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.

The UK participation in its preparation was entrusted to Technical Committee SME/32/-/2, Ships and marine technology - Maritime environment protection.

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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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*

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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.

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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.

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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.

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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.

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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 from[1)];

— container/location from which the sample was taken[1)];

— date and time of sampling[1)].

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 from[1)];

— container/location from which the sample was taken[1)];

— date and time of sampling[1)];

— name and signature of person who sampled

— date when the sample left the site.

1) Mandatory only in cases of dispute.

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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.

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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.

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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 %.

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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.

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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.

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