

British Standard Methods of test for

Petroleum and its products

Part 2. Aniline point and mixed aniline point of petroleum products and hydrocarbon solvents

(Identical with IP 2/78)

Méthodes d'essai pour le pétrole et les produits pétroliers

Partie 2. Point d'aniline et point d'aniline en mélange des produits pétroliers et des solvants hydrocarbonés

Prüfmethoden für Erdöl und Erdölprodukte

Teil 2. Anilipunkt und Mischanilinpunkt von Erdölprodukten und Kohlenwasserstoff-lösungsmitteln

Foreword

BS 2000 comprises a series of test methods for petroleum and its products that have been published previously by the Institute of Petroleum (IP) and have been accorded the status of British Standards. Each method should be read in conjunction with BS 2000 : Part 0 'General introduction', which gives details of the BSI/IP agreement for publication of the series, lists more than 60 methods scheduled to be published in this way in 1982, and provides general information on safety precautions, sampling and other matters.

The numbering of the other Parts follows that of the corresponding methods published in 'IP Standards for petroleum and its products, Part 1 Methods for analysis and testing'. BS 2000 : Part 2 is thus identical with IP 2/78. IP 2/61 was previously published in A5 format as BS 4715* and has now been re-numbered and issued in the BS 2000 series. It is intended to issue further revisions when necessary in line with changes that may be made in subsequent editions of the IP method.

When published in 1972, BS 4715 was identical with IP 2/61 and covered only four manual methods for determining aniline point. This Part of BS 2000, however, also includes a procedure using an automatic apparatus and a method for determining mixed aniline point, both of which methods were added when IP 2 was revised in 1978.

This Part is similar in many respects to ISO 2977-1974, but differs technically from that standard. This method is also under consideration by the European Committee for Standardization (CEN) as prEN 5.

Caution. Attention is drawn to the safety precautions mentioned in Part 0 of this British Standard, and also to the specific hazards indicated in 5.1, 8.1, A1.2.1, A2.2.1, A3.2.1 and A4.2.1.

NOTE. The titles of the publications referred to in this Part of this British Standard are listed on the inside back cover.

* Now withdrawn.

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Method (identical with IP 2/78¹).

1. SCOPE

1.1. This method describes procedures for the determination of the aniline point of petroleum products and hydrocarbon solvents. Method A is suitable for transparent samples with an initial boiling point above room temperature and where the aniline point is below the bubble point and above the solidification point of the aniline-sample mixture. Method B, a thin film method, is suitable for samples too dark for testing by Method A. Methods C and D are for samples that may vaporize appreciably at the aniline point. Method D is particularly suitable where only small quantities of sample are available. Method E describes a procedure using an automatic apparatus suitable for the range covered by Methods A and B.

1.2. This method also covers the determination of the mixed aniline point of petroleum products and hydrocarbon solvents having aniline points below the temperature at which aniline will crystallize from the aniline-sample mixture.

2. DEFINITIONS

2.1 *Aniline point* – the minimum equilibrium solution temperature for equal volumes of aniline and sample.

2.2 *Mixed aniline point* – the minimum equilibrium solution temperature of a mixture of two volumes of aniline, one volume of sample, and one volume of *n*-heptane of specified purity.

3. SUMMARY OF METHOD

3.1. Specified volumes of aniline and sample, or aniline and sample plus *n*-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 two phases separate is recorded as the aniline point or mixed aniline point.

4. APPARATUS

4.1. For details of the aniline point apparatus required for each method see:

Appendix A1 for Method A
Appendix A2 for Method B
Appendix A3 for Method C
Appendix A4 for Method D
Appendix A5 for Method E

NOTE 1: Alternative apparatus may be used, such as the U-tube method for dark oils, provided it has been shown to give results of the same precision and accuracy as those described in the Appendix.

4.2. *Heating and Cooling Bath* – A suitable air bath, a non-volatile, transparent liquid bath, or an infrared lamp (250 to 375 W), provided with means for controlling the rate of heating.

NOTE 2: Water should not be used as either a heating or cooling medium since aniline is hygroscopic and moist aniline will give erroneous test results. For example, the aniline point of the *n*-heptane reagent as measured with aniline containing 0.1% water is approximately 0.5°C (0.9°F) higher than that measured with dry aniline. 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.

