BS 5117 : Section 1.1 : 1985

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British Standard

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

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

Section 1.1 Ancillary procedures

Essais du liquide de refroidissement anti-rouille du moteur (antigel)
Partie 1. Méthodes d'essai de détermination des propriétés physiques et chimiques
Section 1.1 Méthodes auxiliaires

Prüfung von korrosionshemmendem Kühlmittelkonzentrat für Motoren (Frostschutzmittel)
Teil 1. Prüfverfahren zur Bestimmung der physikalischen und chemischen Eigenschaften
Abschnitt 1.1 Hilfsverfahren

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 methods for the determination of concentration, pH and reserve alkalinity of aqueous solutions of engine coolant concentrate.

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

NOTE 2. 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 Definitions

For the purposes of this British Standard the definitions given in BS 5117 : Part 0 together with the following apply.

reserve alkalinity. The number of millilitres of hydrochloric acid solution (c(HCl) = 0.1 mol/L) required for titration of a solution containing 10 mL of product to a pH value of 5.5.

3 Determination of concentration of diluted product

3.1 Introduction

This clause describes methods for the determination of the approximate concentration of aqueous solutions of the product.

3.2 Principle

Either the density at 20 °C or the refractive index at 20 °C of a test portion is determined and the concentration is then obtained by reference to tables of the density or refractive index of solutions of the product under test, as appropriate.

3.3 Apparatus

Ordinary laboratory apparatus and the following are required.

British Standards Institution

3.3.1 *Hydrometer,* complying with BS 718, type L 50 or M 50 for medium surface tension liquids, density range 1.0 g/mL to 1.5 g/mL.

3.3.2 *Hydrometer cylinder* and *water bath,* as described in **3.3.2.1** and **3.3.2.2** or *hydrometer tube with water jacket,* as described in **3.3.2.3.**

3.3.2.1 *Hydrometer cylinder,* complying with BS 718.

3.3.2.2 *Water bath,* capable of being controlled at a temperature of 20 ± 0.2 °C, of depth sufficient to immerse the hydrometer cylinder to 25 mm above the level of the test portion in the cylinder.

3.3.2.3 *Hydrometer tube with water jacket,* in which water temperature of 20 ± 0.2 °C, of depth sufficient to immerse the jacket.

3.3.3 *Thermometer,* complying with BS 593, of appropriate range, capable of indicating a temperature of 20 °C to the nearest 0.1 °C.

3.3.4 *Density meter,* capable of determining the density of solutions of the product at 20 °C to ± 0.001.

3.3.5 *Refractometer,* capable of determining refractive index to the nearest 0.001, equipped with means of temperature control.

3.3.6 *Reference tables,* relating concentration of solutions of the product under test to density or refractive index, as appropriate.

NOTE. Published tables are available for certain formulations: otherwise, special reference tables will have to be prepared.

3.3.7 *Source of illumination for refractometer,* preferably a sodium lamp.

3.4 Method using a hydrometer

3.4.1 *Test portion.* Pour sufficient of the diluted product into the hydrometer cylinder to allow the hydrometer to float clear of the bottom.

3.4.2 *Procedure*

3.4.2.1 Carry out the following as described in either (a) or (b).

(a) Place the hydrometer cylinder **(3.3.2.1)** in the water bath **(3.3.2.2)** controlled at a temperature of 20 ± 0.2 °C and adjust the water level to not less than 25 mm above that of the test portion. Stir the test portion gently with the thermometer **(3.3.3)** and observe the temperature until the test portion is uniformly at a temperature of 20 ± 0.2 °C.

(b) If a hydrometer tube with water jacket **(3.3.2.3)** is used, circulate water, at a temperature of 20 ± 0.2 °C, through the jacket and bring the temperature of the test portion to 20 ± 0.2 °C.

3.4.2.2 Remove the thermometer and insert the hydrometer **(3.3.1)** slowly, avoiding the inclusion of air bubbles, until it just floats. Take care not to wet the emergent stem of the hydrometer.

3.4.2.3 After a further 15 min, slightly depress the hydrometer **(3.3.1)** into the test portion and then allow it to return to its floating position.

3.4.2.4 Read the hydrometer, as described in BS 718, to ± 0.001 and record the result.

3.4.2.5 Using the density reference table **(3.3.6)**, read the concentration of the diluted product.

3.5 Method using a density meter

Determine the density of the diluted product at 20 ± 0.2 °C to ± 0.001 by means of the density meter **(3.3.4)**.

Using the density reference table **(3.3.6)**, read the concentration of the diluted product.

3.6 Method using a refractometer

Ensure that the prisms of the refractometer **(3.3.5)** are clean and dry. Circulate water, at a temperature of 20 ± 0.2 °C, through the water jacket of the refractometer.

Place a few drops of the well mixed diluted product on the prisms of the refractometer and, after closing the prism block, read off the refractive index of the solution. Record the result to the nearest 0.001.

Using the reference tables of refractive index, read the concentration of the diluted product.

3.7 Expression of results

The concentration of the diluted product is expressed, to the nearest 0.5, as a percentage by volume [% (*V/V*)].

4 Determination of pH

4.1 Introduction

This clause describes a method for the determination of the pH of aqueous solutions of the product at a specified temperature.

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

4.2 Principle

The product is diluted with water to the specified concentration. The pH of this solution is determined electrometrically at the specified temperature.

