BRITISH STANDARD

BS 5117-2.1: 1985

Testing corrosion inhibiting, engine coolant concentrate ("antifreeze") —

Part 2: Methods of test for corrosion inhibition performance —

Section 2.1 General procedures

NOTE It is recommended that this Section be read in conjunction with the information given in the "General Introduction" published separately as BS 5117-0.

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Contents

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 6, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This Section of BS 5117 specifies general procedures which are common to the corrosion inhibition tests described in BS 5117-2.

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 cover.

2 Sampling of the product

Take a representative sample of not less than 500 mL, preferably from previously unopened containers in which the product is normally offered for sale[1)] 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.

3 Preparation and assembly of metal test specimens

3.1 Specimens

Each of the metal test specimens shall be 50 mm × 25 mm, with a central 6 mm hole. The specimens taken from cast materials shall be 3 mm thick, and the remainder 1.5 mm thick. The metals[2)] to be used are:

a) *Steel*, complying with class CR 4 or CS 4, general purpose finish, of BS 1449-1.

b) *Copper*, complying with grade Cu-ETP-2 of BS 6017, cut from annealed copper strip complying with the requirements of BS 1432.

c) *Brass*, complying with type CZ 106 of BS 2870, cut from half-hard sheet.

d) *Solder*[3)], containing tin 2.4 % to 2.6 %; silver 0.45 % to 0.55 %; antimony 0.40 % max.; arsenic 0.05 % max.; bismuth 0.15 % max.; copper 0.05 % max.; nickel 0.01 % max.; iron 0.01 % max.; zinc nil; aluminium nil; others 0.10 % max; lead remainder. Either cut or cast solid specimens are suitable.

e) *Aluminium alloy*, complying with type LM4 of BS 1490.

f) *Cast iron*, complying with grade 260 of BS 1452, cut from suitable 30 mm diameter cast bar.

NOTE It is advisable that each metal test specimen be carefully stamped with an identification mark to prevent confusion between similar specimens.

3.2 Preparation of specimens

Rub all specimens except those of solder, on wet sheets of grade P280 silicon carbide waterproof paper complying with BS 871, to give a uniform surface finish. Use separate sheets for each material.

Brush all specimens vigorously, using a moist bristle brush.

Rinse the specimens thoroughly with running tap water, rinse with acetone, dry and weigh to the nearest 1 mg.

Store the specimens in a desiccator for a minimum of 48 h before use. At all times handle the washed specimens only with forceps or clean, grease-free cotton gloves.

