
Petroleum products — Determination of turbidity point and aniline point equivalent

Produits pétroliers — Détermination du point de turbidité et d'un équivalent du point d'aniline

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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Contents

	Page
Foreword.....	iv
Introduction.....	v
1 Scope.....	1
2 Normative references.....	1
3 Terms and definitions.....	1
4 Principle.....	2
5 Reagents and materials.....	2
6 Apparatus.....	2
7 Sampling.....	2
8 Preparation of test sample.....	2
9 Procedure.....	3
10 Evaluation and expression of results.....	3
11 Precision.....	4
11.1 General.....	4
11.2 Repeatability, r	4
11.3 Reproducibility, R	4
12 Conversion from turbidity point to aniline point equivalent.....	4
13 Test report.....	4
Annex A (informative) Summary description of the precision study.....	6
Bibliography.....	8

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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

This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*.

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

Introduction

This document is intended to be a complement, not a replacement, to ISO 2977^[1] for the determination of aniline point and mixed aniline point.

The same apparatus is used for ISO 2977^[1], wherein method 5 is used to determine the turbidity point. It is also possible to convert the turbidity point to the aniline point equivalent and vice versa. The aniline point equivalent is useful when comparing results from using the method described in this document to the aniline point according to ISO 2977^[1], method 5.

The turbidity point and the aniline point equivalent are useful as an aid in the analysis of hydrocarbon mixtures. Aromatic hydrocarbons exhibit the lowest values and paraffins the highest, with cycloparaffins and olefins exhibiting intermediate values. In a homologous series, the turbidity point and the aniline point equivalent increase with increasing molecular mass.

Although the turbidity point and the aniline point equivalent can be used in combination with other physical properties in correlative methods for hydrocarbon analysis, the most frequent usage is to provide an estimate of the aromatic content (or "aromaticity") of hydrocarbon mixtures.

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WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to its application, and to determine the applicability of any other restrictions for this purpose.

1 Scope

This document specifies a method to determine the turbidity point of petroleum products based on distillates from crude oil.

This document also specifies how to convert the turbidity point to an aniline point equivalent.

This document describes a procedure using automated or automatic apparatus suitable for transparent samples with an initial boiling point above ambient temperature.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 3170, *Petroleum liquids — Manual sampling*

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

3.1 turbidity point

τ

minimum equilibrium solution temperature, in degrees Celsius, of a mixture of equal volumes of *p*-anisaldehyde and the product under test

3.2 aniline point equivalent

$\hat{A}_{eq}$

calculated temperature based on the *turbidity point* (3.1)

4 Principle

Specified volumes of *p*-anisaldehyde and sample are placed in a tube and are mixed mechanically. The mixture is heated at a controlled rate until the two phases become miscible. The mixture is then cooled at a controlled rate and the temperature at which the two phases separate is recorded as the turbidity point.

5 Reagents and materials

5.1 *p*-Anisaldehyde, purity of 97,5 % or better, giving a $\tau = 73,06 \pm 1,576$ °C when tested with heptane (5.3) in accordance with the procedure described in [Clause 9](#).

5.2 Drying agent, consisting of anhydrous calcium sulfate or sodium sulfate.

5.3 Heptane, spectroscopic or HPLC grade with a mass fraction of 98% or higher. Alternative grades with the same mass fraction may be used.

6 Apparatus

6.1 Turbidity point apparatus, automated or automatic, with

- a device for the detection of the change in sample turbidity,
- a means of heating the sample/*p*-anisaldehyde mixture, and
- a thermometer or temperature sensor conforming to the measurement sensitivity and accuracy described in [6.2](#).

The automatic apparatus should be designed in such a way that it is also possible to use the apparatus for ISO 2977^[1], method 5.

6.2 Thermometers, provided with current calibration certificates, traceable to national standards, and giving corrections over the range to 0,02 °C.

