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**Diesel engines — NO<sub>x</sub> reduction agent  
AUS 32 —**

**Part 2:  
Test methods**

*Moteurs diesel — Agent AUS 32 de réduction des NO<sub>x</sub> —*

*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](http://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](http://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 voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

This document was prepared by Technical Committee ISO/TC 22, *Road vehicles*, Subcommittee SC 34, *Propulsion, powertrain and powertrain fluids*.

This second edition cancels and replaces the first edition (ISO 22241-2:2006), which has been technically revised. It also incorporates the Technical Corrigendum ISO 22241-2:2006/Cor. 1:2008. The main changes compared to the previous edition are as follows:

- Major revisions to test methods of [Annex C](#) and [Annex I](#),
- Precision values for all test methods were revised,
- [Annex K](#) was updated.

A list of all parts in the ISO 22241 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

# Diesel engines — NO<sub>x</sub> reduction agent AUS 32 —

## Part 2: Test methods

**WARNING** — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all the safety issues associated with its use. It is the responsibility of users of this document to respond appropriately to ensure the safety and health of personnel prior to application of the document.

### 1 Scope

This document specifies test methods required for determination of the quality and chemical characteristics of NO<sub>x</sub> reduction agent AUS 32 (aqueous urea solution) as specified in ISO 22241-1.

### 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 4259 (all parts), *Petroleum and related products — Precision of measurement methods and results*

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

ISO 17034, *General requirements for the competence of reference material producers*

ISO 22241-1, *Diesel engines — NO<sub>x</sub> reduction agent AUS 32 — Part 1: Quality requirements*

### 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 22241-1 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

#### 3.1

##### certified reference material

##### CRM

substance or material used to check the quality and metrological traceability of products, to validate analytical measurement methods or for the calibration of instruments

### 4 Specifications

Conformance with the limits specified in ISO 22241-1 shall be determined by the test methods specified in [Annexes B](#) through [I](#) of this document. If necessary, the identity of the product can be determined as specified in [Annex J](#).

## 5 Sampling

Samples shall be taken in accordance with [Annex A](#).

## 6 Precision and dispute

Each of the test methods specified in [Annex B](#) through [Annex I](#) include a precision statement using guidance from ISO 4259 (all parts). In cases of dispute, the procedures described in ISO 4259 (all parts) shall be used for resolving the dispute, and interpretation of the results based on the test method precision shall be used.

For the convenience of the user, the respective precision data are summarized in Table K.1.

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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 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.

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

#### A.3 Possible contaminants

During the sampling process, foreign matter can 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.

1 000-ml wide neck bottles shall be used. Suited materials for these bottles are high density polyethylene (HDPE), high density polypropylene (HDPP), polyvinylidene fluoride (PVDF) and perfluoroalkoxy alkane (PFA).

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

##### A.4.2 Labels.

Each bottle shall be labelled using labels of approximately 10 cm by 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 shall be opened and the cap shall be placed on a clean surface with the opening turned downward. After flushing the sampling pipe, the bottle shall be filled completely with AUS 32 from the container. The first filling shall be discarded, and the bottle shall immediately be re-filled with AUS 32 and closed tightly. The label shall be attached to the bottle (see A.4.2). During the filling of the sample, maximum 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 0 °C and 15 °C.

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 32 specifications. In case of dispute, a sufficient number of samples shall be taken according to ISO 4259 (all parts).

## A.7 Labelling

The label should contain the following information:

- product name;
- name of the company which owns the sample product<sup>1)</sup>;
- address where the sample was taken from<sup>1)</sup>;
- manufacturer of the sample product<sup>1)</sup>;
- batch or lot number;
- container from which the sample was taken<sup>1)</sup>;
- part of the container where the sample was taken from (sampling point)<sup>1)</sup>;
- date and time of sampling<sup>1)</sup>;
- sample shipment date<sup>1)</sup>;
- name and signature of the person who took the sample<sup>1)</sup>.

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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 32.

The method is applicable for the determination of the urea content in the range of 30 to 35 % (mass fraction).

#### 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**, 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 % of the weighed portion or better.

**B.3.3 Auxiliary devices for sample preparation**, for example:

- tweezers with a blunt tip;
- micro-spatula with a flattened tip;
- 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 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 Distilled or deionized water**, conductivity less than 0,1 mS/m, according to ISO 3696 grade 2.

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

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, 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 may 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<sub>2</sub>.

**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 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 document. Rather, operation is 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 three-fold 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 density shall be determined 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:

$$w_U = 2,1438 \times (w_N - F \times w_{Bi})$$

where

$w_U$  is the urea content (mass fraction, in %);

$w_N$  is the mean value of the nitrogen content (mass fraction, in %) to the nearest 0,01 %;

$w_{Bi}$  is the mean value of the biuret content (mass fraction, in %), determined according to [Annex E](#);

$F$  is the factor for converting the biuret content to nitrogen (0,4076).

### 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

### B.7.1 General

The precision evaluation programme with a matrix of only four samples of AUS 32 solutions with urea content in the range 31,09 % (mass fraction) to 35,12 % (mass fraction) did not conform to the requirements of ISO 4259 (all parts), and thus only an estimate of precision based upon inter-laboratory test results is given in [B.7.2](#), [B.7.3](#) and [Table B.1](#).

