Draft of 8 May 2025

REGULATION

OF THE MINISTER FOR INDUSTRY[1)]

of [date]

on methods for testing the quality of liquid biofuels[2)]

Pursuant to Article 26(2) of the Act of 25 August 2006 on the system for monitoring and controlling fuel quality (Journal of Laws 2024, items 1209, 1940 and 1946; and 2025, item 303), the following is hereby decreed:

§ 1. Methods for testing the quality of the following liquid biofuels:

1) methyl ester as a stand-alone fuel used in vehicles, including agricultural tractors, as well as in non-road mobile machinery with diesel engines designed to burn this liquid biofuel;
2) diesel fuel containing 20 % methyl ester, used in vehicles, including agricultural tractors, as well as in non-road mobile machinery, with diesel engines designed to burn this liquid biofuel;
3) liquid biohydrocarbons constituting a stand-alone fuel and paraffinic diesel used in vehicles, including agricultural tractors, as well as in non-road mobile machinery, with diesel engines designed to burn this liquid biofuel;
4) motor gasoline containing between 70 % and 85 % bioethanol, used in vehicles equipped with spark-ignition engines designed to burn this liquid biofuel

— are set out in the Annex to the Regulation.

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[1] The Minister for Industry heads the government administration department for energy raw materials, based on § 1(2) of the Regulation of the Prime Minister of 19 December 2023 on the detailed scope of the activities of the Minister for Industry (Journal of Laws [Dziennik Ustaw], item 2727 and 2024, items 289 and 999).

[2])This Regulation was notified to the European Commission on ……….. under number ……/……/PL, pursuant to § 4 of the Cabinet Regulation of 23 December 2002 on the functioning of the national system for notification of standards and legal acts (Journal of Laws, item 2039, and 2004, item 597), which implements the provisions of Directive (EU) 2015/1535 of the European Parliament and of the Council of 9 September 2015 laying down a procedure for the provision of information in the field of technical regulations and of rules on Information Society services (harmonisation) (OJ L 241 of 17.9.2015, p. 1).

§ 2. The Regulation of the Minister for Energy of 14 October 2016 on testing of the quality of liquid biofuels (Journal of Laws, item 1802) is repealed.

§ 3. The Regulation shall enter into force 14 days after its publication.

THE MINISTER FOR INDUSTRY

Annex to the Regulation of the
Minister for Industry of [date]
(Journal of Laws, item)

METHODS FOR TESTING THE QUALITY OF LIQUID BIOFUELS

I. Methods for testing the quality of methyl ester as a stand-alone fuel for use in vehicles, including agricultural tractors, as well as in non-road mobile machinery with diesel engines designed to burn this liquid biofuel[3)]

1. The content of the fatty acid methyl esters is determined by gas chromatography using an internal reference, which consists in the separation of the mixture into individual components in the gaseous phase.

1.1. The method of determination of the content of fatty acid methyl esters (FAME), the reagents and materials used, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the Polish standard implementing the EN 14103 standard.

2. The density at 15 °C is determined using the following method:

1) oscillation, by inserting a sample (of approximately 1 ml) into the measuring cell of an oscillating densimeter thermostated to maintain the reference temperature of 15 ºC, or
2) with a hydrometer, consisting in measuring the density of a test sample at a given temperature using a hydrometer immersed in the sample placed in a cylinder.

2.1. For the determination of density with the method referred to in subparagraph 2(1):

1) the method of determination, the reagents used, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the Polish standard implementing the EN ISO 12185 standard.
2) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

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[3] Drawn up based on the standard PN-EN 14214 Liquid petroleum products — Fatty acid methyl esters (FAME) for use in diesel engines and heating applications — Requirements and test methods.

2.2. For the determination of density with the method referred to in subparagraph 2(2):

1) the indication on the scale of the hydrometer is read, the temperature of the test sample between 20 °C and 60 °C is recorded and the measurement result in reference to the temperature of 15 °C is calculated using the conversion formula in Annex B to the PN-EN 14214 standard;

2) the method of determination, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN ISO 3675 standard.

3) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

3. Viscosity at 40 °C is determined using the method consisting in:

1) the measurement of the time necessary for a given volume of the test sample to flow under gravitational force through a calibrated glass capillary viscometer, under repeatable conditions, at a known and strictly controlled temperature; or

2) the introduction of the test sample into strictly temperature-controlled measuring chambers consisting of a pair of rotating, co-axial cylinders and an oscillating U-tube, followed by the determination of the dynamic viscosity on the basis of the equilibrium rotational speed of the internal cylinder and of the density on the basis of the frequency of the U-tube oscillations.

3.1. For the determination of viscosity with the method referred to in subparagraph 3(1):

1) viscosity is calculated by multiplying the measured flow time of constant fluid volume between the lines of the measuring vessel by a viscometer calibration constant;

2) the method of determination, the reagents used, the type of apparatus used and its preparation and calibration, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the Polish standard implementing the EN ISO 3104 standard;

3) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

3.2. For the determination of viscosity with the method referred to in subparagraph 3(2):

1) viscosity is calculated by dividing the measured dynamic viscosity by the measured density;

2) the method of determination, the reagents used, the type of apparatus used and its preparation and calibration, the method of calculation and presentation of the results,

and the method of drawing up the test report are laid down in the PN EN 16896 standard;

3) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

4. The flash point is determined:

1) through a fast equilibrium method, in a closed cup, by placing the test sample in the cup and heating it until vapour ignition is observed on the surface of the test sample, or

2) by means of the Pensky-Martens closed-cup test by placing the test sample in the cup and heating it whilst mixing continuously, until the source of ignition introduced through the opening in the cup lid causes the vapour on the surface of the test sample to ignite.

4.1. For the determination of the flash point with the method referred to in subparagraph 4(1):

1) 2 ml of the test sample is used for the test and a measuring instrument fitted with a temperature recording device is used;

2) the lowest temperature at which vapour ignition occurs on the surface of the test sample is taken as the flash point at ambient conditions;

3) the measured flash point of the test sample at ambient conditions is adjusted to standard atmospheric pressure;

4) flash point determination apparatus, equipped with a suitable thermal or ionising device for detecting ignition, is used;

5) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in Procedure B of the Polish standard implementing the EN ISO 3679 standard;

6) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

4.2. For the determination of the flash point with the method referred to in subparagraph 4(2):

1) the lowest temperature at which the application of the ignition source causes the vapours of the test sample to ignite and the flame to spread over the surface of the liquid is taken as the flash point at ambient conditions;

2) the measured flash point of the test sample at ambient conditions is adjusted to standard atmospheric pressure;

3) procedure C laid down in the PN-EN ISO 2719 standard is used;

4) flash point determination apparatus, equipped with a suitable thermal or ionising device for detecting ignition, is used;

5) the method of determination, the reagents used, the type of apparatus used and its preparation, handling of the sample, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN EN ISO 2719 standard;

6) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

5. The cetane number is determined using the following method:

1) engine test consisting in comparing the self-ignition properties of the tested fuel with the corresponding properties of reference fuel mixtures of known cetane numbers, using a test engine under standardised conditions, or

2) combustion in a constant volume chamber consisting in injecting a fuel sample into a compressed air charge in a constant volume chamber, detecting the start of injection and the beginning of its combustion by sensors for a specified number of cycles and determining the ignition delay time, or

3) combustion in a high-temperature and pressure-controlled constant volume chamber consisting in injecting the test fuel sample into a heated, compressed, synthetic air of required quality, generating a dynamic pressure wave due to the combustion of the test sample and detecting it by means of a pressure sensor, or

4) combustion in a constant volume chamber with direct injection of fuel into heated, compressed air and determination of the indicated cetane number (ICN) by comparing the ignition characteristics of the tested fuel with mixtures of primary reference fuels of a known indicated cetane number (ICN) value under standardised operating conditions.

