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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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Foreword

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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.

Petroleum products — Determination of turbidity point and aniline point equivalent

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 τ = 73,06 ± 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 ml ± 0,02 ml of *p*-anisaldehyde (5.1) and 10 ml ± 0,02 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 ml ± 0,02 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 °C/min to 3 °C/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 °C/min to 1 °C/min, during continuous stirring, to a temperature 1 °C to 2 °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 °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 °C, compute and report the average of the three determinations as the turbidity point, to the nearest 0,1 °C.

If the range of the three successive determinations do exceed 0,5 °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 °C, the test method and equipment is deemed to have failed the qualification requirement.