INTERNATIONAL STANDARD ISO 2977

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Petroleum products and hydrocarbon solvents — Determination of aniline point and mixed aniline point

Produits pétroliers et solvants hydrocarbonés — Détermination du point d'aniline et du point d'aniline en mélange

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2977 was drawn up by Technical Committee ISO/TC 28, *Petroleum products*, and circulated to the Member Bodies in March 1973.

It has been approved by the Member Bodies of the following countries :

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Petroleum products and hydrocarbon solvents — Determination of aniline point and mixed aniline point

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1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the test procedures for determining the aniline point of petroleum products and hydrocarbon solvents, provided the aniline point is below the bubble point and above the solidification point of the aniline-sample mixture.

It also provides a procedure for determining 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 REFERENCE

ISO 2049, *Petroleum products — Determination of colour.*

3 DEFINITIONS

3.1 aniline point : The lowest temperature at which equal volumes of aniline and the product under test are completely miscible under standardized conditions.

3.2 mixed aniline point : The lowest temperature at which two volumes of aniline and one volume of the product under test and one volume of *n*-heptane are completely miscible under standardized conditions.

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

4 PRINCIPLE

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 the two phases separate is recorded as the aniline point or mixed aniline point.

5 REAGENTS

5.1 Aniline

Dry aniline of analytical reagent quality over potassium hydroxide pellets, decant, and distill fresh on the day of use, discarding the first and last 10 %. Aniline thus prepared shall give an aniline point of 69,3 ± 0,2 °C as determined from the average of two independent tests having a difference of not more than 0,1 °C when tested with *n*-heptane according to clause 8.

As an alternative to distilling the aniline on the day of use, the aniline may be distilled as described above, the distillate being collected 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 must be taken to avoid contamination from atmospheric moisture (see note to 6.2). Experience shows that under these conditions, aniline should remain unchanged for at least 6 months.

NOTES

1 The aniline point of aniline and *n*-heptane determined with automatic apparatus shall be 69,3 ± 0,2 °C when corrected in accordance with the equation in C.2.1.

2 **CAUTION : *Aniline should not be pipetted directly by mouth because of its extreme toxicity. Aniline is also toxic by absorption through the skin, even in very small quantities, and should be handled with great caution.***

5.2 Calcium sulphate, anhydrous, or **sodium sulphate**, anhydrous.

5.3 *n*-Heptane, conforming to the following requirements as determined by the specified or equivalent methods.

Motor octane number (ASTM D2700)	0,0 ± 0,2
Density at 20 °C (ASTM D1217)	0,683 80 ± 0,000 15 g/ml
Refractive index $n_D^{20°C}$ (ASTM D1218)	1,387 70 ± 0,000 15

Freezing point (ASTM D1015)	− 90,710 °C min.
Distillation[1] :	
50 % recovered (at 1,013 bar)	98,427 ± 0,025 °C
Differential, 80 % recovered minus 20 % recovered	0,020 °C max.

6 APPARATUS

6.1 Test tube or **thin-film apparatus**, made of heat-resistant glass and conforming to the dimensions prescribed in annexes A and B respectively.

Automatic aniline point apparatus is permitted if the requirements of note 1 to 5.1 are fulfilled (see annex C).

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

NOTE – Water must 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 dry aniline is increased from that specified by approximately 0,5 °C if the aniline reagent contains 0,1 % of 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.

6.3 Aniline point thermometers, partial immersion type, having ranges of − 38 to + 42 °C, 25 to 105 °C, and 90 to 170 °C and conforming to the specifications in the table below.