4.3. *Thermometers* – having the following ranges and conforming to the requirements of the designated ASTM or IP specification:

Range	ASTM (E 1) ²	IP ³
–38°C (–36.5°F) to +42°C (+107.5°F)	33C, 33F	20C
25°C (77°F) to 105°C (221°F)	34C, 34F	21C
90°C (194°F) to 170°C (338°F)	35C, 35F	59C

NOTE to 4.3 and footnote 3.
Specification details for IP thermometers 20C, 21C and 59C are given in BS 2000 : Part 0 : Addendum 1.

¹ This method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and in the IP it is under the jurisdiction of Standardization Committee

² 1974 Annual Book of ASTM Standards, Parts 25 and 44.

³ IP Specifications for Standard Thermometers.

4.4. *Pipettes* – with capacities of 10 ± 0.04 ml and 5 ± 0.02 ml. Provide a rubber suction bulb for use with pipettes when measuring aniline.

4.5. *Balance* – A laboratory balance sensitive to 0.01 g, suitable for weighing the tube and sample when the sample cannot be pipetted conveniently.

4.6. *Safety Goggles*.

4.7. *Plastic Gloves*, impervious to aniline.

NOTE to 4.4. Pipettes complying with BS 1583 are suitable.

5. REAGENTS

5.1. *Aniline* – Dry chemically pure aniline over potassium hydroxide pellets, decant, and distill fresh on the day of use, discarding the first and last 10%. Aniline thus prepared when tested with *n*-heptane according to Section 7 shall give an aniline point of $69.3 \pm 0.2^\circ\text{C}$ ($156.7 \pm 0.4^\circ\text{F}$) as determined from the average of two independent tests having a difference of not more than 0.1°C (0.2°F).

NOTE 3: For routine purposes the distillation process is not mandatory provided the aniline meets the requirements of the test with *n*-heptane.

NOTE 4: The aniline point of aniline and *n*-heptane determined with automatic apparatus (Method E) shall be $69.3 \pm 0.2^\circ\text{C}$ ($156.7 \pm 0.4^\circ\text{F}$) when corrected in accordance with the equation in Section A5.2.1.

NOTE 5: As an alternative to distilling the aniline on the day of use, the aniline may be distilled as described in 5.1, collecting the distillate in ampoules, sealing the ampoules under vacuum or dry nitrogen, and storing in a cool dark place for future use. In either case, rigid precaution must be taken to avoid contamination from atmospheric moisture (Note 2). It is believed that under these conditions the aniline will remain unchanged for a period exceeding 6 months.

CAUTION: *Aniline is extremely toxic and must not be sucked into a pipette with the mouth. Aniline is also toxic by absorption through the skin, even in very small quantities, and must consequently be handled with great caution.*

5.2. *Anhydrous Sodium Sulphate*.

5.3. *Anhydrous Calcium Sulphate*.

5.4 *n*-Heptane, conforming to the requirements listed in Table 1⁴.

TABLE 1 Requirements for *n*-Heptane

ASTM Motor Octane Number ^a	0.0 ± 0.2
Density at 20°C , ^b g/ml	0.68380 ± 0.00015
Refractive index, ^c $n_D^{20^\circ\text{C}}$	1.38770 ± 0.00015
Freezing point, ^d deg C	-90.710 min
Distillation ^e	
50% recovered, deg C (at 760 mm Hg)	98.427 ± 0.025
Differential, 80% recovered minus 20% recovered, deg C	0.020 max.

^a Determined in accordance with ASTM Method D2700/IP 236 Test for Knock Characteristics of Motor and Aviation-Type Fuels by the Motor Method.⁵

^b Determined in accordance with ASTM Method D1217, Test for Density and Specific Gravity of Liquids by Bingham Pycnometer.⁵

^c Determined in accordance with ASTM Method D1218, Test for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids.⁵

^d Determined in accordance with ASTM Method D1015, Test for Freezing Points of High-Purity Hydrocarbons.⁵

^e For equipment and method used, see *Journal of Research*, Nat. Bureau of Standards, Vol. 44, No. 3, 1950, pp. 309 and 310 (RP2079).

