

Methods of test for

Petroleum and its products

**Part 2. Petroleum products and hydrocarbon
solvents - Determination of aniline point and
mixed aniline point**

(Identical with IP 2/98)

National foreword

This British Standard was published under the authority of the Materials and Chemicals Sector Board and comes into effect on 1st March 1998. It is the English language version of ISO 2977 : 1997 *Petroleum products and hydrocarbon solvents - Determination of aniline point and mixed aniline point*, published by the International Organization for Standardization (ISO).

This British Standard supersedes BS 2000 : Part 2 : 1994, which is withdrawn.

BS 2000 comprises a series of test methods for petroleum and its products that are published by the Institute of Petroleum (IP) and have been accorded the status of a British Standard. Each method should be read in conjunction with the preliminary pages of 'IP Standard methods for analysis and testing of petroleum and related products and British Standard 2000 Parts' which gives details of the BSI/IP agreement for publication of the series, provides general information on safety precautions, sampling and other matters, and lists the methods published as Parts of BS 2000.

Under the terms of the agreement between BSI and the Institute of Petroleum, BS 2000 : Part 2 / ISO 2977 will be published by the IP (in 'Standard methods for analysis and testing of petroleum and related products and British Standard 2000 Parts' and as a separate publication). The numbering of the Parts of BS 2000 follows that of the corresponding IP methods. BS 2000 : Part 2 : 1998 is thus identical with IP 2/98.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

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The following BSI references
relate to the work of this
standard:

Committee reference PTI/13
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Petroleum products and hydrocarbon solvents - Determination of aniline point and mixed aniline point

WARNING - The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the determination of the aniline point of petroleum products and hydrocarbon solvents, and the mixed aniline point of those products having aniline points below the temperature at which aniline will crystallize from the aniline-sample mixture.

Method 1 describes a procedure for transparent samples with an initial boiling point above ambient temperature, and for those with an aniline point below the bubble point and above the solidification point of the aniline-sample mixture.

Method 2, a thin film method, describes a procedure for samples too dark for testing by method 1.

Methods 3 and 4 are for samples that may vaporize appreciably at the aniline point.

NOTE 1 Method 4 is particularly suitable where only small quantities of sample are available.

Method 5 describes a procedure using automated or automatic apparatus suitable for the range covered by methods 1 and 2.

NOTE 2 The aniline point (or mixed aniline point) is useful as an aid in the characterization of pure hydrocarbons and 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 aniline points increase with increasing molecular mass.

NOTE 3 Although the aniline point 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.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 648: 1977, *Laboratory glassware - One-mark pipettes*.

ISO 2049: 1996, *Petroleum products - Determination of colour (ASTM scale)*.

3 Definitions

For the purposes of this International Standard, the following definitions apply:

3.1 aniline point: The minimum equilibrium solution temperature, in degrees Celsius, of a mixture of equal volumes of aniline and the product under test.

3.2 mixed aniline point: The minimum equilibrium solution temperature, in degrees Celsius, of a mixture of two volumes of aniline, one volume of the product under test and one volume of heptane.

3.3 bubble point: The temperature, in degrees Celsius, noted at the moment when bubbles first appear in the body of the mixture when heated under standardized conditions.

4 Principle

Specified volumes of aniline and sample, or aniline and sample plus heptane, are placed in a tube and 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 aniline point or mixed aniline point.

5 Reagents and materials

5.1 Aniline

CAUTION - Aniline is extremely toxic, even in very small quantities, and is absorbed through the skin. Handle with great caution. Wear goggles of safety glass and gloves impervious to aniline for all operations where aniline is handled directly.

Dry analytical reagent grade aniline over potassium hydroxide pellets, decant and distil fresh on the day of use, discarding the first and last 10 % (V/V). Aniline thus prepared shall give an aniline point of $69,3\text{ }^{\circ}\text{C} \pm 0,2\text{ }^{\circ}\text{C}$ as determined from the average of two independent tests having a difference of not more than $0,1\text{ }^{\circ}\text{C}$ when tested with heptane (5.3) in accordance with the procedure described in 8.1.

As an alternative to distilling the aniline on the day of use, distil the aniline as described above and collect the distillate in ampoules which are then sealed under vacuum or dry nitrogen and stored in a cool dark place for future use. In either case, rigid precautions shall be taken to avoid contamination by atmospheric moisture (see 6.2).

NOTE 4 Experience shows that, under these conditions, the aniline should remain unchanged for at least six months.

NOTE 5 For routine analysis, distillation is not mandatory provided that the aniline meets the requirements of the test with heptane.

NOTE 6 For the purposes of this International Standard, the term "% (V/V)" is used to represent the volume fraction of a material.