4.3 Apparatus

Ordinary laboratory apparatus and the following are required.

4.3.1 *pH meter,* with glass/calomel electrode assembly, complying with BS 2586 and BS 3145.

4.3.2 *Beakers,* tall-form, 100 mL capacity.

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

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

4.3.5 *Thermometer,* general purpose type, complying with BS 1704, of appropriate range, capable of indicating the temperature to an accuracy of ± 1 °C.

4.3.6 *Water bath,* capable of being controlled at the test temperature to within ± 1 °C (optional).

4.4 Reagents

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

4.4.2 *Buffer solutions,* at least two, of appropriate pH values, to standardize the pH meter over the required range.

4.5 Test temperature

Unless otherwise specified, the test temperature shall be 20 °C.

4.6 Sampling of the product and preparation of test solution

4.6.1 *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.

4.6.2 *Preparation of test solution.* Prepare the test solution at 25 % (*V*/*V*) concentration, or as otherwise specified, as follows.

Using the burette (**4.3.4**) measure accurately the required volume of the sample, maintained at the test temperature ± 1 °C, into the one-mark volumetric flask (**4.3.3**). Make up to within about 10 mm of the mark with water. Adjust the temperature of the solution to the test temperature ± 1 °C and carefully make up to the mark with water. Stopper the flask and invert it several times to mix the solution.

4.7 Procedure

4.7.1 *Standardization of the pH meter.* Standardize the pH meter at the test temperature, using the buffer solutions, controlled at the test temperature ± 1 °C, in beakers and following the instrument manufacturer's instructions.

NOTE. Attention is drawn to BS 1647 which may provide further information to that given by the instrument manufacturer's instructions.

4.7.2 *Determination.* Decant about 50 mL of the test solution into a beaker and bring it to the test temperature. Determine the pH of the solution using the standardized pH meter (**4.3.1**). Record the result to the nearest 0.1 pH unit.

4.8 Reporting of results

The pH of the diluted product is reported as follows:

pH of x % (V/V) aqueous solution at t °C = Y

where

x is the percentage by volume of product in the test solution;

t is the temperature at which the pH is measured (in °C);

Y is the pH of the test solution, recorded to the nearest 0.1 pH unit.

5 Determination of reserve alkalinity

5.1 Introduction

This clause describes a method for the determination of the reserve alkalinity of the product.

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

5.2 Principle

A test portion of 10 mL of the product is diluted to about 100 mL with water and titrated potentiometrically with hydrochloric acid solution (c(HCl) = 0.1 mol/L). The pH of the solution is measured using a glass electrode and a calomel reference electrode after each addition of acid. The end point is a pH value of 5.5.

5.3 Reagents

5.3.1 *General.* The reagents used shall be of a recognized analytical grade. Water complying with BS 3978, further purified by removal of dissolved carbon dioxide so that its pH value at 25 °C is between 6.2 and 7.2 and its conductivity is not greater than 200 µS/m, shall be used throughout.

5.3.2 *Hydrochloric acid,* standard volumetric solution, c(HCl) = 0.1 mol/L.

5.4 Apparatus

Ordinary laboratory apparatus and the following are required.

5.4.1 *pH meter,* with glass and calomel electrode assembly, complying with BS 2586 and BS 3145.

*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.

5.4.2 *Stirrer,* mechanical or magnetic.

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

5.5 Sampling of the product

Sample the product as described in **4.6.1.**

5.6 Procedure

5.6.1 *Standardization of the pH meter.* Standardize the pH meter using two buffer solutions having pH values of 4.0 and 6.9 and following the instrument manufacturer's instructions.

NOTE. Attention is drawn to BS 1647 which may provide further information to that given by the instrument manufacturer's instructions.

5.6.2 *Determination.* Transfer by means of a safety pipette a 10 mL test portion from the sample to a 250 mL tall-form beaker and dilute to about 100 mL with the water. (Owing to the viscosity of the product it is important to allow sufficient time for the pipette to drain.) If greater accuracy is required, determine the quantity of the test portion by weighing. Introduce the electrodes into the diluted test portion and stir.

Fill the burette (**5.4.3**) with the hydrochloric acid solution (**5.3.2**) and position it so that the tip is 10 mm below the surface of the diluted test portion. Titrate with stirring, at first adding the hydrochloric acid solution in 1 mL to 2 mL portions and allowing the meter reading to become steady between each addition. As the pH value approaches 5.5, add smaller portions of the acid until the pH value of 5.5 is reached.

5.7 Expression of results

Reserve alkalinity, expressed as millilitres of hydrochloric acid solution, $c(HCl) = 0.1$ mol/L, is the volume, in millilitres, of the hydrochloric acid solution (**5.3.2**) required to titrate the test portion to a pH value of 5.5.

Record the value to the nearest 0.1 mL.

Publications referred to

BS 593	Laboratory thermometers
BS 718	Specification for density hydrometers
BS 846	Specification for burettes
BS 1647	pH scale
BS 1704	Specification for solid-stem general purpose thermometers
BS 1792	Specification for one-mark volumetric flasks
BS 2586	Specification for glass and reference electrodes for the measurement of pH
BS 3145	Specification for laboratory pH meters
BS 3195	Methods for sampling petroleum products Part 1 Liquid hydrocarbons : manual sampling
BS 3978	Water for laboratory use
BS 5117	Testing corrosion inhibiting, engine coolant concentrate ('antifreeze') Part 0 General introduction

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