6. SAMPLE

6.1. Dry the sample by shaking vigorously for 3 to 5 min with about 10% by volume of a suitable drying agent such as anhydrous calcium sulphate or anhydrous sodium sulphate. Reduce the viscosity of viscous samples by warming to a temperature below that which would cause the loss of light ends or the dehydration of the drying agent. Remove any suspended drying agent by use of a centrifuge or by filtration. Heat samples containing separated wax until they are homogeneous and keep heated during filtration or centrifugation to ensure against separation of wax. When suspended water is visibly present and the sample material is known to dissolve less than 0.03 weight per cent of water, the use of a centrifuge for the removal of suspended water is an acceptable procedure.

⁴ These requirements for *n*-heptane are identical, except for tetraethyllead, with those prescribed in the 1974–75 ASTM Manual for Rating Motor, Diesel and Aviation Fuels.

⁵ 1974 Annual Book of ASTM Standards, Part 23.

⁶ ASTM Manual for Rating Motor, Diesel and Aviation Fuels, Fourth Edition, 1974–75.

7. PROCEDURE FOR ANILINE POINT

7.1. The following methods, to be used as applicable, are covered as follows:

Method A, described in detail in Appendix A1 is applicable to clear samples or to samples not darker than No. 6.5 ASTM colour, as determined by ASTM D1500/IP 196, Test for ASTM Colour of Petroleum Products (ASTM Colour Scale)⁵ having initial boiling points well above the expected aniline point.

Method B, described in detail in Appendix A2, is applicable to light coloured samples, moderately dark samples and to very dark samples. It is suitable for samples that are too dark to be tested by Method A.

Method C, described in detail in Appendix A3 is applicable to clear samples or to samples not darker than No. 6.5 ASTM colour, as determined by ASTM D1500/IP 196, Test for ASTM Colour of Petroleum Products (ASTM Colour Scale) having initial boiling points sufficiently low as to give incorrect aniline point readings by Method A, e.g. aviation gasoline.

Method D, described in detail in Appendix A4 is applicable to the same type of sample as Method C. It is particularly useful when only limited quantities of sample are available.

Method E, applicable when using automatic apparatus in accordance with the instructions in Appendix A5.

NOTE to 7.1. BS 5859, which is the dual-numbered version of ISO 2049, is technically equivalent to IP 196.

8. PROCEDURE FOR MIXED ANILINE POINT

8.1. This procedure is applicable to samples having aniline points below the temperature at which aniline crystallizes from the mixture. Pipette 10 ml of aniline (**CAUTION** – see Section 5.1), 5 ml of sample and 5 ml of *n*-heptane into a clean, dry apparatus. Determine the aniline point of the mixture by Method A or B as described in Appendixes A1 or A2.

9. REPORT

9.1. If the range of three successive observations of the aniline point temperature is not greater than 0.1°C (0.2°F) for light coloured samples or 0.2°C (0.4°F) for dark samples, report the average temperature of these observations, corrected for thermometer calibration errors to the nearest 0.05°C (0.1°F) as the 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 and if consecutive temperature observations show a progressive change, or if the range of observations is greater than the repeatability given in 10.1, report the method as being inapplicable.

NOTE to 9.1. Test results should be reported as having been obtained in accordance with BS 2000 : Part 2.

10. PRECISION

10.1. The following criteria should be used for judging the acceptability of results (95% confidence):

10.1.1. *Repeatability* – Duplicate results, that is, two average temperatures obtained by the same operator in a series of observations as described under Section 9 should be considered suspect if they differ by more than the following amounts:

Repeatability

Aniline point of:	
Clear, light-coloured samples	0.16°C (0.3°F)
Moderately dark to very dark samples	0.3°C (0.6°F)*
Mixed aniline point of:	
Clear, light-coloured samples	0.16 (0.3°F)*
Moderately dark to very dark samples	0.3°C (0.6°F)*

10.1.2. *Reproducibility* – When a result is submitted by each of two laboratories, these two results should be considered suspect if they differ by more than the following amounts:

Reproducibility

Aniline point of:	
Clear, light-coloured samples	0.5°C (0.9°F)
Moderately dark to very dark samples	1.0°C (1.8°F)*
Mixed aniline point of:	
Clear, light-coloured samples	0.7°C (1.3°F)*
Moderately dark to very dark samples	1.0°C (1.8°F)*

* Not determined from recent cooperative tests; however, the ratios with those given in the 1953 version of ASTM method D611 are believed to apply.