PUBLICLY AVAILABLE SPECIFICATION

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Diesel engines — NOx reduction agent AUS 32 —

Part 2: Test methods

Moteurs diesel — Agent AUS 32 de réduction des NOx — 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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

— an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;

— an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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.

ISO/PAS 22241-2 was prepared by Technical Committee ISO/TC 22, *Road vehicles*, Subcommittee SC 5, *Engine tests*.

ISO/PAS 22241 consists of the following parts, under the general title *Diesel engines — NOx reduction agent AUS 32*:

— *Part 1: Quality requirements*

— *Part 2: Test methods*

Diesel engines — NOx reduction agent AUS 32 —

Part 2: Test methods

This Publicly Available Specification specifies test methods required for determination of the quality characteristics of the NOx reduction agent AUS 32 (aqueous urea solution) specified in ISO 22241-1.

In the remaining parts of ISO 22241, the term “NOx reduction agent AUS 32” will be abbreviated to “AUS 32”.

1 Normative references

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

ISO 22241-1, *Diesel engines — NOx reduction agent AUS 32 — Part 1: Quality requirements*

ISO 3675, *Crude petroleum and liquid petroleum products — Laboratory determination of density or relative 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 12185, *Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method*

2 Specifications

Compliance with the limits specified in Table 1 of ISO 22241-1 shall be determined by the test methods specified in Annexes B through J of this Publicly Available Specification.

Determination of the density shall be conducted in accordance with ISO 3675 or ISO 12185.

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

3 Sampling

Samples shall be taken in accordance with Annex A.

4 Precision and dispute

4.1 General

All test methods referred to in this Publicly Available Specification 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 precision of the test methods, as determined by statistical examination in accordance with ISO 4259, is summarized in Annex K for all test methods. Additionally, each test method specified in this part of ISO PAS 22241 contains this information, too.

The statistical significance of the precision quoted in this Publicly Available Specification is generically defined in 4.2 and 4.3, in which the “xx (unit)” stands for the repeatability and reproducibility in question.

4.2 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 would, in the long run, in the normal and correct operation of the test method, exceed xx (unit) in only one case in 20.

4.3 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 32 throughout the supply chain after the shipment from the manufacturer's site to the AUS 32 containers of the vehicles.

A.2 Principle

The limits for the quality characteristics of AUS 32, which are specified in ISO 22241-1, 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 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 area, 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

1 000 ml wide neck bottles shall be used. Suited materials for these bottles are HD-Polyethylene, HD-Polypropylene, Polyfluorethylene, Polyvinylidenedifluoride and Poly(perfluoroalkoxy) PFA. In case of dispute, PFA bottles should be used.

Prior to the first use with AUS 32, the bottles shall be cleaned and finally rinsed with de-ionized water followed by AUS 32.

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 32.

A.5 Sampling

The locked wide neck bottle is opened, the cap is put down on a clean surface with the opening turned downward. After flushing the sampling pipe, the bottle is filled completely with AUS 32 from the container. The first filling is discarded, and the bottle is immediately re-filled with AUS 32 and locked tightly. The label is attached to the bottle (see A.4.2).

During the filling of the sample, maximum care shall be taken that neither dust nor liquid pollution gets 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 0 °C and 15 °C, and kept away from daylight to prevent growth of algae.

NOTE 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, a sufficient volume of sample material should be made available (recommendation: 1 litre), and at least double that required for complete verification of AUS 32 specifications. In case of dispute, a sufficient number of samples shall be taken according to ISO 4259.

A.7 Labelling

The label should contain the following information:

— product name;

— name of the company that owns the sample product; [1)]

— address where the sample has been taken; [1)]

— manufacturer of the sample product; [1)]

— batch or lot number;

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

— part of the container from where the sample has been taken; [1)]

— date and time of sampling;

— sample shipment date; [1)]

— name and signature of the person who took the sample. [1)]

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 32.

The method is applicable for the determination of the urea content in the range 30 % to 35 % (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 analyzer**, based on combustion methods.

B.3.2 **Analytical balance**. The accuracy of the balance is a function of the analyzer used and the required weighed portions. Resolution should be 0,1 % or better of the weighed portion.

B.3.3 Auxiliary contrivances for sample preparation, for example:

— **tweezers** with a blunt tip;

— **micro-spatula** with a flattened tip;

— **pipette**.

NOTE The pipette is required 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 from 10 µl to 1 000 µl or single-trip Pasteur pipettes with a fine tip may also be used.

B.3.4 Customary chemically resistant glass.

B.4 Chemicals

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

B.4.2 **Auxiliary combustion agent**, appropriate for use with the selected nitrogen analyzer.

The following materials are merely examples. Other or similar materials may 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 or 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 standards include: ethylenediamine tetraacetic acid (EDTA), nicotinic acid amide.

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

NOTE Liquid standards (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 analyzer, 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 may 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 Publicly Available Specification. Rather, operation should be based on the respective operation manual.

B.5.2 Reference curve

Perform calibration as required for the specific type of analyzer and according to the respective operation manuals (for example, after replacement of the combustion tube, reagent or similar). Weigh in an appropriate amount of standard substance repeatedly as appropriate for the respective type of apparatus to obtain a reference curve.

Perform measurement as described in B.5.4.

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

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

Frequency of inspection is a function of the analyzer used.

Perform measurements as described in B.5.4.

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 three-fold amount of combustion agent (for example, non-nitrogenous cellulose), and additional binders (for example, magnesium oxide) as required.

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

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

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 of the nitrogen determination:

$$w_U = 2{,}143\,8 \times (w_N - F \times w_{Bi})$$

where

w_U is the urea content [% (m/m)];

w_N is the nitrogen content [% (m/m)] (to the nearest 0,01 %);

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

F is the factor for converting the biuret content to nitrogen (0,407 6).

B.6.2 Expression of results

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

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

B.7 Precision

B.7.1 Repeatability, *r*

(see 4.2)

$r = 0{,}4$ % (m/m).

B.7.2 Reproducibility, *R*

(see 4.3)

$R = 1{,}0$ % (m/m).

B.8 Test report

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

— type and description of tested product;

— reference to this Publicly Available Specification;

— sampling method used;

— test result (see B.6);

— deviations from the specified mode of operation, if any;

— 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 32. The test method is applicable to liquids having refractive indices in the range 1,33 to 1,39 and at temperatures of 20 °C to 30 °C.

Based on the measurement of refractive index the method is used for determining the content of urea in the range 30 % to 35 % (m/m).

C.2 Principle

Measurement is based on the dependence of 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 1,390 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 < 0,1 mS/m according to ISO 3696 grade 2.

C.4.2 **Urea**, crystalline, with biuret content < 0,1 % (m/m).
Prior to weighing the urea to draw the reference curve, it shall be dried for 2 hours at 105 °C.

C.4.3 **Urea test solution** 32,5 % (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 ten-fold measurement.

The solution shall be kept air-tight in the refrigerator and should be used within four (4) weeks maximum.