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Thermometer specifications

Range	− 38 to + 42 °C	25 to 105 °C	90 to 170 °C
Immersion	51 mm	51 mm	51 mm
Graduation at each	0,2 °C	0,2 °C	0,2 °C
Longer lines at each	1 °C	1 °C	1 °C
Figured at each	2 °C	2 °C	2 °C
Scale error, not to exceed	0,2 °C	0,2 °C	0,2 °C
Expansion chamber permitting heating to	100 °C	150 °C	220 °C
Overall length	419 ± 5 mm	419 ± 5 mm	419 ± 5 mm
Stem diameter	6 to 7 mm	6 to 7 mm	6 to 7 mm
Bulb length	10 to 20 mm	10 to 19 mm	10 to 20 mm
Bulb diameter	5 to 6 mm	5 to 6 mm	5 to 6 mm
Distance from bottom of bulb to line at	− 35 °C : 112 to 124 mm	25 °C : 106 to 120 mm	90 °C : 101 to 115 mm
Distance from bottom of bulb to line at	40 °C : 352 to 377 mm	105 °C : 358 to 379 mm	170 ° C : 358 to 384 mm
Distance from bottom of bulb to top of contraction chamber	–	–	35 mm (max.)

1) For equipment and method used, see Journal of Research, U.S. National Bureau of Standards, Volume 44, No. 3, 1950, pp. 309 and 310 (RP2079).

6.4 Pipettes, with capacities of 10 ± 0,04 ml and 5 ± 0,02 ml (see note). Provide a rubber suction bulb for use with one of the 10 ml pipettes when measuring aniline.

NOTE – The 10 and 5 ml one-mark pipettes specified in ISO/R 648, Class B, and ISO/R 648, Class A, respectively are suitable.

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, to guard against eye splashes.

7 PREPARATION OF THE TEST SAMPLE

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 centrifugation or filtration. When suspended water is visibly present and when the sample material is known to dissolve less than 0,03 % of water by mass, centrifuging for the removal of suspended water is an acceptable procedure.

8 PROCEDURE

8.1 Choice of method

Three 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 or to samples not darker than No. 6.5 colour, as determined according to ISO 2049.

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

Method 3 : applicable when using automatic apparatus in accordance with instructions in annex C.

8.2 Procedure for mixed aniline point

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 note 2 to 5.1), 5 ml of the sample, and 5 ml of *n*-heptane into a clean, dry apparatus. Determine the aniline point of the mixture by method 1 as described in annex A.

9 PRECISION[1)]

The following criteria shall be used for judging the acceptability of results (95 % confidence level) :

9.1 Repeatability

Duplicate results, that is, two average temperatures obtained by the same operator in a series of observations as specified in clause 10, shall be considered suspect if they differ by more than the following amounts :

	Repeatability
Aniline point of :	
– clear, light-coloured samples	0,2 °C
– moderately dark to very dark samples	0,3 °C
Mixed aniline point of :	
– clear, light-coloured samples	0,2 °C
– moderately dark to very dark samples	0,3 °C

9.2 Reproducibility

When a result is submitted by each of two laboratories, these two results shall be considered suspect if they differ by more than the following amounts :

	Reproducibility
Aniline point of :	
– clear, light-coloured samples	0,5 °C
– moderately dark to very dark samples	1,0 °C
Mixed aniline point of :	
– clear, light-coloured samples	0,7 °C
– moderately dark to very dark samples	1,0 °C

10 TEST REPORT

10.1 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 temperature of these observations, corrected for thermometer calibration errors, to the nearest 0,1 °C as the aniline point.

10.2 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 0,2 °C for light-coloured samples or 0,3 °C for dark samples, report the method as being inapplicable.

1) The precision of the method for the determination of the aniline point of moderately dark to very dark samples and the precision of the method for the determination of the mixed aniline point were not determined from recent co-operative tests. Co-operative tests are being carried out. It is believed that the results of these tests will contribute towards obtaining reliable values.

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ANNEX A

METHOD 1

A.1 APPARATUS

The apparatus shown in figure 1 shall consist of the following :

A.1.1 Test-tube, approximately 25 mm in diameter and 150 mm in length, made of heat-resistant glass.

A.1.2 Jacket, approximately 37 to 42 mm in diameter and 175 mm in length, made of heat-resistant glass.