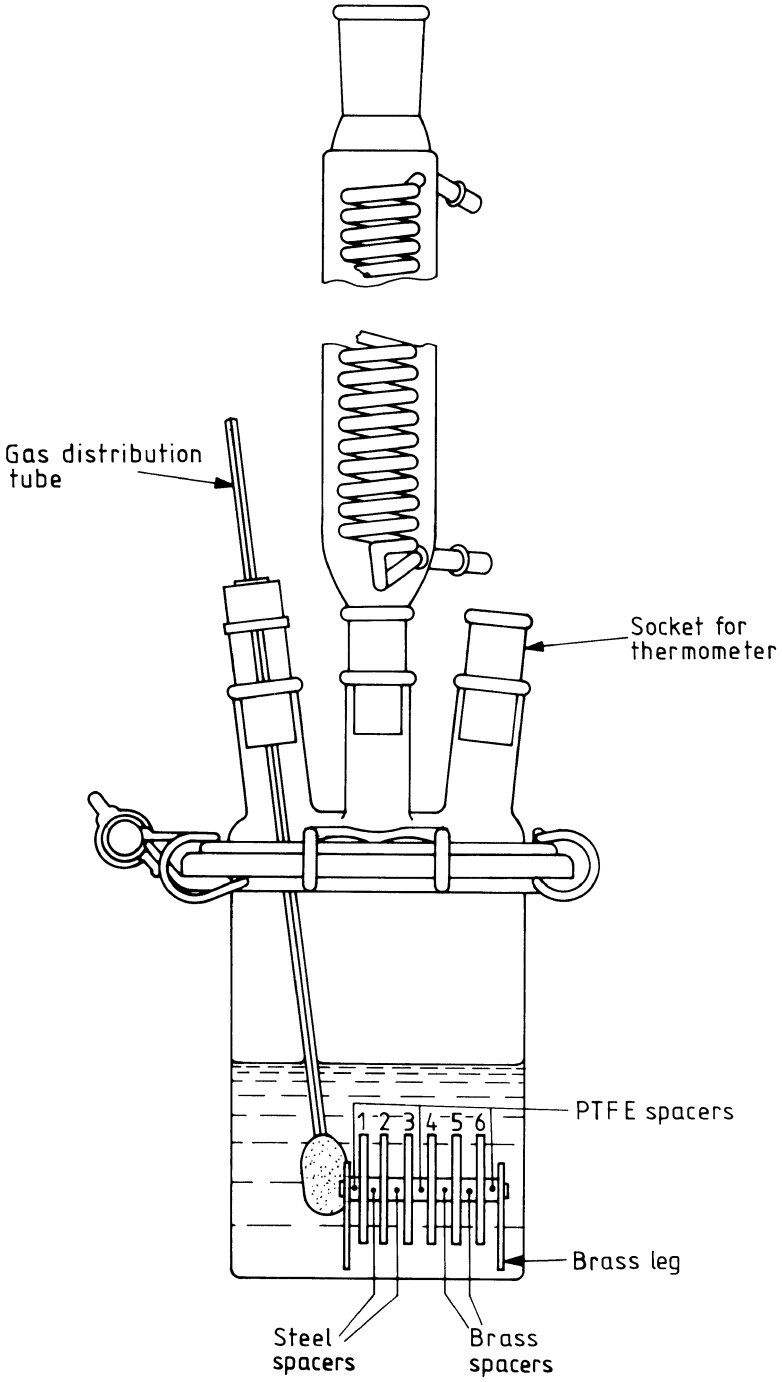
3.3 Assembly of specimens

Assemble the specimens on a non-conducting rod of diameter small enough to slip easily through the central hole in the specimens. Separate the specimens from each other by cylindrical spacers, 5 mm long, 11 mm outside diameter and 6 mm inside diameter. At each end of the assembly place a brass "leg", 50 mm by 25 mm, cut from 1.5 mm brass sheet, and with a hole 6 mm in diameter, off-centre towards one end of the brass piece so that the "legs" are orientated with their long axes in the same directions as the long axes of the specimens (see Figure 1).

NOTE For the recirculating rig test, brass legs are not used (see Figure 2).

Assemble the specimens and "legs" in the following order: brass "leg", copper, solder, brass, steel, cast iron, aluminium alloy, brass "leg". Use spacers of polytetrafluoroethylene (PTFE), for electrical insulation, between the brass "legs" and the adjacent specimens and between the brass and steel specimens; use brass spacers between the brass, solder and copper specimens; use steel spacers between the steel, aluminium alloy and iron specimens.

[1)] See **A.2** of BS 5117-0:1985 and clauses **4** and **5** of BS 3195-1:1978 for further guidance on sampling procedures and equipment.
[2)] For information on the availability of suitable test metals, apply to Enquiry Section (London), British Standards Institution, enclosing a stamped addressed envelope for reply.
[3)] Solder complying with Ford Motors specification M11A 10A has been found suitable.