6.3 Pipettes, conforming to ISO 648 Class A, and provided with indirect suction. Capacities required are 10,0 ml.

6.4 Balance, accurate to 0,01 g, suitable for weighing the tube and the sample when the sample cannot be pipetted conveniently.

6.5 Safety goggles, manufactured using safety glass.

6.6 Safety gloves, impervious to *p*-anisaldehyde.

7 Sampling

Unless otherwise specified, samples shall be taken by the procedures described in ISO 3170 or ISO 3171.

8 Preparation of test sample

If the sample has a high moisture content, dry the sample by shaking vigorously for 3 min to 5 min with approximately a volume fraction of 10 % of the drying agent (5.2).

Reduce the viscosity of viscous or waxy samples by warming to a temperature below that which would cause the loss of light ends or the dehydration of the drying agent.

If suspended water is visibly present, centrifuge the sample to remove the water before carrying out the final drying with drying agent.

Remove any suspended drying agent by centrifuge or filtration.

Heat samples containing separated wax until they are homogenous and keep heated during the centrifuging or filtration operations.

9 Procedure

9.1 Clean and dry the apparatus.

9.2 Pipette $10 \text{ ml} \pm 0,02 \text{ ml}$ of *p*-anisaldehyde (5.1) and $10 \text{ ml} \pm 0,02 \text{ ml}$ of the sample into the test tube.

If the material is too viscous for pipetting, weigh to the nearest 0,01 g, a quantity of the sample corresponding to $10 \text{ ml} \pm 0,02 \text{ ml}$ at room temperature.

9.3 Prepare the apparatus in accordance with the manufacturer's instructions.

Use the expected temperature for the sample as guidance for setting up the apparatus. If the sample is unknown, perform an exploratory scan on the sample to determine the approximate turbidity point, and use that temperature for the immediate repeat analysis of the same material.

9.4 Stir the *p*-anisaldehyde-sample mixture rapidly, avoiding the inclusion of air bubbles and, if necessary, heating at a rate of $1 \text{ }^{\circ}\text{C}/\text{min}$ to $3 \text{ }^{\circ}\text{C}/\text{min}$ until complete miscibility is obtained by applying heat directly to the jacket tube.

If the *p*-anisaldehyde-sample mixture is completely miscible at room temperature, substitute a non-aqueous cooling bath for the heat source.

9.5 Allow the mixture to cool slowly at a rate of between $0,5 \text{ }^{\circ}\text{C}/\text{min}$ to $1 \text{ }^{\circ}\text{C}/\text{min}$, during continuous stirring, to a temperature $1 \text{ }^{\circ}\text{C}$ to $2 \text{ }^{\circ}\text{C}$ below the temperature at which turbidity first appears according to 9.6.

9.6 Record as the turbidity point the temperature, to the nearest $0,1 \text{ }^{\circ}\text{C}$, at which the mixture suddenly becomes cloudy throughout.

NOTE 1 This temperature, and not the temperature of the separation of small amounts of material, is the minimum equilibrium solution temperature.

NOTE 2 The true turbidity point is characterized by a turbidity which increases sharply as the temperature is lowered.

9.7 Repeat the observation of the turbidity point three times by heating and cooling of the same sample.

10 Evaluation and expression of results

If the range of the three successive determinations does not exceed $0,5 \text{ }^{\circ}\text{C}$, compute and report the average of the three determinations as the turbidity point, to the nearest $0,1 \text{ }^{\circ}\text{C}$.

If the range of the three successive determinations do exceed $0,5 \text{ }^{\circ}\text{C}$, investigate the equipment, reagent, and overall execution of the test method. Repeat the test after investigations.

If after several attempts the maximum range of the three determinations still exceeds $0,5 \text{ }^{\circ}\text{C}$, the test method and equipment is deemed to have failed the qualification requirement.

11 Precision

11.1 General

The precision, as determined by statistical examination in accordance with ISO 4259-1^[2] of interlaboratory test results on petroleum products based on distillates from crude oil with test results in the range (59 °C to 124 °C), is given in [11.2](#) and [11.3](#).