5.1. For the determination of the cetane number with the method referred to in subparagraph 5(1):

1) the method of determination, the reagents used, the type of apparatus used and its preparation and the preparation of the sample, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN ISO 5165 standard;

2) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

5.2. For the determination of the cetane number with the method referred to in subparagraph 5(2):

1) the mean ignition delay value determined for a specified number of cycles is used in the equation for the calculation of the derived cetane number (DCN); the derived cetane number (DCN) is an approximation (estimation) of the cetane number determined according to the PN-EN ISO 5165 standard in a conventional full-size test engine;
2) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN EN 15195 standard;
3) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

5.3. For the determination of the cetane number with the method referred to in subparagraph 5(3):

1) the derived cetane number (DCN) is calculated from the determined ignition delay and combustion delay rate using an equation;
2) the method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN EN 16715 standard;
3) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

5.4. For the determination of the cetane number with the method referred to in subparagraph 5(4):

1) the analytical sample of the tested material is taken automatically from the sample vial, placed in the carousel of the automatic sample feeder, heated during the pressure increase and then injected at the beginning of the combustion cycle into a constant volume combustion chamber with controlled temperature and pressure, which has been charged with compressed air of a specified quality; each injection and resulting ignition causes a sharp increase in the pressure in the combustion chamber, which is detected by the dynamic pressure sensor;
2) the method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN EN 17155 standard;
3) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

6. The testing of the corrosive effect on copper is carried out comparatively to standardised corrosion benchmarks.

6.1. A copper plate is immersed in the test sample of a specific volume and then heated under strictly specified conditions. After the heating is finished, the copper plate is removed, washed and its colour is assessed by comparing it with corrosion benchmarks.

6.2. The method of testing the corrosive effect on copper, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN ISO 2160 standard.

6.3. The precision of the method referred to in subparagraph 6 is specified in Annex A to the PN-EN 14214 standard.

7. Oxidative stability (at 110 °C) is determined with the following method:

1) consisting in releasing a purified air stream through the test sample, or

2) accelerated oxidation consisting in subjecting the test sample to ageing at 110 °C in a purified air stream.

7.1. For the determination of oxidative stability (at 110 °C) with the method referred to in subparagraph 7(1):

1) volatile compounds released from the sample by oxidation process pass, together with air, into a vessel containing demineralised or distilled water, fitted with an electrode for measuring the conductivity, combined with a measuring unit indicating the end of the induction period;

2) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the Polish standard implementing the EN ISO 14112 standard;

3) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

7.2. For the determination of oxidative stability (at 110 °C) with the method referred to in subparagraph 7(2):

1) volatile compounds released from the sample by oxidation process pass, together with air, into a vessel containing demineralised or distilled water, fitted with an electrode for measuring the conductivity, combined with a measuring unit indicating the end of the induction period;

2) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN 15751 standard;

3) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

8. The acid value is determined by the titration method by dissolving the test sample in a solvent mixture and titration with a diluted potassium hydroxide solution using phenolphthalein as an indicator to determine the titration end point.

8.1. The method of determination of acid value, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the Polish standard implementing the EN ISO 14104 standard;

8.2. The precision of the method referred to in subparagraph 8 is specified in Annex A to the PN-EN 14214 standard.

9. The iodine value is determined with the following method:

1) titration, consisting in dissolving the test sample in a mixture of solvents, adding Wijs reagent and then, after a certain time, adding to the sample potassium iodide and water, and the titration of the released iodine as sodium thiosulphate solution, or

2) calculation on the basis of gas chromatography data, in which the gas chromatography results for individual fatty acid methyl esters are used as inputs.

9.1. For the determination of iodine value with the method referred to in subparagraph 9(1):

1) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the Polish standard implementing the EN ISO 14111 standard;

2) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

9.2. For the determination of iodine value with the method referred to in subparagraph 9(2):

1) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the Polish standard implementing the EN ISO 16300 standard;

2) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

10. The content of linolenic acid methyl ester is determined by gas chromatography using an internal reference, which consists in the separation of the mixture into individual components in the gaseous phase.

10.1. The method of determination of the content of linolenic acid methyl ester, the reagents and materials used, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the Polish standard implementing the EN 14103 standard.

10.2. The precision of the method referred to in subparagraph 10 is specified in Annex A to the PN-EN 14214 standard.

11. The content of polyunsaturated fatty acid methyl esters (≥ 4 double bonds) is determined by gas chromatography using the internal reference C23:0 methyl ester.

11.1. The method of determination of the content of polyunsaturated fatty acid methyl esters (≥ 4 double bonds), the type of apparatus used and its preparation, and the method of drawing up the test report are laid down in the Polish standard implementing the EN 15779 standard.

12. The methyl alcohol content is determined with a method consisting in heating the sample at a temperature of 80 °C in a hermetically sealed vial and then, after reaching the equilibrium, injecting a specific part of the gaseous phase into the chromatograph where methanol is detected using a flame ionisation detector and its quantity is determined by reference to the external reference solution.

12.1. Methyl alcohol content can also be determined by adding an internal reference solution to the sample and then using an internal calibration factor.

12.2. The method of determination of the content of methyl alcohol, the reagents and reference solutions used, the type of apparatus used and its preparation, as well as the method of preparing the test report are specified in the Polish standard implementing the EN 14110 standard.

12.3. The precision of the method referred to in subparagraph 12 is specified in Annex A to the PN-EN 14214 standard.

13. The content of monoacylglycerols, diacylglycerols, triacylglycerols and general glycerols is determined by a method of analysis of silane derivatives using gas

chromatography on a short capillary column with a thin-layer film, using direct column dispensation and flame ionisation detectors.

13.1. After calibration, the quantitative analysis of monoacylglycerols, diacylglycerols, triacylglycerols and general glycerols is performed using the internal reference method and the total glycerol content is calculated on the basis of the results obtained.

13.2. The method of determination of the content of monoacylglycerols, diacylglycerols, triacylglycerols and general glycerols, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the Polish standard implementing the EN ISO 14105 standard.

13.3. The precision of the method referred to in subparagraph 13 is specified in Annex A to the PN-EN 14214 standard.

14. The free glycerol content is determined with a method consisting in:

1) an analysis of silicone derivatives using gas chromatography on a short capillary column with a thin-layer film, using direct column dispensation and flame ionisation detectors, or

2) adding ethanol, water, hexane and an internal reference solution to the test sample, which will result in the formation of two phases and quantitative transfer of free glycerol to the lower phase, the analysis of which, using gas chromatography, allows quantification of the concentration of free glycerol.

14.1. For the determination of free glycerol content with the method referred to in subparagraph 14(1):

1) after calibration, the quantitative analysis of free glycerol is performed using the internal standard method;

2) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the Polish standard implementing the EN ISO 14105 standard;

3) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

14.2. For the determination of free glycerol content with the method referred to in subparagraph 14(2):

1) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN 14106 standard;

2) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

15. The water content is determined by coulometric titration consisting in introducing the weighed sample into the titration vessel of the Karl Fischer coulometric titrator in which iodine for the Karl Fischer reaction is electrolytically released at the anode proportionally to the amount of water contained in the sample.

15.1. When all water content is titrated, the excess iodine is detected by the electrometric endpoint sensor and the titration is stopped.

15.2. The method of determination of water content, the reagents and materials used, the type of apparatus used and its preparation and the preparation of the sample, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN ISO 12937 standard.

16. The total contamination is determined by determining the proportion of the weight of the impurities filtered out by the filter in relation to the total mass of the sample.