The aniline point of aniline and heptane determined with automated or automatic apparatus shall be $69,3\text{ }^{\circ}\text{C} \pm 0,2\text{ }^{\circ}\text{C}$ when corrected using the formula given in E.4.1.

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

5.3 Heptane, of minimum purity 99.75 %.

6 Apparatus

6.1 Aniline point apparatus, conforming to the individual method requirements as detailed in:

- annex A for method 1;
- annex B for method 2;
- annex C for method 3;
- annex D for method 4;
- annex E for method 5.

NOTE 7 Alternative apparatus may be used, such as the U-tube method for dark oils, provided that it has been shown to give results of the same precision as given in clause 10 and accuracy as defined by the aniline point of heptane (see 5.1), as those described in annex A to annex E.

6.2 Heating and cooling bath, consisting of a suitable air bath, a bath using a non-aqueous, non-volatile transparent liquid, or an infrared lamp (250 W to 375 W), provided with means for controlled heating.

Water shall not be used as either a heating or a cooling medium, since aniline is hygroscopic and moist aniline will give erroneous test results.

NOTE 8 As an example, the aniline point of dry aniline with heptane is increased by approximately $0,5\text{ }^{\circ}\text{C}$ if the aniline contains 0,1 % (V/V) water.

If the aniline point is below the dew point of the atmosphere, pass a slow stream of dry inert gas into the aniline point tube to blanket the aniline-sample mixture.

6.3 Thermometers, conforming to the specifications in annex F. The thermometers shall be provided with current calibration certificates, traceable to national standards, and giving corrections over the range to $0,02\text{ }^{\circ}\text{C}$.

6.4 Pipettes, conforming to ISO 648 Class A, and provided with indirect suction. Capacities required are 10,0 ml, 5,0 ml and 0,5 ml. The 0,5 ml pipette (method 4) shall be provided with a long fine tip.

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

6.6 Safety goggles, manufactured using safety glass.

6.7 Safety gloves, impervious to aniline.

7 Preparation of test sample

Dry the sample by shaking vigorously for 3 min to 5 min with approximately 10 % (V/V) 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.

8. Procedure

8.1 Choice of method

Five methods, to be used as applicable, are specified as follows:

Method 1: described in detail in annex A, is applicable to clear, light-coloured samples and to samples not darker than 6,5 by ISO 2049, having initial boiling points well above the expected aniline point.

Method 2: described in detail in annex B, is applicable to light-coloured samples, to moderately dark samples, and to very dark samples.

NOTE 9 Method 2 is applicable to samples too dark to be tested by method 1.

Method 3: described in detail in annex C, is applicable to clear samples and to samples not darker than 6,5 by ISO 2049, having initial boiling points sufficiently low to give incorrect aniline point readings by method 1.

NOTE 10 An example is aviation gasoline.

Method 4: described in detail in annex D, is a small-scale method, applicable to the same type of sample as method 3, and is applied when only limited quantities of sample are available.

Method 5: is applicable when using automated or automatic apparatus in accordance with the instructions in annex E.

8.2 Mixed aniline point

The procedure is applicable to samples having aniline points below the temperature at which aniline crystallizes from the mixture. Pipette 10 ml of aniline, 5 ml of the sample and 5 ml of heptane (5.3) into a clean, dry

apparatus. Determine the aniline point of the mixture by method 1 or method 2 as described in annex A or annex B.

9 Evaluation and expression of results

9.1 If the range of three successive observations of the aniline point temperature is not greater than 0,1 °C for light-coloured samples or 0,2 °C for dark samples, report the average of these temperature readings, corrected for thermometer calibration errors, to the nearest 0,05 °C, as the aniline point or mixed aniline point.

9.2 If such a range is not obtained after five observations, repeat the test using fresh quantities of aniline and sample in a clean, dry apparatus; if consecutive temperature observations show a progressive change, or if the range of observations is greater than 0,16 °C for light coloured samples, or 0,03 °C for dark samples, report the method as being inapplicable.

10 Precision

The precision of the method, as obtained by statistical examination of inter-laboratory test results is given in 10.1 and 10.2 below. The precision values, with the exception of those for clear, light-coloured samples, were not obtained from recent co-operative tests.

10.1 Repeatability, r

The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the normal and correct operation of the test method, exceed the values below only in one case in twenty.

Aniline point of:

— clear light-coloured samples $r = 0,16\text{ °C}$

— moderately dark to very dark samples $r = 0,3\text{ °C}$

Mixed aniline point of:

— clear light-coloured samples $r = 0,16\text{ °C}$

— moderately dark to very dark samples $r = 0,3\text{ °C}$

10.2 Reproducibility, R

The difference between two single and independent results obtained by different operators working in different laboratories on nominally identical test material would, in the normal and correct operation of the test method, exceed the values below only in one case in twenty.