blank test.

H.5.3 Process examination

H.5.3.1 Purpose

It shall be checked whether the method delivers correct results.

H.5.3.2 Principle

The stock solution (see H.4.12) will be determined like a routine sample. The phosphate amount of the stock solution is the measured value that shall be proved.

H.5.3.3 Execution

Pipette the phosphate stock solution of 5 ml and put it into a 50 ml graduated flask (see H.3.7), analyse it (see H.5.7), and calculate the content of phosphate (see H.6). Repeat this procedure three times.

The method is deemed valid if the measured values differ less than ±2 % related to the given phosphate amounts.

H.5.3.4 Frequency

The examination of the method should be done once every three months.

H.5.4 Examination of the calibration curve

H.5.4.1 Purpose

It shall be checked in fixed intervals if the slope of the calibration curve is correct.

H.5.4.2 Principle

Phosphate stock solutions will be determined and the results will be compared with the values from the calibrating curve.

H.5.4.3 Execution

Similar to H.5.1, a minimum of three concentrations in measurement range of the calibrating curve should be determined three times.

The calibration curve is deemed valid if the mean value of the calculated values differs less than ±2 % related to the given point of the calibration curve. If the difference is greater, repeat the procedure.

If the greater differences are confirmed, the analysing method for the determination of phosphate contents cannot be used until a new calibrating curve (see H.5.1) is established.

H.5.4.4 Frequency

The examination of the calibrating curve should be done minimum once every three years.

H.5.5 Sample preparation

The sample shall be completely dissolved and shall be without any urea crystals. If required, the sample can be heated to ≤40 C.

H.5.6 Decomposition

Weigh in approximately 100 g (record the mass) of the prepared sample (see H.5.5) into an incineration dish (see H.3.2) and add 100 mg calcium carbonate (see H.4.2). Put the prepared sample on the heating plate and dry it slowly. Afterwards, incinerate the sample in the muffle furnace at 700 °C (see H.3.4) until the sample is completely decomposed. Cool the sample and add 1 ml hydrochloric acid (see H.4.3) and 20 ml to 30 ml water (see H.4.1) into the dish. Boil it until the residues are dissolved and the CO_2 is removed. Transfer the solution completely into a 100 ml graduated flask (see H.3.7), fill it with water (see H.4.1) to the calibration mark, and homogenize it.

H.5.7 Photometric determination

Pipette (using a bulb pipette; see H.3.8) an exact volume from the solution (see H.5.6) into a 50 ml graduated flask (see H.3.7). Use a ≤40 ml sample solution. If less than 40 ml is used, dilute it with water (see H.4.1) until 40 ml is reached.

While stirring, add 1 ml ascorbic acid solution (H.4.8) and 2 ml molybdate solution (see H.4.9), fill the flask with water (see H.4.1) up to the calibrating mark, and homogenize. The blank test shall be made in the same way but without sample solution.

After 10 min to 30 min, determine the extinction of the sample and the blank test with the photometer (see H.3.5) at 800 nm.

H.6 Results

H.6.1 Calculation

The phosphate content is calculated by Formula (H.2):

$$w_P = \frac{(E_S - E_B) \times C \times V_S \times F_1}{V \times F_2 \times m_S} \qquad \text{(H.2)}$$

where

w_P is the content of phosphate (mg/kg);

E_S is the sample extinction;

E_B is the blank test extinction;

C is the calibration factor (µg);

V_S is the volume of the decomposed solution (ml);

F_1 is 1 000 (conversion factor from kg to g);

V is the volume used for the photometrical determination (ml);

F_2 is 1 000 (conversion factor from mg to µg);

m_S is the mass of urea solution (g).

H.6.2 Expression of results

Express the result to the nearest 0,01 mg/kg.

H.7 Precision

See 5.2, 5.3, and Table H.1.

Table H.1 — Precision

Phosphate content w_P mg/kg	**Repeatability** r mg/kg	**Reproducibility** R mg/kg
0,1 to 1	0,02	0,03

H.8 Test report

The report should include the following data as a minimum requirement:

a) type and description of tested product;

b) reference to this part of ISO 18611 (i.e. 18611-2);

c) sampling method used;

d) test result (see H.6);

e) deviations from the specified mode of operation, if any;

f) test date.