16.1. A certain amount of the prepared sample is filtered in vacuum through a pre-weighted filter. The filter with the residue is washed, dried, and weighed. The contamination is calculated based on the difference in the filter masses and determined by reference to the sample weight in mg/kg.

16.2. The method of determination of total contamination, the reagents used, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the Polish standard implementing the EN 12662-2 standard.

16.3. The precision of the method referred to in subparagraph 16 is specified in Annex A to the PN-EN 14214 standard.

17. The sulphated ash content is determined by weight, by calculating the content of sulphated ash obtained by burning the test sample and reaction of the incineration residue with sulphuric acid.

17.1. The test sample is burned until only ash and coal remain. After cooling the combustion products, they are subjected to sulphuric acid and roasted at 775 °C until carbon oxidation is complete. The ash is then cooled, re-treated with sulphuric acid and roasted until solid mass is obtained.

17.2. The method of determination of sulphated ash content, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-ISO 3987 standard.

17.3. The precision of the method referred to in subparagraph 17 is specified in Annex A to the PN-EN 14214 standard.

18. The sulphur content is determined using the following method:

1) X-ray fluorescence spectroscopy with wavelength dispersion, consisting in exposing the test sample placed in a cuvette to primary radiation with specified wavelengths generated by an X-ray tube, or

2) ultraviolet fluorescence consisting in the application of the fluorescence of sulphur dioxide excited by ultraviolet radiation, previously formed by oxidation of sulphur compounds contained in the test sample under specified conditions, or

3) X-ray fluorescence spectrometry with energy dispersion, consisting in exposing the test sample, placed in a cuvette adapted to an X-ray permeable window, to a stream of exciting radiation emitted by an X-ray lamp.

18.1. For the determination of sulphur content with the method referred to in subparagraph 18(1):

1) sulphur content is determined based on the measured count rate of X-ray fluorescent radiation S-K_α lines and the background radiation using the calibration curve;

2) the method of determination, the reagents used, the type of apparatus used and its preparation and the method of calculation and presentation of the results are laid down in the PN-EN ISO 20884 standard;

3) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

18.2. For the determination of sulphur content with the method referred to in subparagraph 18(2):

1) the measure of the sulphur content of a test sample is the intensity of fluorescent ultraviolet radiation;

2) the method of determination, the reagents used, the type of apparatus used and its preparation and the method of calculation and presentation of the results are laid down in the PN-EN ISO 20846 standard;

3) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

18.3. For the determination of sulphur content with the method referred to in subparagraph 18(3):

1) the method of determination, the reagents used, the type of apparatus used and its preparation and the method of calculation and presentation of the results are laid down in the Polish standard implementing the EN ISO 13032 standard;

2) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

19. Sodium (Na) content is determined:

1) directly — by atomic absorption spectrometry at a wavelength of 589.0 nm, having previously dissolved the test sample in a solution of xylene, or

2) by optical emission spectral analysis with inductively excited plasma, having previously diluted the test sample with naphtha.

19.1. For the determination of the sodium content with the method referred to in subparagraph 19(1):

1) reference solutions are used, prepared from an organic sodium compound in the form of salt dissolved in a mixture of xylene and diluting oil;

2) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 14108 standard.

19.2. For the determination of the sodium content with the method referred to in subparagraph 19(2):

1) sodium content is determined by comparing the atomic emission intensity of the reference solution and the sample at specified wavelengths.

2) the method of determination, the reagents and reference solutions used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 14538 standard.

20. Potassium (K) content is determined:

1) directly — by atomic absorption spectrometry at a wavelength of 766.5 nm, having previously dissolved the test sample in a solution of xylene and a stabiliser, or

2) by optical emission spectral analysis with inductively excited plasma, having previously diluted the sample with naphtha.

20.1. For the determination of potassium content with the method referred to in subparagraph 20(1):

1) reference solutions are used, prepared from an organic potassium compound in the form of salt, dissolved in a mixture of xylene and a stabiliser;

2) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 14109 standard.

20.2. For the determination of potassium content with the method referred to in subparagraph 20(2):

1) potassium content is determined by comparing the atomic emission intensity of the reference solution and the sample at specified wavelengths;

2) the method of determination, the reagents and reference solutions used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 14538 standard.

21. The precision of determining the total content of group I metals (Na + K), for the determination of sodium content in accordance with standard PN-EN 14108 or PN-EN 14538 and of potassium content in accordance with standard PN-EN 14109 or PN-EN 14538, is specified in Annex A to the PN-EN 14214 standard.

22. The content of group II metals (Ca + Mg) is determined by optical emission spectral analysis with inductively excited plasma after diluting the test sample with naphtha.

22.1. The content of group II metals (Ca + Mg) is determined by comparing the atomic emission intensity of the reference solution and the sample at specified wavelengths.

22.2. The method of determination of the content of group II metals (Ca + Mg), the reagents and reference solutions used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN EN 14538 standard.

22.3. The precision of the method referred to in subparagraph 22 is specified in Annex A to the PN-EN 14214 standard.

23. Phosphorus content is determined by:

1) dissolving the test sample in xylene and its introduction, in the form of an aerosol, together with reference solutions prepared from an organic phosphorus compound, into an inductively coupled argon plasma, or
2) inductively excited plasma optical emission spectrometry, which consists in passing the sample dissolved in naphtha through a spectrometer.

23.1. For the determination of phosphorus content with the method referred to in subparagraph 23(1):

1) phosphorus content is determined by comparing the emission of the element in the test sample solution with the reference emissions at the same wavelength;
2) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN 14107 standard;
3) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

23.2. For the determination of phosphorus content with the method referred to in subparagraph 23(2):

1) phosphorus content is determined by comparison with the reference solution;
2) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN 16294 standard;
3) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

24. Cold filter plugging point (CFPP) is determined using the following method:

1) aspirating the test sample through a standardised mesh filter into a pipette under controlled negative pressure and at a temperature decreased by 1 °C increments by means of a cooling bath, the temperature of which is lowered in leaps, until the flow time is stopped or slowed down so that the pipette filling time exceeds 60 seconds or the fuel does not flow completely into the measuring vessel, or
2) aspirating the test sample through a standardised mesh filter into a pipette under controlled negative pressure of 2 kPa and at a temperature lowered in 1 °C decreased in a linear manner in a cooling bath until the flow time is stopped or slowed down so that

the pipette filling time exceeds 60 seconds or the fuel does not flow completely into the measuring vessel.

24.1. For the determination of cold filter plugging point (CFPP) with the method referred to in subparagraph 24(1):

1) the method of determination, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN ISO 116 standard;

2) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

24.2. For the determination of cold filter plugging point (CFPP) with the method referred to in subparagraph 24(2):

1) the method of determination, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN ISO 16329 standard;

2) the precision of the method is specified in Annex A to the PN-EN 14214 standard.

25. The criteria set out in the PN-EN ISO 4259-2 standard are used to interpret the test results referred to in subparagraphs 1 to 24.2.

II. Methods for testing the quality of diesel fuel containing 20 % methyl ester, used in vehicles, including agricultural tractors, as well as in non-road mobile machinery, with diesel engines designed to burn this liquid biofuel4)

1. Fatty acid methyl ester (FAME) content is determined by infrared spectroscopy consisting in recording the infrared spectrum of the test sample diluted with cyclohexane, and then measuring the absorbance at a maximum peak of approximately 1 745 cm-^{1} ± 5 cm-^{1} and comparing it with that of reference fatty acid methyl ester solutions.

1.1. The method of determination of the content of fatty acid methyl esters (FAME), the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN 14078 standard.

2. The cetane number is determined with the following method:

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[4] Prepared in accordance with the PN-EN 16709 standard: Fuels for motor vehicles — High FAME diesel fuel (B20 and B30) — Requirements and test methods.