Key to test specimens

1 Aluminium alloy
2 Cast iron
3 Steel
4 Brass
5 Solder
6 Copper

Figure 1 — Diagram of the glassware test apparatus

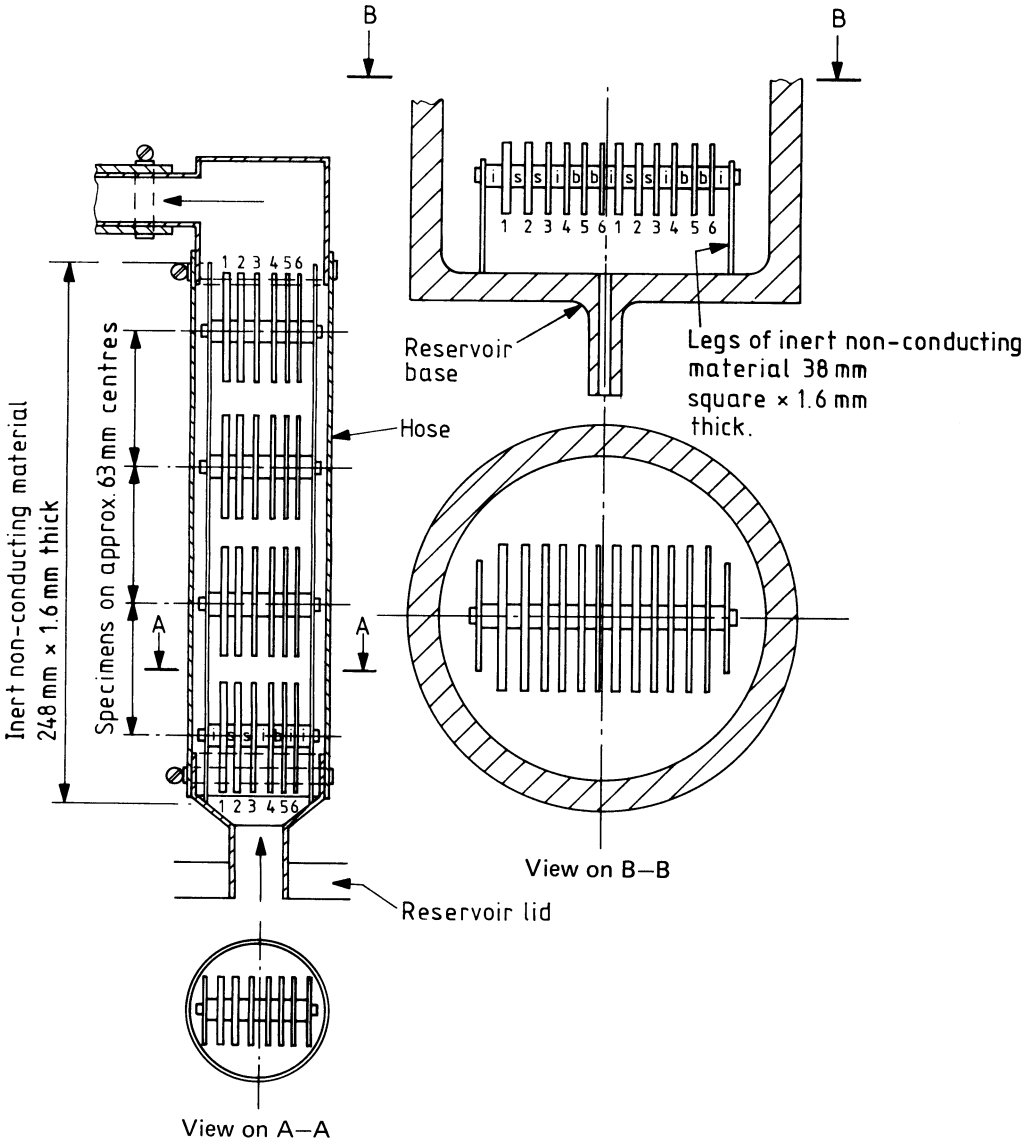


Figure 2 — Recirculating rig test: specimen assembly

Provide an arrangement to tighten the assembly of "legs", spacers and specimens together so as to ensure that the three specimens in each section of the assembly are held firmly in good electrical contact with each other.[4)]

4 Additional cleaning procedures for metal test specimens carrying adherent corrosion products

WARNING. The cleaning solutions described in this clause, and their constituents, are toxic and/or corrosive.

Avoid breathing associated dust or fumes, and prevent contact with the eyes and skin, during preparation and use.

Take appropriate precautions in disposal of waste solutions.

4.1 Reagents

The reagents used shall be of recognized analytical grade. Unless otherwise stated, water complying with BS 3978 shall be used throughout.

[4)] One such arrangement comprises the use of a lock nut and spring washer on the outside of the legs.

4.2 Chemical treatment[5)]

4.2.1 *Iron and steel*

4.2.1.1 *General*. Clean the specimen either electrolytically or by using "Clarke's solution" as described in **4.2.1.2** and **4.2.1.3** respectively.

4.2.1.2 *Electrolytic cleaning*. The specimen is made the cathode in an electrolytic cell using a suitable inert anode, for example lead or graphite. The electrolyte may be an inhibited sulphuric acid solution, prepared as described below, or a 50 g/L solution of *tri*ammonium citrate.

Prepare the inhibited sulphuric acid solution by carefully adding, with stirring, 55 mL of sulphuric acid, approximately 98 % (*m*/*m*) solution, to 945 mL of water contained in a suitable beaker. Then add an appropriate quantity of a suitable organic inhibitor, for example 2 g of 1,3-di-*o*-tolyl-2-thiourea, and stir to dissolve or disperse the inhibitor.

Current is passed through at about 4 A per specimen for 5 min at a solution temperature of 70 °C.