Annex I

(normative)

Determination of trace element content (Ca, Fe, K, Mg, Na) by ICP-OES method

I.1 General

This annex specifies the procedure for the determination of calcium, chromium, iron, potassium, magnesium, and sodium in AUS 40.

I.2 Principle

The trace element content is determined by using an **I**nductively **C**oupled **P**lasma-**O**ptical **E**mission **S**pectrometer (ICP-OES). This method needs reference curves for each element.

Two different analytical procedures of sampling preparation can be optionally applied:

a) Drying and incineration (ashing) of the urea solution (using a hot plate or using a microwave muffle furnace). This procedure needs more time to do, but is a little better in sensitivity.

b) Direct determination, which includes a dilution of 1:5 with water (base procedure).

I.3 Apparatus

I.3.1 Apparatus for the incineration procedure

I.3.1.1 Volumetric flask, with nominal volume of 100 ml, class A or B.

Plastic flasks or volumetric flasks made of quartz glass may be used. Borosilicate glass flasks shall not be used.

I.3.1.2 Muffle furnace.

The thermostat should preferably allow the possibility for programming temperature ramps, and the muffle furnace should be provided with a gas exhaust. If the gas exhaust is not available, a gas burner is required in addition.

I.3.1.3 Bunsen burner, if applicable.

Caution: In case of excessive incineration temperatures, alkali elements will vaporize.

I.3.1.4 Hot plate, with a reachable surface temperature of 500 °C.

Alternatively, a microwave muffle furnace with a gas exhaust and quartz glass plate above the sample can be used.

I.3.1.5 Analytical balance, resolution 0,1 g or better.

I.3.1.6 Quartz glass dishes, nominal volume of 100 ml.

Platinum dishes shall not be used, as they will lead to reduced results.

I.3.2 Apparatus for direct determination

I.3.2.1 Volumetric flask, with nominal volume of 100 ml, class A or B.

Plastic flasks or volumetric flasks made from quartz glass can be used. Borosilicate glass flasks shall not be used.

I.3.2.2 Fixed volume pipettes, of 50 µl, 100 µl, 200 µl, 500 µl, 1 000 µl, 10 ml, or variable piston pipettes.

The pipettes shall be calibrated.

I.3.3 Measuring instrument, ICP-OES

A nebulizer system shall be used which can convert even high salt loads into an aerosol (cross flow, V-groove, or similar). Humidification of the ICP gas (argon) is recommended.

Where autosamplers are used, the vessels, the needle, and the supply hoses to the spectrometer shall be manufactured from polymer material (HDPE, PP, PTFE, etc.). Borosilicate glass flasks shall not be used.

I.4 Chemicals

I.4.1 General

Unless otherwise indicated, chemicals, at least corresponding to the purity level “analytical grade”, and distilled/de-ionized water (conductivity of less than 0,5 mS/m, according to ISO 3696, grade 3) shall be used.

The following procedures shall be conducted with only one kind of acid.

I.4.2 Chemicals for the incineration procedure

These include the following:

- nitric acid of min. 65 % (*m/m*), alternative hydrochloric acid of max. 37 % (*m/m*);
- ICP standard solution, 1,000 g/l per element;

Commercially available certified standard ICP solutions may be used.

I.4.3 Chemicals for direct determination

These include the following:

- 40 % urea solution, made of urea “for biological purposes” and water, by weighing both components;
- components nitric acid of min. 65 % (*m/m*), alternative hydrochloric acid of max. 37 % (*m/m*);
- ICP standard solution, 1,000 g/l per element;

  Commercially available certified standard ICP solutions can be used.
- multi-element standard solution, 10 mg/l per element: pipette 1 000 µl from each of the ICP standard solutions into a 100 ml volumetric flask.

Fill up the flask with water and shake. The solution shall be prepared fresh each working day.

I.5 Procedure

I.5.1 Interferences

Result reductions may occur due to splashing of sample material during incineration or excessive temperatures during pre-incineration over the Bunsen burner or in the muffle furnace (in particular in case of Na and K); result increases may occur due to transferred mineral constituents (e.g. transferred furnace insulating material). Relevant activities for preventing such errors shall be taken.

Incineration procedure: Element P cannot be determined, as polyphosphates are formed during incineration which cannot be dissolved later.

