BS 5117 : Section 1.3 : 1985

UDC 661.17 : 621.43 – 715.4 : 620.197.3

British Standard

Testing corrosion inhibiting, engine coolant concentrate ('antifreeze')

Part 1. Methods of test for determination of physical and chemical properties

Section 1.3 Determination of freezing point

Essais du liquide de refroidissement anti-rouille du moteur (antigel)
Partie 1. Méthodes d'essai de détermination des propriétés physiques et chimiques
Section 1.3 Détermination du point de congélation

Prüfung von korrosionshemmendem Kühlmittelkonzentrat für Motoren (Frostschutzmittel)
Teil 1. Prüfverfahren zur Bestimmung der physikalischen und chemischen Eigenschaften
Abschnitt 1.3 Bestimmung des Gefrierpunkts

NOTE. It is recommended that this Section be read in conjunction with the information given in the 'General introduction', published separately as BS 5117 : Part 0.

Contents

1 Scope

This Section of BS 5117 describes a method for the determination of the freezing point of solutions of engine coolant concentrate.

NOTE 1. The engine coolant concentrate is referred to hereafter as 'the product'.

NOTE 2. The method as described is intended for the determination of the freezing point of solutions prepared from the product as supplied but the procedure may be adapted for solutions obtained from engine cooling systems, test rigs, etc.

NOTE 3. The titles of the publications referred to in this standard are listed on the inside back page.

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

2 Principle

A test portion is cooled under specified conditions; a cooling graph is plotted from which is obtained the freezing point.

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3 Reagents

3.1 *General.* The reagents used shall be of a recognized analytical grade. Water complying with BS 3978 shall be used throughout.

3.2 *Propan-2-ol.*

3.3 *Solid carbon dioxide.*

4 Apparatus

4.1 *General.* The apparatus described in **4.2** to **4.8** is required. The items described in **4.4** to **4.7** are shown in figure 1.

4.2 *One-mark volumetric flask,* of 100 mL capacity, complying with class A of BS 1792.

4.3 *Burette,* of 50 mL capacity, complying with class A of BS 846.

4.4 *Cooling bath,* consisting of a Dewar flask, capacity approximately 2 L, firmly mounted in a close fitting container and containing a pad of glass wool.

4.5 *Test portion holder,* consisting of an unevacuated, unsilvered Dewar flask, capacity 200 mL, fitted with a stopper through which are bored three holes. The central hole accommodates the thermometer (**4.6**), and the side holes permit the passage of the stirrer (**4.7**) and the seeding wire (**4.8**). The latter hole, 6 mm to 7 mm in diameter, is closed with a stopper when not in use.

4.6 *Thermometer,* designated A10C/100, complying with BS 593, mounted so that the tip of the bulb is about 10 mm from the base of the test portion holder (**4.5**).

4.7 *Vertically agitated stirrer,* mechanically operated, made of stainless steel wire 1.6 mm diameter, having five coils spaced so as to remain totally immersed in the test portion during the determination. Its action is such that it just clears the base of the test portion holder (**4.5**) at the bottom of its vertical stroke.

4.8 *Seeding wire,* made of stainless steel, by means of which small quantities of frozen test solution may be introduced into the test portion during the determination.