4.2.1.3 *Cleaning using "Clarke's solution"* (hydrochloric acid approximately 36 % (*m*/*m*) solution, containing 5 % of tin(II) chloride and 2 % of *di*antimony trioxide). The specimen is kept agitated during immersion in the derusting solution, for periods of about 20 s to 30 s, rinsing thoroughly with running tap water after each immersion, until complete derusting has been obtained.

4.2.2 *Copper and brass*. Immerse the specimen for 15 s in cold hydrochloric acid solution prepared as follows.

Carefully dilute 430 mL of hydrochloric acid, approximately 36 % (*m*/*m*) solution, to 1 000 mL with water.

4.2.3 *Aluminium alloy*. Immerse the specimen for 5 min in a boiling solution prepared as follows.

Carefully dilute 20 mL of orthophosphoric acid, approximately 88 % (*m*/*m*) solution, to 1 000 mL with water and add either 20 g of chromium(VI) oxide or 30 g of potassium dichromate, stirring to dissolve the reagent.

4.2.4 *Solder*. Immerse the specimen for 5 min in boiling acetic acid solution prepared as follows.

Carefully dilute 12 mL of glacial acetic acid to 1 000 mL with water.

4.3 Washing

After each cleaning treatment described above, wash the specimen in running water, brush with a moist bristle brush, dry and weigh.

4.4 Blank value

When a suitable set of conditions has been established for cleaning a specimen, determine the average mass loss resulting from cleaning three uncorroded specimens and deduct this blank value from all mass losses obtained on similar specimens cleaned in this way.

5 Special procedure for cleaning heavily corroded metal test specimens and determination of mass loss

5.1 Introduction

Using the procedures described in clause **4** to determine the mass losses in a corrosion test may not be reliable for cases where heavily corroded specimens are to be cleaned. A corroded surface, particularly of a multiphase alloy, is often more susceptible than a new, machined or polished, surface to corrosion by the cleaning procedure; thus the blank value determined in accordance with **4.4** may not be valid.

In such cases the procedure described in **5.2**, **5.3** and **5.4** should be followed.

5.2 Cleaning

The cleaning cycle is as described in clause **4** and comprises chemical treatment, washing, drying and weighing. For each specimen, repeat the cleaning cycle, in identical fashion, several times and determine the mass loss after each cycle to the fifth significant figure.

NOTE Repetition of the cleaning treatment may be necessary initially in order to remove completely the corrosion products. This removal can often be confirmed by examination with a low power (× 7 to × 30 magnification) microscope. This is particularly useful with pitted surfaces when corrosion products may accumulate in pits.

5.3 Determination of mass loss corresponding to removal of corrosion products

Plot the total mass loss after each cleaning cycle as a function of the number of cleaning cycles (see Figure 3). Two lines will be obtained, AB and BC, the latter of which corresponds to corrosion of the metal by the cleaning treatment after removal of the corrosion products. The true mass loss in the corrosion test, corresponding to removal of the corrosion products, is obtained by extrapolating the line BC to the mass loss axis, at point D.

NOTE If the cleaning treatment does not corrode the metal, the line BC will be horizontal and point B will represent the true mass loss.

[5)] For the purposes of this British Standard, chemical treatment includes electrolytic treatment and chemical immersion treatment.

5.4 Choice of cleaning method

To minimize the problem of corrosion of the metal by the cleaning treatment, the cleaning method should be chosen so that the slope of the line BC is as low (near to horizontal) as possible.

6 Preparation of standard water used for test solutions in the glassware, recirculating rig and static engine tests

6.1 Reagents

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

6.2 Solution preparation

Weigh, to the nearest 0.001 g, 0.444 g of anhydrous sodium sulphate, 0.495 g of dry sodium chloride and 0.414 g of dry sodium hydrogen carbonate. Dissolve the salts in water and dilute to 3 000 mL.

7 Determination of volume of suspended solids

7.1 Introduction

This clause describes a method for the determination of the volume content of suspended solids in solutions of the product, particularly those obtained from engine cooling systems during field trials.

7.2 Principle

A 100 mL test portion is centrifuged at a specified relative centrifugal force until the volume of sediment is constant.

7.3 Apparatus

7.3.1 *Centrifuge*, capable of centrifuging two or more filled tubes at a speed which can be controlled to give a relative centrifugal force of between 500 and 800 at the tips of the tubes.