1) engine test consisting in comparing the self-ignition properties of the tested fuel with the corresponding properties of reference fuel mixtures of known cetane numbers, using a test engine under standardised conditions, or
2) combustion in a constant volume chamber consisting in injecting a fuel sample into a compressed air charge in a constant volume chamber, detecting the start of injection and the beginning of its combustion by sensors for a specified number of cycles and determining the ignition delay time, or
3) combustion in a high-temperature and pressure-controlled constant volume chamber consisting in injecting the test fuel sample into a heated, compressed, synthetic air of required quality, generating a dynamic pressure wave due to the combustion of the test sample and detecting it by means of a pressure sensor, or
4) engine method consisting in comparing the combustion characteristics of the test engine fuel with those of reference fuel mixtures of a known cetane number under standardised operating conditions by comparing the mass of the intake air with the ignition delay specified, or
5) combustion in a constant volume chamber consisting in the direct injection of fuel into compressed air, the pressure and temperature of which are regulated to specific values.

2.1. For the determination of the cetane number with the method referred to in subparagraph 2(1), the method of determination, the reagents and materials used, the type of apparatus used and its preparation, sample preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN ISO 5165 standard.

2.2. For the determination of the cetane number with the method referred to in subparagraph 2(2):

1) the mean ignition delay value determined for a specified number of cycles is used in the equation for the calculation of the derived cetane number (DCN); the derived cetane number (DCN) is an approximation (estimation) of the cetane number determined according to the PN-EN ISO 5165 standard in a conventional full-size test engine;
2) the method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 15195 standard.

2.3. For the determination of the cetane number with the method referred to in subparagraph 2(3):

1) the derived cetane number (DCN) is calculated from the determined ignition delay and combustion delay rate using an equation;

2) the method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 16715 standard.

2.4. For the determination of the cetane number with the method referred to in subparagraph 2(4), the method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the Polish standard implementing the EN 16906 standard.

2.5. For the determination of the cetane number with the method referred to in subparagraph 2(5):

1) the complete test sequence consists of two initial combustion cycles for the apparatus to achieve equilibrium and 25 further test cycles to determine the ignition delay; the mean ignition delay (ID), determined based on the results of the 25 cycles, is inserted into the equation for the calculation of the derived cetane number (DCN);

2) the method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 16144 standard.

3. The density at 15 °C is determined using the following method:

1) oscillation, by inserting a sample of approximately 1 ml into the measuring cell of an oscillating densimeter thermostated to maintain the reference temperature of 15 ℃, or

2) with a hydrometer, consisting in measuring the density of a test sample at a given temperature using a hydrometer immersed in the sample placed in a cylinder.

3.1. For the determination of density with the method referred to in subparagraph 3(1), the method of determination, the reagents, the type of apparatus used and its preparation, and sample preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the Polish standard implementing the EN ISO 12185 standard.

3.2. For the determination of density with the method referred to in subparagraph 3(2):

1) the indication on the hydrometer scale is read, the temperature of the test sample is recorded, and the result of the measurement relative to a temperature of 15 °C is read with the use of appropriate conversion tables;

2) the method of determination, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN ISO 3675 standard.

4. Flash point is determined by means of the Pensky-Martens closed-cup test by placing the test sample in a cup and heating it whilst mixing continuously, until the source of ignition introduced through the opening in the cup lid causes the vapour on the surface of the test sample to ignite.

4.1. The lowest temperature at which the application of the ignition source causes the vapours of the test sample to ignite and the flame to spread over the surface of the liquid is taken as the flash point under absolute atmospheric pressure.

4.2. The measured flash point of the test sample is adjusted to standard atmospheric pressure.

4.3. The method of determination of flash point, the reagents and materials used, the type of apparatus used and its preparation, sample handling, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN ISO 2719 standard.

5. The viscosity of methyl ester at 40 °C is determined by measuring the time necessary for a given volume of the test sample to flow under gravitational force through a calibrated glass capillary viscometer, under repeatable conditions, at a known and strictly controlled temperature.

5.1. The viscosity is calculated by multiplying the measured flow time of constant fluid volume between the lines of the measuring vessel by a viscometer calibration constant.

5.2. The method of determination of viscosity at 40 °C, the reagents and materials used, the type of apparatus used and its preparation and calibration, the method of calculation and presentation of the results as well as the precision of the method, and the method of drawing up the test report are laid down in the Polish standard implementing the EN ISO 3104 standard.

6. The sulphur content is determined with the following method:

1) X-ray fluorescence spectroscopy with wavelength dispersion, consisting in exposing the test sample placed in a cuvette to primary radiation with specified wavelengths generated by an X-ray tube, or

2) ultraviolet fluorescence consisting in the application of the fluorescence of sulphur dioxide excited by ultraviolet radiation, previously formed by oxidation of sulphur compounds contained in the test sample under specified conditions, or

3) X-ray fluorescence spectrometry with energy dispersion, consisting in exposing the test sample, placed in a cuvette adapted to an X-ray permeable window, to a stream of exciting radiation emitted by an X-ray lamp.

6.1. For the determination of sulphur content with the method referred to in subparagraph 6(1):

1) sulphur content is determined based on the measured count rate of X-ray fluorescent radiation S-K_α lines and the background radiation using the calibration curve;

2) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and reporting of the results and the precision of the method are laid down in the PN-EN ISO 20884 standard.

6.2. For the determination of sulphur content with the method referred to in subparagraph 6(2):

1) the measure of the sulphur content of a test sample is the intensity of fluorescent ultraviolet radiation;

2) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and reporting of the results and the precision of the method are laid down in the PN-EN ISO 20846 standard.

6.3. For the determination of sulphur content with the method referred to in subparagraph 6(3):

1) the intensity of the characteristic sulphur X-ray line K-$L_{2,3}$ is measured and the cumulative number of counts is compared with the values of the calibration curve obtained for the reference solutions with sulphur content adequate for the concentration range tested;

2) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the Polish standard implementing the EN-ISO 13032 standard.

7. Manganese content is determined by inductively excited plasma optical emission spectrometry, which consists in dissolving a specified amount of the sample in a hydrocarbon solvent and introducing this solution into the spectrometer.

7.1. Manganese content is determined based on the calibration curve drawn up for the corresponding reference manganese solutions.

7.2. An internal reference is used to adjust the viscosity-related effects of the test solutions.

7.3. The method of testing for manganese content, the type of apparatus used and its preparation, the method of determination , the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 16576 standard.

8. The content of polycyclic aromatic hydrocarbons is determined by high performance liquid chromatography with a refractive index detector by diluting the test sample of a known mass with heptane and injecting a specific volume of this solution into a high performance liquid chromatograph equipped with a polar column.

8.1. The polar column is to feature weak affinity with non-aromatic hydrocarbons allowing the separation and selective division of aromatic hydrocarbons resulting in the aromatic hydrocarbons being separated from the non-aromatic hydrocarbons and washed out in appropriate ranges corresponding to their ring structure.

8.2. The polar column is connected to the refraction index change detector which detects the components that are being washed out from the column. The electronic signal from the detector is monitored continuously by means of a data processor. The amplitudes of the signals of the aromatic compounds in the sample are compared with those obtained during the previously performed determination of reference values in order to calculate the mass fraction expressed as a percentage of individual aromatic hydrocarbon groups.

8.3. The sum of the mass fractions of bi- (DAH), tri-, and polycyclic aromatic hydrocarbons (T+AH) expressed as a percentage, provided as a mass fraction, represents the polycyclic aromatic hydrocarbon content of POLY-AH.

8.4. The method of determination of polycyclic aromatic hydrocarbons content, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results as well as the precision of the method, and the method of drawing up the test report are laid down in the Polish standard implementing the EN 12916 standard.