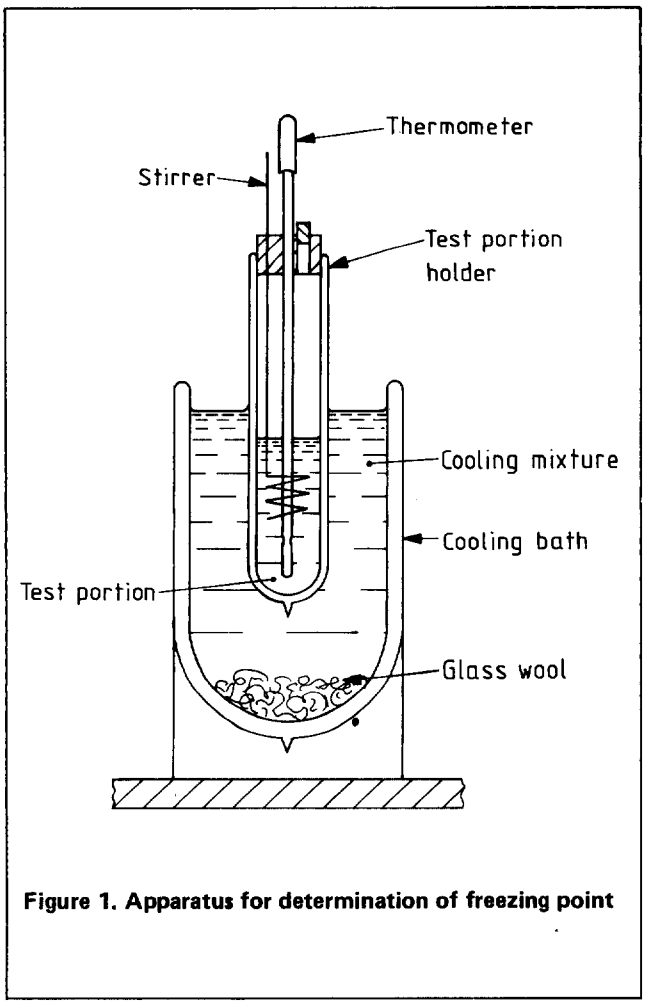


Figure 1. Apparatus for determination of freezing point

5 Sampling of the product and preparation of test solution

5.1 Sampling

Take a representative sample of not less than 500 mL, preferably from previously unopened containers in which the product is normally offered for sale*. Place the sample in clean, dry, stoppered glass bottles of a dark colour. Agitate all containers before sampling to ensure homogeneity of the contents. Where a batch of containers is to be sampled, it is essential that the number of containers sampled is not less than the cube root of the number of containers in the batch. Prepare the final sample by taking equal portions from each container sampled and mix them together thoroughly. Take care to ensure that any method used for sealing the sample does not cause contamination.

NOTE. A series of different tests may be carried out by using separate portions taken from one sample.

5.2 Preparation of test solution

Prepare the test solution at 50 % (*V*/*V*) concentration, or as otherwise specified, as follows.

Using the burette (**4.3**) measure accurately the required volume of the sample into the one-mark volumetric flask (**4.2**) and make up to the mark with water. Stopper the flask and invert it several times to mix the solution.

*See **A.2** of BS 5117 : Part 0 : 1985 and clauses **4** and **5** of BS 3195 : Part 1 : 1978, for further guidance on sampling procedures and equipment.

6 Procedure

Assemble the apparatus. Fill the cooling bath (**4.4**) surrounding the test portion holder (**4.5**) with some of the propan-2-ol (**3.2**) and add small lumps of the solid carbon dioxide (**3.3**) to lower the temperature of the cooling bath to about 8 °C to 10 °C below the anticipated freezing point of the test solution (**5.2**). Ensure that a layer of the solid carbon dioxide at least 13 mm thick is maintained in the cooling bath throughout the determination.

Temporarily remove the stopper from the test portion holder (**4.5**) and add a test portion of about 80 mL of the test solution (**5.2**), the approximate freezing point of which has previously been determined by cooling rapidly.

NOTE. The test portion may be precooled to about 8 °C above the anticipated freezing point immediately prior to the determination.

Start the stirrer and adjust its rate of stirring to about 60 to 80 strokes/min. Observe the temperature of the test portion at this point and thereafter record the temperature at 30 s intervals, increasing the frequency of recording (if possible to once every 15 s) towards the anticipated freezing point.

At the anticipated freezing point, seed the test portion (to prevent supercooling) by introducing a small quantity of the test solution (**5.2**), frozen on to the tip of the seeding wire (**4.8**). At this stage the rate of cooling shall be less than 1 °C/min.

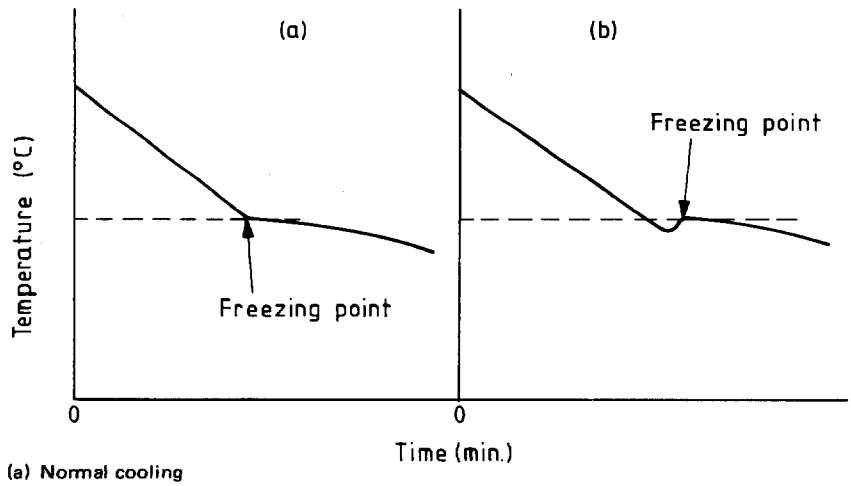
Continue recording the temperature for at least 10 min after the apparent freezing point.

7 Preparation of cooling graph

Plot a graph having, for example, the temperature, expressed in °C, as ordinates and the time, expressed in minutes, as abscissae. Typical graphs are shown in figure 2, the effect of supercooling being illustrated in figure 2(b).

8 Expression of results

The freezing point, expressed in °C to one place of decimals, is read from the cooling graph as shown in figure 2.



(a) Normal cooling

(b) Supercooling

Figure 2. Typical cooling graphs

Publications referred to

BS 593	Laboratory thermometers
BS 846	Specification for burettes
BS 1792	Specification for one-mark volumetric flasks
BS 3978	Water for laboratory use
BS 3195	Methods for sampling petroleum products Part 1 Liquid hydrocarbons : manual sampling
BS 5117	Testing corrosion inhibiting, engine coolant concentrate ('antifreeze') Part 0 General introduction

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Board of BSI and comes into effect on 28 June 1985.

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The Committees responsible for this British Standard are shown in Part 0.

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