BS 5117:

Part 0: 1992

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

Part 0. General introduction

Essais du liquide de refroidissement anti-rouille du moteur (antigel) Partie 0. Introduction générale Prüfung von korrosionshemmendem Kühlmittelkonzentrat für Motoren (Frostschutzmittel) Teil O. Allgemeine Einführung



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Foreword

This Part of BS 5117 has been prepared under the direction of the Chemicals Standards Policy Committee and is a new edition of BS 5117: Part 0: 1985, which is withdrawn. This edition introduces technical changes but it does not reflect a full review or revision of the standard, which will be undertaken in due course. The changes take account of the publication in 1992 of two new Sections of BS 5117; BS 5117: Section 1.5 describes a coolant hard water stability test, whilst BS 5117: Section 2.6 describes a test for corrosion of cast aluminium alloys under heat-transfer conditions.

The related British Standard BS 6580: 1985 has likewise been superseded by BS 6580: 1992, which specifies requirements for corrosion inhibiting, engine coolant concentrate ('antifreeze'), based on the tests described in Sections of BS 5117, including the new Sections 1.5 and 2.6.

The overall title of BS 5117 includes the term 'engine coolant concentrate' (see 4.1). Traditionally the term 'antifreeze' has been used to describe the concentrate which is added to the water in the cooling system of an internal combustion engine. However, the term 'antifreeze' takes into account only the frost-protective role of the product, so implying that its use is a seasonal requirement, and ignores its function as a heat exchange medium which is designed to protect the cooling system of the engine from corrosion and damage under all operating conditions. The term 'engine coolant concentrate' embraces all these requirements and is preferred. For the purposes of this standard, the engine coolant concentrate is generally referred to as 'the product'.

Annex A of this Part of BS 5117 gives guidance on the significance of test results obtained by the methods described in BS 5117: Part 1 and, where possible, indicates typical values which might be expected for an acceptable product. It is, however, intended solely as a guide.

The test methods described in BS 5117: Part 1 relate solely to simple physical and chemical properties and need no explanation. BS 5117: Part 2 describes laboratory tests (which attempt to simulate service conditions) and engine tests for the evaluation of corrosion inhibition performance. Such tests (particularly the recirculating rig test) are under continuous development. Annex B of this Part of BS 5117 has therefore been included to indicate the basis on which the test conditions were selected and to indicate how the standard conditions, specified in the recirculating rig test, could be varied so as to align the conditions with those associated with current developments in engine design.

At the time of publication of this British Standard no corresponding international standard exists.

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

General introduction

1 Scope

This Part of BS 5117 describes the structure of the other Parts and Sections of BS 5117 and defines terms used. Guidance on the significance of test results is given in annex A and comments on the choice of test conditions in corrosion inhibition testing are given in annex B.

2 Informative references

This Part of BS 5117 refers to other publications that provide information or guidance. Editions of these publications current at the time of issue of this standard are listed on the inside back cover, but reference should be made to the latest editions.

3 Structure of BS 5117

The structure of the various Parts and Sections of BS 5117 is given in table 1.

NOTE. Each Section of Parts 1 and 2 is published separately.

4 Definitions

For the purposes of this Part of BS 5117 the following definitions apply.

4.1 engine coolant concentrate

A liquid consisting of diols, such as ethanediol (ethylene glycol) and/or propane-1,2-diol (propylene glycol), together with corrosion inhibitors and possibly additives used for purposes other than freezing point depression.

4.2 coolant

The liquid which is caused to circulate through the cooling system of an internal combustion engine and which consists of an aqueous solution of an engine coolant concentrate.

4.3 test solution

An aqueous solution of an engine coolant concentrate, prepared at a specified concentration for test purposes.

4.4 test portion

A measured amount of an engine coolant concentrate, coolant or test solution used for a particular test.