A.1.3 Stirrer, manually operated, of soft iron wire, approximately 2 mm in diameter, as shown in figure 1, having a concentric ring of diameter approximately 19 mm at the bottom. The length of the stirrer to a right-angle bend shall be approximately 200 mm. The right-angled portion shall be approximately 55 mm long. A glass sleeve approximately 65 mm in length and of 3 mm inside diameter shall be used as a guide for the stirrer. Any suitable mechanical device for operating the stirrer as specified is an approved alternative for the manual operation.

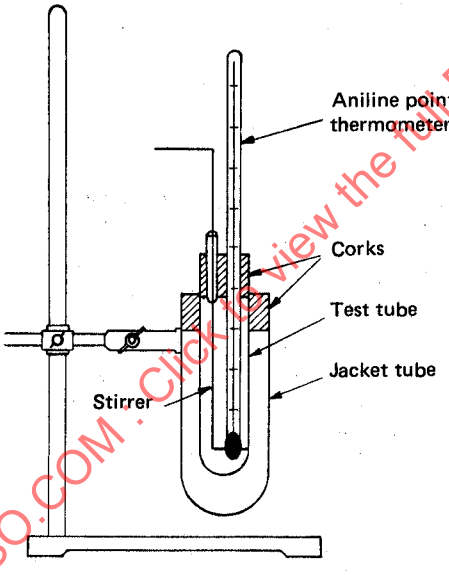

FIGURE 1 — **Aniline point apparatus**

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A.2 PROCEDURE

A.2.1 Clean and dry the apparatus. Pipette 10 ml of aniline (**Caution** : See note 2 to 5.1) and 10 ml of the dry sample into the air-jacketed test tube fitted with stirrer and thermometer. If the material is too viscous for pipetting, weigh, to the nearest 0,01 g, a quantity of the sample corresponding to 10 ml at room temperature. Centre the thermometer in the test tube so that the immersion mark is at the liquid level, making sure that the bulb does not touch the side of the tube.

A.2.2 Stir the sample rapidly, using a 50 mm stroke, avoiding the inclusion of air bubbles and, if necessary, heating at a rate of approximately 1 to 3 °C per minute until complete miscibility is obtained, by applying heat directly to the jacket tube. If the aniline-sample mixture is completely miscible at room temperature, substitute a non-aqueous cooling bath for the heating source. Continue stirring and allow the mixture to cool slowly at a rate of 0,5 to 1,0 °C per minute. Continue cooling to a temperature of 1 to 2 °C below the first appearance of turbidity and record as the aniline point the temperature, to the nearest 0,1 °C, at which the mixture suddenly becomes cloudy throughout (see note). This will be the minimum equilibrium solution temperature and not the temperature of separation of minor portions of the samples.

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

A.2.3 Repeat the observation of aniline point temperature by heating and cooling repeatedly until a report as specified in clause 10 can be made.

ANNEX B

METHOD 2

B.1 APPARATUS

Thin-film apparatus made of heat-resistant glass and stainless steel, conforming to the dimensions given in figure 2. A suggested assembly is shown in figure 3.