Some elements can be disturbed during direct determination due to the matrix containing carbon. Interference can also arise due to the atomizing system used. In case of problems relating to instruments during direct determination, the sample shall be processed in accordance with I.2 b).

Traces of the elements to be determined may be present on the inside of plastic vessels (sample bottles, volumetric flasks, etc.); the insides shall therefore always be cleaned with acid (HCl, HNO_3) before use.

I.5.2 Sample preparation

I.5.2.1 Sample preparation for the incineration procedure

Weigh 100 g of the sample with a tolerance of 0,1 g into a quartz dish. Slowly concentrate the solution on the hot plate by evaporation until it is dry. Once the sample is so dry that it cannot splash, fume off the sample in the muffle furnace, with temperatures starting at 350 °C, increasing to 700 °C within two hours, until the sample is completely fumed off. Hold the temperature of 700 °C for at least 30 min.

If no temperature-controlled muffle furnace with a gas exhaust is available, the sample should be largely fumed off on the flame (fume hood) and only then ignited at 700 °C in the muffle furnace.

If using a microwave muffle furnace for the incineration procedure, the following temperature sequence shall be used:

1) start at room temperature;
2) ramp up to 200 °C within 30 min;
3) hold at 200 °C for 10 min;
4) ramp up to 700 °C within 120 min;
5) hold at 700 °C for at least 30 min.

Allow the sample to cool to room temperature and take up the residue using 5 ml of nitric acid (or hydrochloric acid) and approximately 20 ml of water while heating. Transfer the solution completely into a 100 ml volumetric flask. Allow the volumetric flask to cool to room temperature, then fill it up with water and shake it.

I.5.2.2 Sample preparation for direct determination

When using suitable nebulizers and spectrometers with sufficiently low detection limits for the individual elements, the samples shall be prepared as follows:

- weigh 20 g of the sample into a 100 ml volumetric flask with a tolerance of 0,01 g;
- add some 50 ml of water and 5 ml of nitric acid (or hydrochloric acid) in this sequence;
- fill up the volumetric flask with water and homogenize the solution.

I.5.3 Generation of the reference curve

The frequency of the acquisition of the reference curve depends on the spectrometer used (in accordance with the instrument manufacturer's specifications and guidelines). To check the reference curve and correct its drift, the lowest and highest standard should be measured on each working day. The element concentrations as specified in Tables I.1 and I.2 are recommended.

The intensities of the individual elements are converted using the reference curve (normally with the assistance of ICP computer software).

See Table I.1 for the incineration procedure.

Table I.1

Solution	Content per element mg/l	Acid addition ml/l
0	0	50
1	0,010	
2	0,030	
3	0,100	
4	0,300	
5	1,000	
6	5,000	

See Table I.2 for direct determination.

Table I.2

Solution	Content per element mg/l	Acid addition ml/l	Urea solution 40 % ml/l
0	0	50	200
1	0,010		
2	0,020		
3	0,050		
4	0,100		
5	0,200		
6	0,500		
7	0,800		

I.5.4 Determination

For emission measurements, the wavelengths specified in Table I.3 shall be used.

Table I.3

Element	**Wavelength** Nm
Ca	396,85 or 317,93 or 393,37
Fe	259,94 or 239,56
K	766,49
Mg	279,55 or 285,21
Na	588,99 or 589,59

Measurements of each prepared solution shall be repeated at least three times, whereby a sufficient rinsing time shall be ensured after each sample change. For intermediate rinses, a 3 % (*m/m*) nitric acid (or hydrochloric acid) solution is suggested.

I.6 Results

I.6.1 Calculation

If the measured values are output in mg/l, convert them afterwards into the corresponding sample-relating contents (mg/kg).

I.6.2 Expression of results

The result per element is defined as the arithmetic mean of all determinations. Indicate the result precisely to two significant digits.

I.7 Precision

See 5.2 and 5.3.

The precision values specified in Table I.4 cover both procedures.

Table I.4

Element	**Repeatability** r mg/kg	**Reproducibility** R mg/kg
Ca	0,02	0,1 × x
Fe	0,01	0,3 × x
Mg	0,02	0,3 × x
Na	0,03	0,5 × x
K	0,03	0,5 × x
NOTE x is mean value.		

I.8 Test report

The report shall include the following data as a minimum requirement:

a) type and description of tested product;

b) reference to this part of ISO 18611 (i.e. 18611-2);

c) sampling method used;

d) test result (see I.6);

e) deviations from the specified mode of operation, if any;

f) test date.