### B.7.2 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 the following in only one case in twenty.

$$r = 0,466 \% \text{ (mass fraction)}$$

### B.7.3 Reproducibility, $R$

The difference between two single and independent 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 the following value in only one case in twenty.

$$R = 1,053 \% \text{ (mass fraction)}$$

**Table B.1 — Precision (estimated)**

Urea content $w_U$ % (mass fraction)	Repeatability $r$ % (mass fraction)	Reproducibility $R$ % (mass fraction)
31,09 to 35,12	0,466	1,053

## B.8 Test report

The test report shall contain at least the following information:

- a) type and description of tested product;
- b) reference to this document, i.e. ISO 22241-2:2019;
- c) sampling method used;
- d) test result (see B.6);
- e) deviations from the specified mode of operation;
- f) any unusual features observed; and
- g) 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, relative to air, for AUS 32 at 20 °C, and at a reference wavelength at 589,3 nm ± 5 nm.

Based on the measurement of refractive index, the method shall be used for determining the concentration of urea in the range of a mass fraction of 30 % to 35 % using existing data.

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

#### 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 and wavelength.

The content is determined by comparison to the agreed upon mathematical relationship between AUS concentration and refraction index.

#### C.3 Apparatus

**C.3.1 Refractometer**, capable of measuring refractive index at a reference wavelength of 589,3 nm, with a measuring range of at least 1,330 00 to 1,390 00, and a resolution of 0,000 01, with means of controlling temperature to (20 ± 0,1) °C.

NOTE Different types of apparatus are available on the market. The resulting various resources and modes of operation are not an object of this document. Rather, operation is based on the respective operation manuals.

#### C.4 Chemicals

**C.4.1 Distilled or deionized water**, conductivity less than 0,5 mS/m according to ISO 3696 grade 3.

**C.4.2 Certified reference material**, fluid of known refractive index, prepared in accordance with ISO 17034 and traceable through an unbroken chain of calibrations to a national measurement institute (NMI). For purposes of this standard, CRMs should have an uncertainty of ±0,000 05 nD or better.

#### C.5 Procedure

##### C.5.1 General

Each test sample should be fully dissolved and free from urea crystals. They may be heated to <40 °C as required prior to further processing. Care should be taken to avoid heating solutions above 40 °C for any time longer than is required to bring the crystals into solution.

### C.5.2 Refractometer calibration and verification

Daily, the refractometer shall be zero-set to distilled or deionized water at  $(20 \pm 0,1)$  °C in accordance with the instructions provided by the refractometer manufacturer.

Weekly, the refractometer shall be verified using distilled or deionized water and at least one CRM with a value greater than or equal to 1,382 7 nD20. If the refractive index of any CRM, as read on the refractometer, deviates by more than 0,000 05 nD20 from the certified value, then the refractometer shall be recalibrated according to the manufacturer's instructions, using a series of CRMs.

Monthly, the refractometer calibration shall be verified using distilled or deionized water and a series of CRMs (a minimum of three) spanning beyond the measuring range (1,381 4 to 1,384 3 nD20). If the refractive index of any CRM, as read on the refractometer, deviates by more than 0,000 05 nD20 from the certified value, then the refractometer shall be recalibrated according to the manufacturer's instructions, using a series of CRMs.

After completion of each verification/calibration, the refractometer shall be marked or tagged with the date and type of that verification/calibration.

If, after the calibration, the certified value for any CRM cannot be verified within  $\pm 0,000 05$  nD20, then the instrument shall be tagged as "Out of service" until instrument calibration can be properly verified.

### C.5.3 Sample preparation and measuring

If using a circulating water bath to maintain a constant temperature in the refractometer, 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 is maintained at  $(20 \pm 0,1)$  °C.

If the refractometer is equipped with a solid-state Peltier temperature control device, adjust the controls so that the refractometer is controlled to a temperature of  $(20 \pm 0,1)$  °C.

Measure the sample refractive index three times and then determine the arithmetic mean of the three refractive index values. If any two of the three measurements deviate by more than 0,000 05 nD20, the measurements shall be repeated.

## C.6 Calculation and expression of results

### C.6.1 Calculation

Urea content,  $w_U$ , shall be calculated from the refractive index determined in [C.5.3](#) using the following formula, which has a correlation of  $R^2 = 1,00$ , less the Biuret content as determined according to [Annex E](#).

$$w_U = (-742,747\ 88 \times (nD20)^2 + 2\ 669,653\ 61 \times (nD20) - 2\ 238,799\ 1) - B$$

where

$nD20$  represents the refractive index of the sample at 20 °C as determined in [C.5.3](#);

$B$  is the Biuret mass fraction (%) according to [Annex E](#).

NOTE Biuret has the same refractive index per unit of mass as urea.

### C.6.2 Expression of results

The result is defined as the arithmetic mean of the three refractive index measurements rounded to the nearest 0,000 1 nD20. For the urea content, the result shall be rounded to the nearest 0,1 % (mass fraction).