Table 1. Structure of BS 5117		
Part and Section No.	Contents	
Part 0	General introduction including:	
	guidance on the significance of test results comments on choice of test conditions in corrosion inhibition testing	
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	determination of concentration of diluted product	
	determination of pH	
	determination of reserve alkalinity	
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Part 2	Methods of test for corrosion inhibition performance	
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	sampling of the product	
	preparation and assembly of metal test specimens	
	additional cleaning procedures for metal test specimens carrying adherent corrosion products	
	special procedure for cleaning heavily corroded metal test specimens and determination of mass loss	
	preparation of standard water used for test solutions in the glassware, recirculating rig and static engine tests	
	determination of volume of suspended solids	
Section 2.2	Glassware tests	
Section 2.3	Recirculating rig test	
Section 2.4	Static engine test	
Section 2.5	Field test	
Section 2.6	Test for corrosion of cast aluminium alloys under heat-transfer conditions	

Annexes

Annex A (informative) Guidance on the significance of test results

A.1 Introduction

Most of the test methods described in BS 5117 are those which are used by manufacturers of engine coolant concentrate and large scale users for the development, approval and quality control of the product.

For some of the results which will be obtained by using these test methods, there is general agreement on what is considered acceptable. In these cases, a precise figure for an acceptance value can usually be quoted. Other tests will produce results where the acceptability is covered by a range and much may depend upon the specific application, e.g. engine type and design, and on the specific preference of the user.

In this annex brief guidance is given on the use of the various tests described in BS 5117: Part 1 and where possible values are quoted.

A.2 Sampling of the product

Elementary guidance is given on a typical sampling procedure. For convenience, this information is given in each Section of BS 5117: Part 1. It is important to remember that diols are highly hygroscopic and are capable of absorbing large quantities of water from the atmosphere if left in open containers. Also it has been found that heavy metals may be taken into solution by the action of the product on parts of the container, e.g. lead, from soldered containers, has been found in solution. Such contamination can affect the inhibitors and, hence, the corrosion protection likely to be afforded by the product. Samples, particularly for corrosion testing, should therefore be taken whenever possible from containers which are representative of those in which the product is normally offered for sale. Ideally, the container and contents should have been in contact for at least three months, but obviously this is not always possible. Where there is any doubt, it may be necessary to repeat certain tests after periods of storage.

A.3 Determination of concentration of diluted product

(see clause 3 of BS 5117: Section 1.1: 1985)
The determination of concentration can provide a rapid field method for assessing the boiling point or freezing point of a coolant. This is achieved by correlating the concentration with the latter properties using standard tables, previously prepared, for the product in question.

A.4 Determination of pH

(see clause 4 of BS 5117: Section 1.1: 1985)
The pH value has no significance in assessing the quality of a product, but may be useful in ensuring quality consistency, by either the manufacturer or the bulk purchaser.

It is usual to measure the pH value of a 25 % (V/V) solution of the product in water. The method may be used for any other dilution, but the dilution used should always be quoted with the pH values obtained.

NOTE. The pH of undiluted product is not particularly meaningful.

A.5 Reserve alkalinity

(see clause 5 of BS 5117 : Section 1.1 : 1985)

In service use and during corrosion testing, it is more usual for acidity to develop in coolants than alkalinity. This is because the oxidation products of diols are acidic and because some products of combustion, which may find their way into the coolant, are also acidic.

Usually the amount of acidity developed is small and this can be counteracted by building into the inhibitor system a buffering agent which provides a reserve of alkalinity. The pH of the coolant is not therefore significantly affected and the coolant is prevented from becoming more acidic and possibly more corrosive.

A.6 Determination of boiling point

(see BS 5117: Section 1.2: 1985)

While the method may be used for any dilution of the product, its primary use is for determining whether lower boiling point materials are present in the undiluted product. Although most of these materials have satisfactory low temperature properties, they tend to evaporate from cooling systems at operating temperatures and are not considered to be satisfactory for normal long-term usage. They may also impose additional flammability and toxic hazards.

All diol-based products should have a boiling point, when evaluated by the method given in BS 5117: Section 1.2, of not lower than 150 °C.

A.7 Determination of freezing point

(see BS 5117: Section 1.3: 1985)

Diol-based products usually have freezing points similar to those of the pure diol, the presence of corrosion inhibitors generally having only a small effect. Similarly aqueous solutions of the products will have freezing points close to those of aqueous solutions, diluted to the same extent, of the pure diol.

Freezing point values indicate the level of frost protection which is likely to be afforded by the product when diluted with water in a stated proportion. Whilst most solutions pass through a range of temperatures when changing from liquid to solid, the temperature indicated from the test is that at which ice crystals first form in the solution. Although the determination of freezing point may be carried out on other concentrations of the product in water, it is usual, for specification purposes, to determine the value of a 50 % (V/V)

solution. At this concentration, an ethanediol-based solution should have a freezing point value not higher than -36 °C and a propanediol-based solution one not higher than -34 °C.