9. Ash content is determined by incinerating a test sample in a special vessel, reducing the carbon residue to ash by heating in a muffle furnace at 775 °C, and weighing the obtained residue.

9.1. The method of determination of ash content, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN ISO 6245 standard.

10. The water content is determined by coulometric titration consisting in introducing the weighed sample into the titration vessel of the Karl Fischer coulometric titrator in which iodine for the Karl Fischer reaction is electrolytically released at the anode proportionally to the amount of water contained in the sample.

10.1. When all water content is titrated, the excess iodine is detected by the electrometric endoint sensor and the titration is stopped.

10.2. The method of determination of water content, the reagents and materials used, the type of apparatus used and its preparation and the preparation of the sample, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN ISO 12937 standard.

11. The total contamination is determined by determining the proportion of the weight of the impurities filtered out by the filter in relation to the total mass of the sample.

11.1. A certain amount of the prepared sample is filtered in vacuum through a pre-weighted filter. The filter with the residue is washed, dried, and weighed. The contamination is calculated based on the difference in the filter masses and determined by reference to the sample weight in mg/kg.

11.2. The method of determination, the reagents used, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the Polish standard implementing the EN 12662-1 standard.

12. Oxidative stability is determined by accelerated oxidation through subjecting the test sample to ageing at 110 °C in a purified air stream.

12.1. Volatile compounds released from the sample by oxidation process pass, together with air, into a vessel containing demineralised or distilled water, fitted with an electrode for measuring the conductivity, combined with a measuring unit indicating the end of the induction period.

12.2. The method of determination of oxidative stability, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 15751 standard.

13. Fractional composition is determined as follows:

1) at atmospheric pressure, using a method consisting in separation of the fractions through a distillation process whose course and parameters depend on the composition and anticipated volatile properties of the sample (group 4), or

2) using gas chromatography consisting in introducing the sample into a chromatographic column and separating hydrocarbons in order of increasing boiling points.

13.1. For the determination of fractional composition with the method referred to in subparagraph 13(1):

1) a 100 ml test sample is distilled under strictly defined conditions according to the requirements of the group referred to in subparagraph 13(1) which includes the sample, and systematic observation of the thermometer and the volume of the obtained condensate is carried out;

2) after distillation, the volume of the liquid remaining in the flask is measured and the quantitative losses in the distillation process are recorded; the readings of the thermometer are adjusted to atmospheric pressure and calculations are carried out on the basis of these data, according to the type of the sample and the specified requirements;

3) the method of determination, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN ISO 3405 standard.

13.2. For the determination of fractional composition with the method referred to in subparagraph 13(2):

1) the temperature of the column is raised repeatedly and the area under the chromatogram is recorded during the analysis;

2) the boiling point is related to the time axis of the reference curve obtained through an analysis carried out under the same conditions of a mixture of known hydrocarbons with boiling points adequate for the expected boiling range of the test sample;

3) on the basis of the data referred to in subparagraphs 1 and 2, the boiling point distribution is determined;

4) the method of determination, the type of apparatus used and its preparation as well as sample preparation, instructions for converting the data to an equivalent laid down in the PN-EN ISO 3405 standard, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN ISO 3924 standard.

14. Cold filter plugging point (CFPP) is determined using the following method:

1) aspirating the test sample through a standardised mesh filter into a pipette under controlled negative pressure and at a temperature decreased by 1 °C increments by means of a cooling bath, the temperature of which is lowered in leaps until the flow time is stopped or slowed down so that the pipette filling time exceeds 60 seconds or the fuel does not flow completely into the measuring vessel, or

2) aspirating the test sample through a standardised mesh filter into a pipette under controlled negative pressure of 2 kPa and at a temperature lowered in 1 °C decreased in a linear manner in a cooling bath until the flow time is stopped or slowed down so that the pipette filling time exceeds 60 seconds or the fuel does not flow completely into the measuring vessel.

14.1. For the determination of the cold filter plugging point with the method referred to in subparagraph 14(1), the method of determination, the type of apparatus used and its preparation and sample preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN 116 standard.

14.2. For the determination of cold filter plugging point with the method referred to in subparagraph 14(2), the method of determination, the type of apparatus used and its preparation and sample preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN 16329 standard.

15. The criteria set out in PN-EN ISO 4259-2 are used to interpret the test results referred to in subparagraphs 1 to 14.2.

III. Methods for testing the quality of liquid biohydrocarbons constituting a stand-alone fuel and paraffinic diesel used in vehicles, including agricultural tractors, as well as in non-road mobile machinery, with diesel engines designed to burn this liquid biofuel[5)]

1. The cetane number is determined with the following method:

1) engine test consisting in comparing the self-ignition properties of the tested fuel with the corresponding properties of reference fuel mixtures of known cetane numbers, using a test engine under standardised conditions, or

2) combustion in a constant volume chamber consisting in injecting a fuel sample into a compressed air charge in a constant volume chamber, detecting the start of injection and the beginning of its combustion by sensors for a specified number of cycles and determining the ignition delay time, or

3) engine method consisting in comparing the combustion characteristics of the test engine fuel with those of reference fuel mixtures of a known cetane number under standardised operating conditions by comparing the mass of the intake air with the ignition delay specified, or

4) combustion in a constant volume chamber with direct injection of fuel into heated, compressed air and determination of the indicated cetane number (ICN) by comparing the ignition characteristics of the tested fuel with mixtures of primary reference fuels of a known indicated cetane number (ICN) value under standardised operating conditions.

1.1. For the determination of the cetane number with the method referred to in subparagraph 1(1), the method of determination, the reagents and materials used, the type of apparatus used and its preparation, sample preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN ISO 5165 standard.

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[5] Drawn up on the basis of the PN-EN 15940 standard: Motor vehicle fuels — Paraffinic diesel fuel from synthesis or hydrotreatment — Requirements and test methods.

1.2. For the determination of cetane number with the method referred to in subparagraph 1(2):

1) the mean ignition delay value determined for a specified number of cycles is used in the equation for the calculation of the derived cetane number (DCN); the derived cetane number (DCN) is an approximation (estimation) of the cetane number determined according to the PN-EN ISO 5165 standard in a conventional full-size test engine;

2) the method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 15195 standard.

1.3. For the determination of the cetane number with the method referred to in subparagraph 1(3), the method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the Polish standard implementing the EN 16906 standard.

1.4. For the determination of the cetane number with the method referred to in subparagraph 1(4):

1) the analytical sample of the tested material is taken automatically from the sample vial, placed in the carousel of the automatic sample feeder, heated during the pressure increase and then injected at the beginning of the combustion cycle into a constant volume combustion chamber with controlled temperature and pressure, which has been charged with compressed air of a specified quality; each injection and resulting ignition causes a sharp increase in the pressure in the combustion chamber, which is detected by the dynamic pressure sensor;

2) the method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 17155 standard.

2. The density at 15 °C is determined using the following method:

1) oscillation, by inserting a sample of approximately 1 ml into the measuring cell of an oscillating densimeter thermostated to maintain the reference temperature of 15 ℃, or

2) with a hydrometer, consisting in measuring the density of a test sample at a given temperature using a hydrometer immersed in the sample placed in a cylinder.

2.1. For the determination of density with the method referred to in subparagraph 2(1), the method of determination, the reagents, the type of apparatus used and its preparation, and sample preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the Polish standard implementing the EN ISO 12185 standard.

2.2. For the determination of density with the method referred to in subparagraph 2(2):

1) the indication on the hydrometer scale is read, the temperature of the test sample is recorded, and the result of the measurement relative to a temperature of 15 °C is given in line with Annex B to the PN-EN 15940 standard;

2) 2) the method of determination, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN ISO 3675 standard.