The results of the interlaboratory test are given in [Annex A](#), for information.

11.2 Repeatability, r

The difference between two independent results obtained in the normal and correct operation of the same method, for test material considered to be the same, within a short interval of time, under the same test conditions, that is expected to be exceeded with a probability of 5 % due to random variation, conforms to the value given in [Formula \(1\)](#):

$$r = f_r(X) = 0,50\text{ °C} \quad (1)$$

where X is the average of the two test results being compared.

11.3 Reproducibility, R

The difference between two independent results obtained in the normal and correct operation of the same method, for test material considered to be the same, under different test conditions, that is expected to be exceeded with a probability of 5 % due to random variation, conforms to the value given in [Formula \(2\)](#):

$$R = f_R(X) = 1,64\text{ °C} \quad (2)$$

where X is the average of the two test results being compared.

12 Conversion from turbidity point to aniline point equivalent

To obtain a predicted aniline point equivalent for a measured turbidity point in the range of 66 °C to 124 °C, [Formula \(3\)](#) shall be used:

$$\hat{A}_{eq} = 0,926\tau + 5,63 \quad (3)$$

EXAMPLE

τ measured using the method in this document = 115,5 °C

$$\hat{A}_{eq} = 0,926 \times 115,5 + 5,63 = 112,6\text{ °C}$$

NOTE This functional relationship between the $\hat{A}_{eq}$ and the τ has been determined using a weighted regression approach where the errors from test method ISO 2977^[1], method 5, and the method in this document were taken into consideration.

13 Test report

The test report shall contain at least the following information:

- reference to this document, i.e. ISO 21493:2019;
- type and complete identification of the product tested;

- c) result of the test ([Clause 10](#));
- d) date of the test.

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Annex A (informative)

Summary description of the precision study

A.1 Pilot study

Prior to the interlaboratory study (ILS), a pilot study with four participants and five samples was performed to estimate the number of labs and samples required to satisfy ISO 4259-1^[2] for the precision statement for the turbidity point. The requirements for ASTM D6708^[3] for the conversion of the turbidity point to the aniline point equivalent were also considered.

Based on the results in the pilot study, it was decided that a minimum of sixteen labs and ten samples were required to fulfil the requirements in both ISO 4259-1 and ASTM D6708.

A.2 Interlaboratory study

For the main ILS, twelve samples with two repeats were selected in the turbidity range from 59 °C to 124 °C. The number of samples was increased to twelve to provide some margin of error for possible outliers. A total of seventeen labs participated in the ILS.

All samples in the ILS were petroleum products based on distillates from crude oil and a summary of the sample types can be found in [Table A.1](#).

Table A.1 — Summary of the sample types used in the ILS

Sample	Turbidity point °C	Aniline point ^a °C	Density ^b @ 15 °C kg/m ³	Viscosity ^c @ 40 °C mm ² /s	ASTM color ^d	Sample type
1	59,2	70,8	949	404,9	8,0	Treated distillate aromatic extract (TDAE)
2	66,3	66,9	861	2,9	<0,5	Naphthenic
3	71,4	71,4	820	2,0	<0,5	Diesel
4	76,1	77,9	923	62,2	>8	Vacuum gas oil (VGO)
5	80,2	79,8	880	8,7	<0,5	Naphthenic
6	87,6	87,2	881	15,0	>8	Vacuum gas oil (VGO)
7	91,7	90,5	868	10,8	<0,5	Group II paraffinic
8	97,5	97,4	923	374,2	3,0	Naphthenic
9	104,6	102,2	893	95,5	<0,5	Naphthenic
10	115,5	113,2	884	111,2	2,0	Group I paraffinic
11	122,4	118,0	840	24,8	<0,5	Group III paraffinic
12	124,0	120,0	889	227,3	4,0	Group I paraffinic

^a According to ISO 2977:1997^[1], method 5.

^b According to ISO 12185:1996^[4].

^c According to ISO 3104:1994^[5].

^d According to ISO 2049:1996^[6].