Annex J
(informative)

Determination of identity by FTIR spectrometry method

J.1 General

This annex specifies the procedure for the determination of identity of AUS 40 samples. All mixtures of urea with a concentration greater than 10 % (*m/m*) urea in water will produce IR spectra with the same characteristic peaks.

Using this method, the AUS 40 sample is compared with a known sample and determined to be identical or not identical. Differences in the concentration or contamination should not be determined using this method.

J.2 Principle

When light is transmitted through a thin layer of urea solution, infrared light is absorbed characteristically; the recorded spectrum allows the identification of the urea. Alternatively, any suitable attenuated total reflection (ATR) method can be used.

J.3 Apparatus

J.3.1 Fourier transform infrared spectrometer (FTIR) or any IR spectrometer capable of recording spectra in the wavenumber range of 600 cm^{-1} to 4 000 cm^{-1}; the selected resolution should be 4 cm^{-1} or better.

J.3.2 Optical cell suitable for aqueous solutions, e.g. KRS5 (TlBr/TlJ), ZnSe, etc., layer thickness approximately 100 µm. Alternatively, any ATR unit suitable for liquids can be used.

CAUTION — Attention is drawn to the extremely toxic characteristics of KRS5 window materials.

J.4 Procedure

Fill a transmission cell with the sample to be analysed without air bubbles. Place the cell in the beam path of the FTIR spectrometer so that the IR absorption spectrum can be recorded. Alternatively, the sample can be placed on an ATR crystal.

Compare visually the spectrum obtained with the reference spectrum of a known 40 % (*m/m*) urea solution.

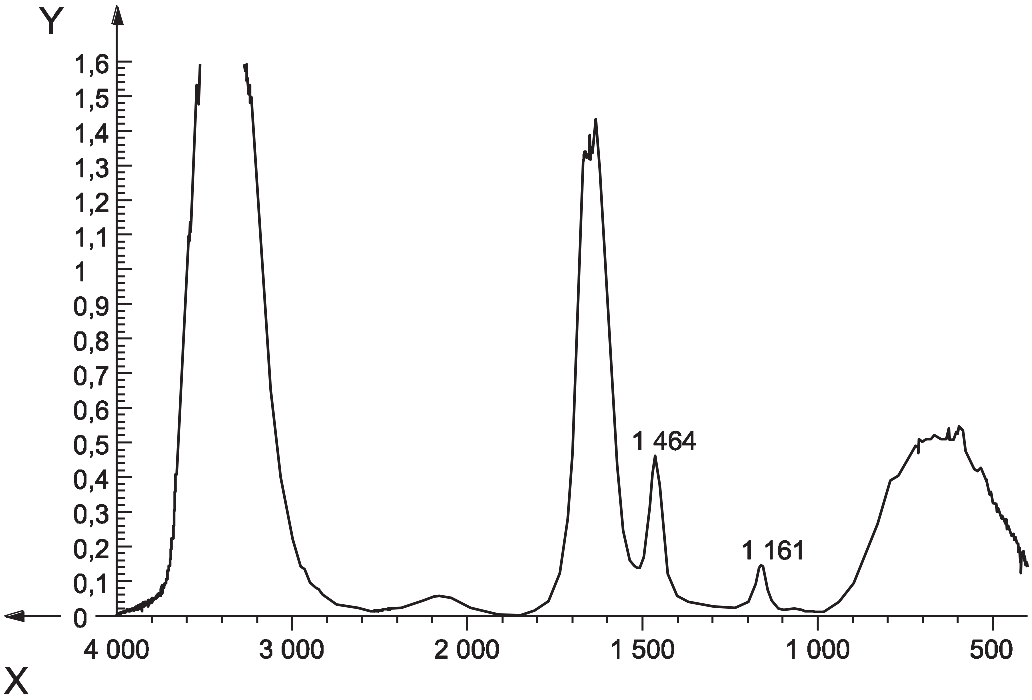
J.5 Expression of results

As a result of the determination based on the peak wave number, one of the statements below should be made:

— **Yes** for “identical to the reference”;

— **No** for “not identical to the reference”.