3. The total aromatic content is determined by high performance liquid chromatography with a refractive index detector by diluting the test sample of a known mass with heptane and injecting a specific volume of this solution into a high performance liquid chromatograph equipped with a polar column.

3.1. The polar column is to feature weak affinity with non-aromatic hydrocarbons allowing the separation and selective division of aromatic hydrocarbons resulting in the aromatic hydrocarbons being separated from the non-aromatic hydrocarbons and washed out in appropriate ranges corresponding to their ring structure.

3.2. The polar column is connected to the refraction index change detector which detects the components that are being washed out from the column. The electronic signal from the detector is monitored continuously by means of a data processor. The amplitudes of the signals of the aromatic compounds in the sample are compared with those obtained during the previously performed determination of reference values in order to calculate the mass fraction expressed as a percentage of individual aromatic hydrocarbon groups.

3.3. The sum of the mass fractions of bi- (DAH), tri-, and polycyclic aromatic hydrocarbons (T+AH) expressed as a percentage, provided as a mass fraction, represents the polycyclic aromatic hydrocarbon content of POLY-AH.

3.4. The method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the Polish standard implementing the EN 12916 standard.

4. The sulphur content is determined with the following method:

1) X-ray fluorescence spectroscopy with wave dispersion, consisting in exposing a test sample placed in a cuvette to primary radiation with a specified wavelength generated by an X-ray tube, or

2) ultraviolet fluorescence consisting in the application of the fluorescence of sulphur dioxide excited by ultraviolet radiation, previously formed by oxidation of sulphur compounds contained in the test sample under specified conditions, or

3) X-ray fluorescence spectrometry with energy dispersion, consisting in exposing a test sample, placed in a cuvette adapted to the X-ray permeable window, to a stream of exciting radiation emitted by an X-ray lamp.

4.1. For the determination of sulphur content with the method referred to in subparagraph 4(1):

1) sulphur content is determined based on the measured count rate of X-ray fluorescent radiation S-K_α lines and the background radiation using the calibration curve;

2) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and reporting of the results and the precision of the method are laid down in the PN-EN ISO 20884 standard.

4.2. For the determination of sulphur content with the method referred to in subparagraph 4(2):

1) the measure of the sulphur content of a test sample is the intensity of fluorescent ultraviolet radiation;

2) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and reporting of the results and the precision of the method are laid down in the PN-EN ISO 20846 standard.

4.3. For the determination of sulphur content with the method referred to in subparagraph 4(3):

1) the intensity of the characteristic sulphur X-ray line K-$L_{2,3}$ is measured and the cumulative number of counts is compared with the values of the calibration curve

obtained for the reference solutions with sulphur content adequate for the concentration range tested;

2) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the Polish standard implementing the EN-ISO 13032 standard.

5. Manganese content is determined by inductively excited plasma optical emission spectrometry, which consists in dissolving a specified amount of the sample in a hydrocarbon solvent and introducing this solution into the spectrometer.

5.1. Manganese content is calculated by comparison to the reference concentrations.

5.2. The method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 16576 standard.

6. Flash point is determined by means of the Pensky-Martens closed-cup test by placing the test sample in a cup and heating it whilst mixing continuously, until the source of ignition introduced through the opening in the cup lid causes the vapour on the surface of the test sample to ignite.

6.1. The lowest temperature at which the application of the ignition source causes the vapours of the test sample to ignite and the flame to spread over the surface of the liquid is taken as the flash point under absolute atmospheric pressure.

6.2. The measured flash point of the test sample is adjusted to standard atmospheric pressure.

6.3. The method of determination, the reagents and materials used, the type of apparatus used and its preparation, sample handling, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN ISO 2719 standard.

7. Carbon residue (from 10 % of the distillation residue) is determined by the micro-gravimetric method as the residue after evaporation and thermal deposition of the test sample under specified conditions.

7.1. The test sample is placed in a glass vial and heated to 500 °C in an inert gas stream under controlled conditions for a specified period of time. Volatile substances released during the reaction are removed with an inert gas and the carbonaceous residue is weighed.

7.2. The method of determination of carbon residue, the reagents and materials used, the type of apparatus used and its preparation and the preparation of the sample, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN ISO 10370 standard.

8. Ash content is determined by incinerating a test sample in a special vessel, reducing the carbon residue to ash by heating in a muffle furnace at 775 °C, and weighing the obtained residue.

8.1. The method of determination of ash content, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN ISO 6245 standard.

9. The water content is determined by coulometric titration consisting in introducing the weighed sample into the titration vessel of the Karl Fischer coulometric titrator in which iodine for the Karl Fischer reaction is electrolytically released at the anode proportionally to the amount of water contained in the sample.

9.1. When all water content is titrated, the excess iodine is detected by the electrometric endoint sensor and the titration is stopped.

9.2. The method of determination of water content, the reagents and materials used, the type of apparatus used and its preparation and the preparation of the sample, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN ISO 12937 standard.

10. The total contamination is determined by determining the proportion of the weight of the impurities filtered out by the filter in relation to the total mass of the sample.

10.1. A certain amount of the prepared sample is filtered in vacuum through a pre-weighted filter. The filter with the residue is washed, dried, and weighed. The contamination is calculated based on the difference in the filter masses and determined by reference to the sample weight in mg/kg.

10.2. The method of determination of total contamination, the reagents used, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results, the precision of the method and the method of drawing up the test report are laid down in the Polish standard implementing the EN 12662-1 standard.

11. The testing of the corrosive effect on copper is carried out comparatively to standardised corrosion benchmarks.

11.1. A copper plate is immersed in the test sample of a specific volume and then heated under strictly specified conditions. After the heating is finished, the copper plate is removed, washed and its colour assessed by comparing it with corrosion benchmarks.

11.2. The method of testing the corrosive effect on copper, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, the precision of the method and the method of drawing up the test report are laid down in the PN-EN ISO 2160 standard.

12. Fatty acid methyl ester (FAME) content is determined by infrared spectroscopy consisting in recording the infrared spectrum of the test sample diluted with a FAME-free solvent, and then measuring the absorbance at a maximum peak of approximately 1 745 cm-$^{-1}$ ± 5 cm-$^{-1}$ and comparing it with that of reference fatty acid methyl ester solutions.

15.1. The method of determination of the content of fatty acid methyl esters (FAME), the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN 14078 standard.

13. Oxidative stability is determined by accelerated oxidation through subjecting the test sample to ageing at 95 °C for 16 hours with oxygen flowing through the test sample.

13.1. At the end of the ageing process, the test sample is cooled to room temperature and then filtered to determine the content of filterable insoluble sediments.

13.2. Insoluble sediments that adhere to the test tube and other glass parts are removed with a ternary solvent. Then, the ternary solvent is evaporated to obtain adherent insoluble sediments.

13.3. The total content of adhering insoluble sediments, which is a measure of oxidation resistance, is given as the sum of filterable insoluble sediments and insoluble sediments adhering to the tube and other glass parts.

13.4. The method of determination of oxidative stability, the reagents and materials used, the type of apparatus used and its preparation and the preparation of the sample, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN ISO 12205 standard.

14. The oxidative stability of a fuel containing more than 2.0 % (V/V) of fatty acid methyl esters (FAME) is determined by accelerated oxidation consisting in:

1) subjecting the test sample to ageing at 110 °C in a purified air stream, or

2) measuring the induction period to the required break point.

14.1. For the determination of oxidative stability with the method referred to in subparagraph 14(1):

1) volatile compounds released from the sample by oxidation process pass, together with air, into a vessel containing demineralised or distilled water, fitted with an electrode for measuring the conductivity, combined with a measuring unit indicating the end of the induction period;

2) the method of determination, the reagents used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 15751 standard.