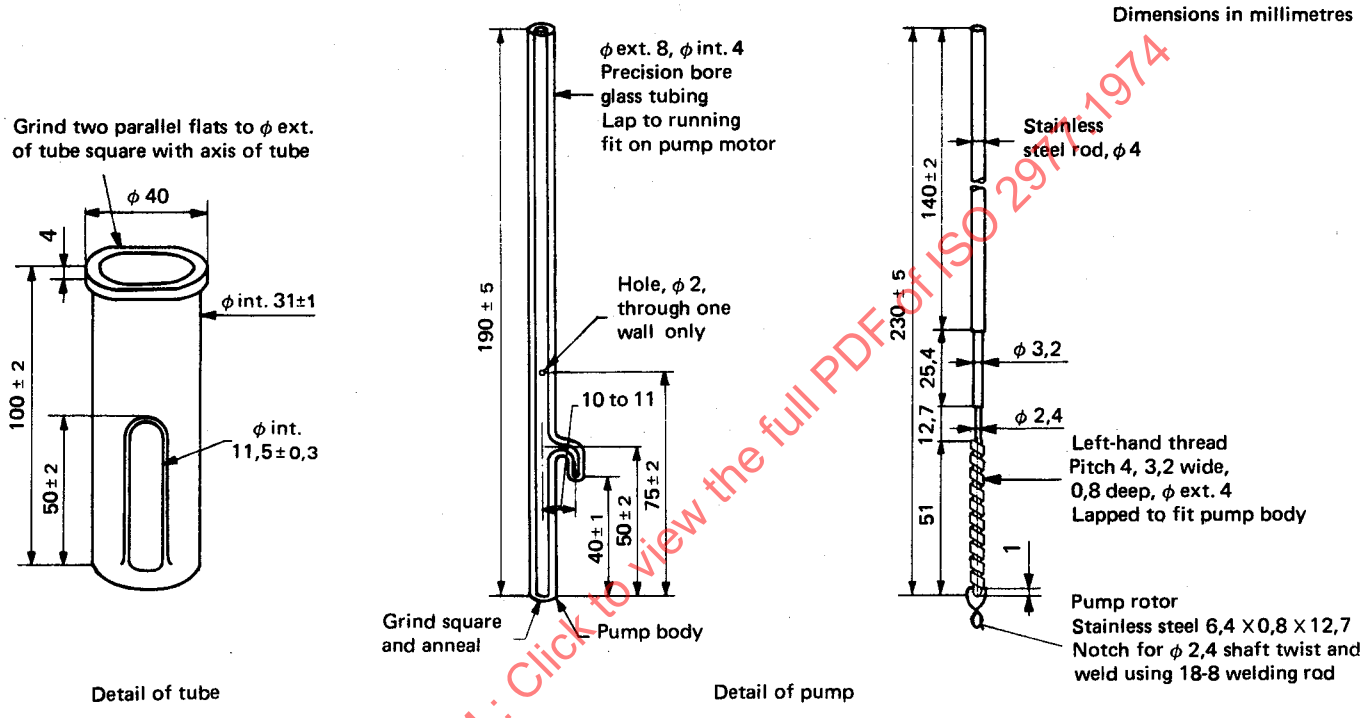

FIGURE 2 – **Details of aniline point thin-film apparatus**

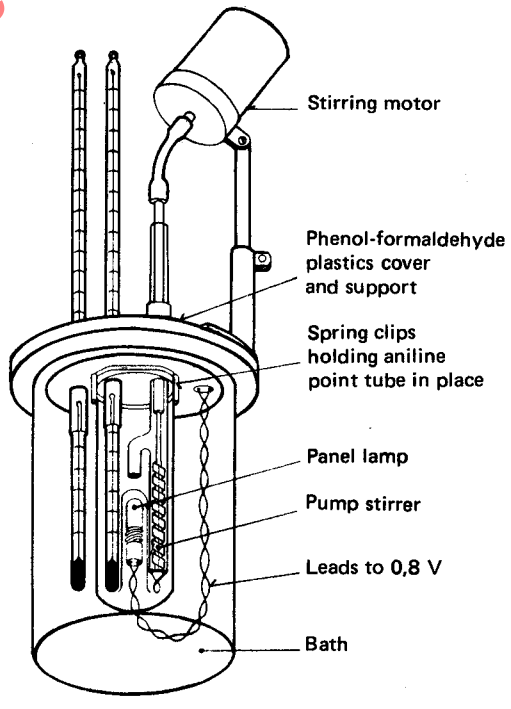

FIGURE 3 – **Assembly of thin-film apparatus**

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B.2 PROCEDURE

B.2.1 Clean and dry the apparatus. Pipette 10 ml of aniline (**Caution** : See note 2 to 5.1) and 10 ml of the sample into the air-jacketed tube fitted with stirrer and thermometer. If the material is too viscous for pipetting, weigh to the nearest 0,01 g, a quantity of sample corresponding to 10 ml at room temperature. Place the thermometer in the tube so that the contraction chamber is below the liquid level, and so that the mercury bulb does not touch the side of the tube.