J.6 Examples

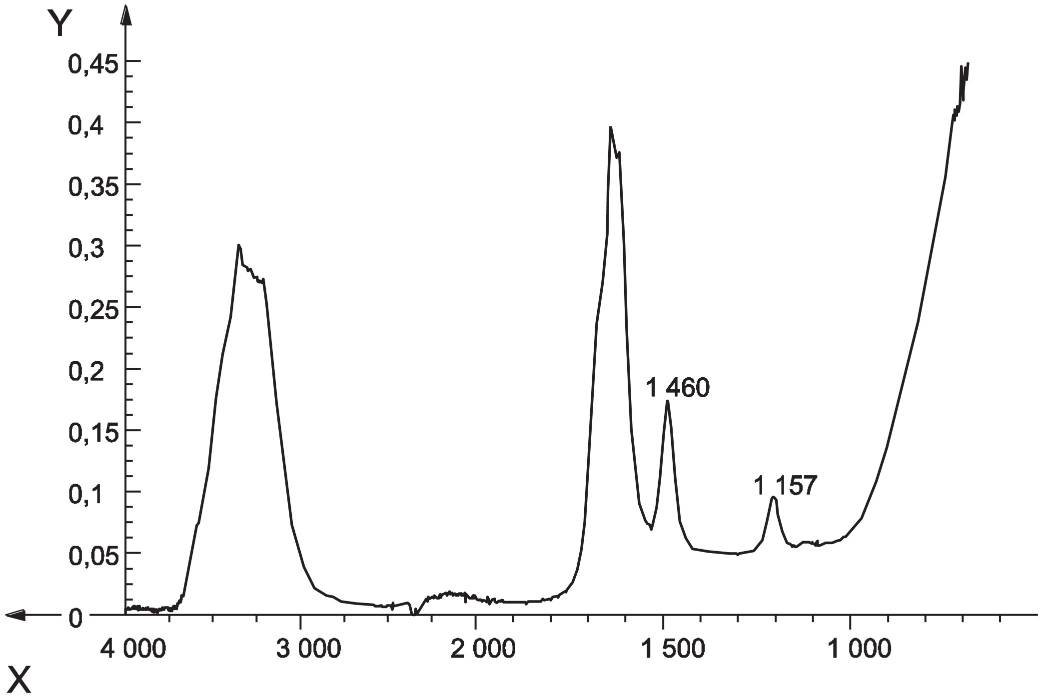


Key

X wave number (cm^{-1})

Y absorption

Figure J.1 — Example of reference spectra — urea in water, transmission technique



Key

X wave number (cm^{-1})

Y absorption

Figure J.2 — Example of reference spectra — urea in water, ATR technique

Annex K
(informative)

Precision of test methods

Characteristics	Unit	Repeatability *r*	Reproducibility *R*
Urea content (total nitrogen)	% (*m*/*m*)	0,4	1,0
Urea content (refractive index)	% (*m*/*m*)	0,1	1,0
Refractive index	—	0,000 1	0,001
Density according to ISO 3675	kg/m^3	0,5	1,2
Density according to ISO 12185	kg/m^3	0,2	0,5
Alkalinity as NH_3 (Free Ammonia)	% (*m*/*m*)	0,01	0,2 × *x*
Biuret	% (*m*/*m*)	0,01	0,04
Aldehyde	mg/kg	0,14	0,5 × *x*
Insolubles	mg/kg	0,23 × *x*	0,38 × *x*
Phosphate (PO_4)	mg/kg	0,02	0,03
Calcium	mg/kg	0,02	0,1 × *x*
Iron	mg/kg	0,01	0,3 × *x*
Magnesium	mg/kg	0,02	0,3 × *x*
Sodium	mg/kg	0,03	0,5 × *x*
Potassium	mg/kg	0,03	0,5 × *x*

NOTE 1 *x* is mean value.

NOTE 2 The precision of the test methods for the determination of the density was taken from the existing ISO 3675 and ISO 12185.

NOTE 3 The precision of all other test methods referenced in this part of ISO 18611 was obtained in an interlaboratory test programme conducted in the year 2004 in conjunction with the establishment of ISO 22241. Eighteen laboratories in Austria, Germany, and the Netherlands participating. The data obtained were evaluated according to ISO 4259.

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[3] ISO 11402, *Phenolic, amino and condensation resins — Determination of free-formaldehyde content*

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