14.2. For the determination of oxidative stability with the method referred to in subparagraph 14(2):

1) a known volume of the sample at ambient temperature is placed in a reaction vessel containing oxygen at a pressure of 700 kPa +/- 5 kPa, heated to 140 ℃; the pressure in the vessel decreases as oxygen is used to oxidise the sample and is recorded at intervals of 1 s until the breakpoint is reached; the time from the start of the determination to the breakpoint is the induction period at test temperature of 140 ℃ +/- 0,5 ℃;

2) the method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 16091 standard.

15. Lubricity, the wear scar diameter (WSD) at 60 °C are determined using the high-frequency reciprocating rig (HFRR).

15.1. Fixed in a vertically mounted holder, a steel test ball is pressed with a set load against a stationary, horizontally mounted steel plate. The test ball oscillates with a fixed stroke frequency and length. During the test, the ball and plate are completely immersed in the test sample.

15.2. The wear scar diameter (WSD) on the test ball under strictly controlled test conditions (at the temperature of 60 °C) is a measure of the lubricity of the test sample.

15.3. The method of determination, the reagents and materials used, the type of apparatus used and its preparation, the measurement of the wear scar diameter (WSD) on the test ball, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the Polish standard implementing the EN ISO 12156-1 standard.

16. Viscosity at 40 °C is determined using a method consisting in:

1) measurement of the time necessary for a given volume of the test sample to flow under gravitational force through a calibrated glass capillary viscometer, under repeatable conditions, at a known and strictly controlled temperature; or

2) introduction of the test sample into measuring cells of known and strictly controlled temperature consisting of a pair of rotating cylinders and an oscillating U-tube.

16.1. For the determination of viscosity with the method referred to in subparagraph 16(1):

1) viscosity is calculated by multiplying the measured flow time of constant fluid volume between the lines of the measuring vessel by a viscometer calibration constant;

2) the method of determination, the reagents and materials used, the type of apparatus used and its preparation and calibration, the method of calculation and presentation of the results as well as the precision of the method, and the method of drawing up the test report are laid down in the Polish standard implementing the EN ISO 3104 standard.

16.2. For the determination of viscosity with the method referred to in subparagraph 16(2):

1) the viscosity is calculated by dividing the dynamic viscosity by the density determined during the test;

2) the method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the Polish standard implementing the EN ISO 23581 standard.

17. Fractional composition is determined as follows:

1) at atmospheric pressure, using a method consisting in separation of the fractions through a distillation process whose course and parameters depend on the composition and anticipated volatile properties of the sample (group 4), or

2) using gas chromatography consisting in introducing the sample into the chromatographic column and separating hydrocarbons in order of increasing boiling points, or

3) at atmospheric pressure, using a distillation method consisting in heating the bottom of the distillation flask containing the test sample, measuring and recording temperature and pressure by automatic micro-distillation apparatus.

17.1. For the determination of fractional composition with the method referred to in subparagraph 17(1):

1) a 100 ml test sample is distilled under strictly defined conditions according to the requirements of the group referred to in subparagraph 17(1) which includes the sample, and systematic observation of the thermometer and the volume of the obtained condensate is carried out;

2) after distillation, the volume of the liquid remaining in the flask is measured and the quantitative losses in the distillation process are recorded; the readings of the thermometer are adjusted to atmospheric pressure and calculations are carried out on the basis of these data, according to the type of the sample and the specified requirements;

3) the method of determination, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN ISO 3405 standard.

17.2. For the determination of fractional composition with the method referred to in subparagraph 17(2):

1) the temperature of the column is raised repeatedly and the area under the chromatogram is recorded during the analysis;

2) the boiling point is related to the time axis of the reference curve obtained through an analysis carried out under the same conditions of a mixture of known hydrocarbons with boiling points adequate for the expected boiling range of the test sample;

3) on the basis of the data referred to in subparagraphs 1 and 2, the boiling point distribution is determined;

4) the method of determination, the type of apparatus used and its preparation as well as sample preparation, instructions for converting the data to an equivalent laid down in the PN-EN ISO 3405 standard, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN ISO 3924 standard.

17.3. For the determination of fractional composition with the method referred to in subparagraph 17(3):

1) the data collected by the automatic micro-distillation apparatus is processed by a data processing system, converted into distillation characteristics and adjusted for atmospheric pressure;

2) the method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the Polish standard implementing the EN 17306 standard;

3) when the test method involves the correction of a systematic error, the corrected result is used to determine compliance with the limit value.

18. Cold filter plugging point (CFPP) is determined using the following method:

1) aspirating the test sample through a standardised mesh filter into a pipette under controlled negative pressure and at a temperature decreased by 1 °C increments by means of a cooling bath, the temperature of which is lowered in leaps until the flow time is stopped or slowed down so that the pipette filling time exceeds 60 seconds or the fuel does not flow completely into the measuring vessel, or

2) aspirating the test sample through a standardised mesh filter into a pipette under controlled negative pressure of 2 kPa and at a temperature lowered in 1 °C decreased in a linear manner in a cooling bath until the flow time is stopped or slowed down so that the pipette filling time exceeds 60 seconds or the fuel does not flow completely into the measuring vessel.

18.1. For the determination of cold filter plugging point (CFPP) with the method referred to in subparagraph 18(1):

1) the method of determination, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN ISO 116 standard;

2) the precision of the method is specified in Annex A to PN-EN 15940.

18.2. For the determination of cold filter plugging point (CFPP) with the method referred to in subparagraph 18(2):

1) the method of determination, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN ISO 16329 standard;

2) the precision of the method is specified in Annex A to PN-EN 15940.

19. Cloud point is determined with the following method:

1) consisting in measuring the temperature of a sample cooled at a specified rate in a cooling bath and observing the appearance of that sample, or

2) through gradual cooling carried out with the use of automatic types of devices with an optical detection method.

19.1. The temperature at which cloudiness at the bottom of the test sample is observed is taken as the cloud point of the sample.

19.2. For the determination of cloud point referred to in subparagraph 19(1), the method of determination, the type of apparatus used and its preparation, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN ISO 3015 standard.

19.3. For the determination of cloud point referred to in subparagraph 19(2), the method of determination, the type of apparatus used and its preparation, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN ISO 22995 standard.

20. The criteria set out in the PN-EN ISO 4259-2 standard are used to interpret the test results referred to in subparagraphs 1 to 19.3.

IV. Methods for testing the quality of motor gasoline containing between 70 % and 85 % bioethanol, for use in vehicles equipped with spark-ignition engines designed to burn this liquid biofuel(6)

1. The density at 15 °C is determined by means of oscillation, by inserting a sample of approximately 1 ml into the measuring cell of an oscillating densimeter thermostated to maintain the reference temperature of 15 °C.

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[6] Drawn up on the basis of the PN-EN 15293 standard: Motor vehicle fuels — Ethanol fuel (E85) for motor vehicles — Requirements and test methods.

1.1. The method of determination of density at 15 °C, the reagents used, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results as well as the precision of the method, and the method of drawing up the test report are laid down in the Polish standard implementing the EN ISO 12185 standard.

2. Oxidative stability is determined by the induction period method under accelerated oxidation conditions, by measuring the time from the start of oxidation to the breakpoint, using a pressure bomb device.

2.1. The test sample is oxidised in a pressure bomb device previously filled with oxygen at 15 °C to 25 °C and at the pressure of 690 kPa, and heated to the temperature from 90 °C to 102 °C. The pressure is read continuously or at equal intervals until the breakpoint is reached.

2.2. The time from the start of oxidation until the breakpoint is reached is equal to the induction period at its determination temperature from which the induction period at 100 °C is calculated.

2.3. The method of determination of oxidative stability, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN ISO 7536 standard.