B.2.2 Adjust the speed of the pump to produce a continuous stream of the sample-aniline mixture in the form of a thin film flowing over the light well. With extremely dark samples, operate the pump slowly and lower it so that the delivery tube nearly touches the top of the light well, thus obtaining a film that is continuous and thin enough to permit observation of the aniline point. Adjust the voltage on the lamp until just enough light is given for the filament to be visible through the film. Raise the temperature of the mixture at a rate of 1 to 2 °C per minute until the aniline point has just been passed as denoted by a definite, sudden brightening of the lamp filament, and by the disappearance of the more or less opalescent condition of the film (see note). Discontinue heating and adjust the lamp voltage so that the filament appears clear and distinct but not uncomfortably bright to the eye. Adjust the temperature of the bath so that the sample-aniline mixture cools at a rate of 0,5 to 1,0 °C per minute and note the appearance of the film and light filament. Record as the aniline point the temperature, to the nearest 0,1 °C, at which a second phase appears as evidenced by the reappearance of the opalescent condition of the film (usually causing a halo to appear around the lamp filament) or by a sudden dimming of the lamp filament, or both. At temperatures above the aniline point, the edge of the light filament appears clear and distinct. At the aniline point temperature, a halo or haze forms around the filament replacing the distinct lines of the filament edge with lines which appear cloudy or hazy. Further darkening of the cloud over the filament occurs at lower temperature, but is not to be confused with the aniline point.

NOTE – For those making the test for the first time, the following procedure may be helpful : Make preliminary operational adjustments and tests, using a colourless sample-aniline mixture and observing the changes taking place in the body of the liquid and film. Make rough tests with dark oils to become familiar with the appearance of the film and light source as the mixture passes from the clear state above the aniline point to the translucent state below. If the sample is such that there is difficulty in observing the exact point of the phase change, make experiments with the sample, using various intensities of light and paying particular attention to the appearance of the light in the immediate vicinity of the lamp filament.

B.2.3 Repeat the observation of aniline point temperature by heating and cooling repeatedly until a report as specified in clause 10 can be made.

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ANNEX C

METHOD 3

C.1 APPARATUS

Automatic aniline point apparatus, commercially available, using a modified thin-film technique and direct heating of the sample with electrical immersion heater. Detection of change of sample turbidity at the aniline point is by response of a photoelectric cell to collimated light directed through the thin film of sample.

C.2 PROCEDURE

C.2.1 Determine the automatic aniline point in accordance with instructions provided with the apparatus. Correct the aniline point as follows :

$$\text{Corrected aniline point} = \frac{X_a - A}{B}$$

where

X_a is the automatic aniline point;

A and B are constants determined for each apparatus as described in C.2.2.

NOTE – It has been established by co-operative tests that observed aniline points determined by some automatic apparatus are lower than those found by methods 1 and 2. The difference is greater for automatic apparatus when relatively high sample-cooling rates are used, and increases as the aniline point increases.

C.2.2 Determine the aniline point by either method 1 or method 2 using the automatic apparatus for three or more samples with aniline points in each of the ranges 43 to 50 °C, 60 to 65 °C and 75 to 80 °C. Calculate A and B by the least squares method by simultaneous solution of the following equations :

$$\Sigma(X_a) = NA + B\Sigma(X_c)$$

$$\Sigma(X_a X_c) = A\Sigma(X_c) + B\Sigma(X_c^2)$$

where

$\Sigma(X_a)$ is the sum of all aniline point data by automatic apparatus;

$\Sigma(X_c)$ is the sum of all aniline point data by either method 1 or method 2;

$\Sigma(X_c^2)$ is the sum of the squares of all aniline point data by either method 1 or method 2;

$\Sigma(X_a X_c)$ is the sum of the products of aniline point data by either method 1 or method 2 using the automatic apparatus for each sample;

N is the number of samples.

NOTE – Co-operative data were obtained from five laboratories for five samples with aniline points in the range 34 to 87 °C.

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