NOTE The required speed of rotation may be calculated using the following equation:

$$N = 1\,336\sqrt{f/d}$$

where

N is the speed of rotation (in r/min);

f is the centrifugal force relative to the standard value of gravity;

D is the diameter of .swing measured during the tips of opposite tubes when in the rotating position (in mm);

For example, if D = 300 mm then a speed of 1 725 r/min is required for f to be 500 and a speed of 2 182 r/min is required for f to be 800.

7.3.2 *Centrifuge tube*, with a conical bottom, made of annealed glass, and graduated as given in Table 1, calibration being made with air-free water at 20 °C, reading the bottom of a shaded meniscus.

Table 1 — Graduation of centrifuge tube

Range	Subdivision	Volume tolerance
mL	mL	mL
0 to 0.1	0.05	± 0.02
above 0.1 to 0.3	0.05	± 0.03
above 0.3 to 0.5	0.05	± 0.05
above 0.5 to 1.0	0.10	± 0.05
above 1.0 to 2.0	0.10	± 0.10
above 2.0 to 100	—	± 0.10

7.4 Procedure

Fill the centrifuge tube to the 100 mL mark with the solution of the product, well shaken to ensure that the solid content is evenly distributed. Place the tube in a trunnion cup opposite another filled tube to establish a balanced condition and centrifuge for 10 min at a rate sufficient to produce a relative centrifugal force between 500 and 800 times the standard value of gravity[6)] at the tips of the whirling tubes. Read and record the volume of sediment to the nearest scale division. Repeat this operation until the volume of sediment remains constant.

Report the volume of sediment as a percentage of the volume of the test portion.

[6)] The standard value of gravity is 9.806 65 m/s^2.

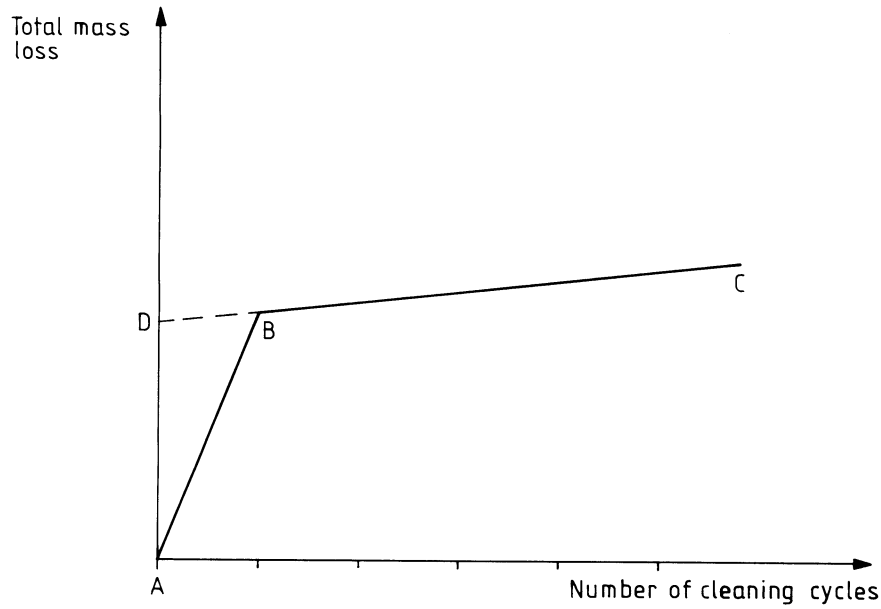


Figure 3 — Graph showing mass loss during repeated cleaning cycles

Publications referred to

BS 871, *Specification for abrasive papers and cloths.*
BS 1432, *Copper for electrical purposes. Strip with drawn or rolled edges.*
BS 1449, *Steel plate, sheet and strip.*
BS 1449-1, *Specification for carbon and carbon manganese plate, sheet and strip.*
BS 1452, *Specification for grey iron castings.*
BS 1490, *Aluminium and aluminium alloy ingots and castings.*
BS 2870, *Specification for rolled copper and copper alloys: sheet, strip and foil.*
BS 3195, *Methods for sampling petroleum products.*
BS 3195-1, *Liquid hydrocarbons: manual sampling.*
BS 3978, *Water for laboratory use.*
BS 5117, *Testing corrosion inhibiting, engine coolant concentrate ("antifreeze").*
BS 5117-0, *General introduction.*
BS 6017, *Specification for copper refinery shapes.*

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