3. The testing of the corrosive effect on copper is carried out comparatively to standardised corrosion benchmarks.

3.1. A copper plate is immersed in the test sample of a specific volume and then heated under strictly specified conditions. After the heating is finished, the copper plate is removed, washed and its colour assessed by comparing it with corrosion benchmarks.

3.2. The method of testing the corrosive effect on copper, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, the precision of the method and the method of drawing up the test report are laid down in the PN-EN ISO 2160 standard.

4. Total acidity (expressed as acetic acid content) is determined by colorimetric titration, by mixing the sample with the same portion of water free of carbon dioxide and titration with potassium hydroxide solution in the presence of phenolphthalein until acidic compounds are neutralised.

4.1. The method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 15491 standard.

5. The electrical conductivity is determined by measuring electrical conductivity by means of a conductivity meter using a measuring vessel, at sample temperature (25 ±0.1) °C.

5.1. The method of determination of electrical conductivity, the type of apparatus used and its preparation, and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 15938 standard.

6. The methanol content is determined by capillary gas chromatography consisting in:

1) introduction of the test sample into a high-polarity capillary column, separation of the sample into methanol, other oxygen compounds and hydrocarbons, and detection of methanol by a flame ionisation detector, or

2) introduction of the test sample into the pre-column, separation of methanol and ethanol and light hydrocarbons from the test sample, and subsequent treatment with the analytical column and detection of methanol by a flame-ionization detector.

6.1. For the determination of methanol content with the method referred to in subparagraph 6(1), the method of determination, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the Polish standard implementing the EN 16761-1 standard.

6.2. For the determination of methanol content with the method referred to in subparagraph 6(2), the method of determination, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the Polish standard implementing the EN 16761-2 standard.

7. The content of higher saturated monohydric alcohols (C3–C5) is determined by multi-dimensional gas chromatography using switching columns, consisting in separating the sample into individual groups and then detecting them with a flame ionisation detector.

7.1. The method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in Procedure B of the PN-EN ISO 22854 standard.

8. The appearance is inspected visually by assessing the colour and transparency and comparing them with a sample of water against a white and black background.

8.1. The appearance is determined at ambient temperature or at 15 °C, whichever is higher, and before any colouring.

8.2. The method of determination, the type of apparatus used and its preparation, the method of calculation and presentation of the results, and the method of drawing up the test report are laid down in the PN-EN 15769 standard.

9. Water content is determined with the following method:

1) Karl Fischer coulometric titration, which consists in the introduction of a weighed sample into the titration vessel of Karl Fischer titrator, where Karl Fischer reaction iodine settles electrolytically on the anode, proportionally to the amount of water contained in the sample, or

2) potentiometric titration, which consists in the introduction of a weighed sample into the Karl Fischer potentiometer titration vessel, in which the water present is titrated using a Karl Fisher reagent.

9.1. For the determination of water content with the method referred to in subparagraph 9(1):

1) when all water is titrated, the excess iodine is detected by the electrometric endpoint sensor and the titration is interrupted; based on the stoichiometry of the reaction, one mole of iodine reacts with one mole of water, therefore the amount of water is proportional to the total charge according to Faraday's law;

2) the method of determination, the reagents and materials used, the type of apparatus used and its preparation as well as the preparation of the sample, apparatus control test, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 15489 standard.

9.2. For the determination of water content with the method referred to in subparagraph 9(2):

1) the reaction iodine is introduced in the presence of sulphur dioxide, methanol and the corresponding nitrogenous base; based on the stoichiometry of reaction, one mole of iodine reacts with one mole of water;

2) the method of determination, the reagents and materials used, the type of apparatus used and its preparation and the preparation of the sample, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 15692 standard.

10. Non-organic chloride content is determined through ion chromatography consisting in evaporating a sample in water bath, dissolving the dry residue in water and determining the chloride content by comparing the peak surface of the test sample aqueous solution with the reference calibration curve.

10.1. The method of determination of non-organic chloride content, the reagents used, the type of apparatus used and its preparation, the preparation of the calibration solution, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 15492 standard.

11. Phosphorus content is determined by spectrometry, by evaporating the sample, dissolving the dry residue in water and adding an acidic solution containing molybdenum and antimony ions to obtain an antimony-phosphorus-molybdenum complex.

11.1. The method of determination of phosphorus content, except for the procedure laid down in subparagraph 11.2, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN 15487 standard.

11.2. The procedure replacing the method of determination referred to in subparagraphs 8.1 to 8.3 of the PN-EN 15487 standard is set out in Annex A to the PN-EN 15293 standard.

12. Sulphur content is determined by X-ray fluorescence spectrometry with wave dispersion, which consists in exposing a test sample placed in a cuvette to primary radiation generated by an X-ray tube.

12.1. Sulphur content is determined based on the measured count rate of X-ray fluorescent radiation S $K\text{-}L_{2\text{-}3}$ lines and the background radiation.

12.2. The method of determination of sulphur content, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the Polish standard implementing the EN 16997 standard.

13. Sulphate content is determined through ion chromatography consisting in evaporating a sample in water bath, dissolving the dry residue in water and determining the sulphate ion content by comparing the peak surface of the test sample with the reference curve.

13.1. The method of determination of sulphate content, except for the procedure laid down in subparagraph 13.2, the type of apparatus used and its preparation, the precision of the method, and the method of drawing up the test report are laid down in the PN-EN 15492 standard.

13.2. The procedure replacing the method of determination referred to in subparagraphs 9.1(3) and (4) of the PN-EN 15492 standard is set out in Annex A to the PN-EN 15293 standard.

14. Vapour pressure is determined with the following method:

1) air-saturated vapour pressure (ASVP), which consists in injecting a cooled, air-saturated sample of a known volume into a vacuum chamber or into a chamber in which vacuum is formed by the displacement of a movable piston after the introduction of the sample, and leaving the sample to reach thermal equilibrium at a test temperature of 37.8 °C; or

2) three-fold expansion, consisting in the aspiration of a known volume sample into a measuring chamber with a built-in piston at 20 °C, sealing the chamber and raising the temperature to 37.8 °C with the first expansion with a piston followed by two successive expansions to the final test volume five times the volume of the analytical sample and measuring the total vapour pressure after each expansion.

14.1. For the determination of vapour pressure with the method referred to in subparagraph 14(1):

1) the resulting total chamber pressure is equivalent to the sample vapour pressure and partial dissolved air pressure, and is measured by a pressure sensor with an indicator; the measured total vapour pressure can be converted into dry vapour pressure equivalent (DVPE) by applying the correlation equation;

2) the method of determination, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results and the

precision of the method, as well as the method of drawing up the test report are laid down in the Polish standard implementing the EN 13016-1 standard.

14.2. For the determination of vapour pressure with the method referred to in subparagraph 14(2):

1) partial pressure originating from dissolved air (PPA) for an analytical sample is calculated on the basis of three obtained pressures; the vapour pressure of the analytical sample is calculated by subtracting the partial pressure originating from dissolved air (PPA) from the final total vapour pressure; the calculated vapour pressure is converted to dry vapour pressure equivalent (DVPE) by applying the correlation equation;

2) the method of determination, the type of apparatus used and its preparation as well as sample preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN 13016-3 standard.

15. The content of ethanol and higher saturated alcohols is determined by multi-dimensional gas chromatography using switching columns, consisting in separating the sample into individual groups and then detecting them with a flame ionisation detector.

15.1. The method of determination, the reagents and materials used, the type of apparatus used and its preparation, the method of calculation and presentation of the results and the precision of the method, as well as the method of drawing up the test report are laid down in the PN-EN ISO 22854 standard.

16. The criteria set out in PN-EN ISO 4259-2 are used to interpret the test results referred to in subparagraphs 1 to 15.1.