A.8 Determination of foaming characteristics (see BS 5117 : Section 1.4 : 1985)

Certain inhibitors and engine manufacturing contaminants may cause coolants to foam if they are shaken or violently agitated. When foaming occurs in the cooling system of an engine, loss of coolant may result. It is usual for the manufacturer to incorporate an antifoam additive in the product to prevent this from happening.

The test specified in BS 5117: Section 1.4 gives a method of assessing the tendency for foaming to occur. When a $33\frac{1}{3}$ % (V/V) solution of the product in water is tested, the volume of foam should not be greater than 50 ml and the break time should not be greater than 5 s. These values have been found to be reasonable, based upon practical experience.

A.9 Coolant hard water stability test

(see BS 5117 : Section 1.5 : 1992)

Use of hard water causes precipitation from the aqueous solution of coolant over a period of time. Synthetic hard water is prepared for the test, using calcium chloride. Under the test conditions, a volume of precipitate not greater than 0.5 cm³ is considered acceptable.

Annex B (informative)

Comments on the choice of test conditions in corrosion inhibition testing

B.1 Introduction

All the methods defined in BS 5117: Part 2 have been used in industry for assessing the performance of coolants but only one, the glassware test, has been generally available. For this reason general experience in the consistent behaviour of these methods exists only in this one case. Four of the methods defined in the standard are therefore novel forms of test in detail, although not in principle, and the parameters chosen for the glassware test also differ in some respects from those quoted previously elsewhere.

At the time when this standard was first published, little experience was available in the operation of any of the test methods. However, all the decisions taken then and with the 1985 revision were the subject of long and detailed discussion by the drafting committee and were revised frequently in the light of comments received from authorities not represented directly on the committee. It therefore seems still useful to detail the reasons which led the committee to select the types of test and the test parameters represented by the five standard

methods. It is hoped that these comments may increase the understanding of the background of the tests to users of the standard and also indicate some of the points of controversy which are likely to require attention if a further revision of the standard is necessary at some future date.

B.2 Glassware test

(see BS 5117 : Section 2.2 : 1985)

B.2.1 General

The test method is based on existing practices.

B.2.2 Test specimens

In order to maintain continuity with past experiences, the metals of the test specimens are kept as close as practicable to existing laboratory test practice. The specimens are similar to the test bundle specified in ASTM D 1384-80 [1].

The galvanic couples in the specimen assembly do not simulate adequately any couples present in a cooling system, but are chosen to accord with existing practice. The specimens are coupled together in two groups, one representative of the metals used in block and head construction, the other representative of those used in radiator construction.

B.2.3 Test solution

To maintain continuity with past experience, a standard test water rather than distilled water is used. For the same reason, the solution concentration is the sample volume diluted with twice its volume of water.

B.2.4 Aeration

The level of aeration used in this test conforms with past practice and would only be encountered in extraordinarily adverse conditions of service.

The tolerance given for the air flow rate is chosen

to conform with limits found to be achievable in practice.

B.2.5 Temperature

The temperature of 90 °C is considered to be most generally typical of the service temperature of coolants.

B.2.6 Duration of test

In general, a period of 14 days is found to be adequate for discrimination.

B.2.7 Cold immersion corrosion test

In order to meet the need to simulate the condition where vehicles are stored with little running time, e.g. reserve stores for the armed forces or emergency services, a cold immersion test is included. This test provides for a preliminary heating of the solution before exposure at ambient temperature, as it is considered that this is necessary to render some solutions fully effective. This may be regarded as simulating the initial parking run which all stored vehicles would presumably have.

B.3 Recirculating rig test

(see BS 5117: Section 2.3: 1985)

B.3.1 Apparatus

In order to achieve the maximum degree of standardization, the rig is constructed from special components, and the use of actual engine parts is avoided.

B.3.2 Metals

The metals used in the construction of the standard rig are representative of those employed in normal British engine design. In the standard test, aluminium alloy heater sheaths are used in combination with the cast iron reservoir. However, a number of alternative metals could be used. Heater sheaths could be made from cast iron, and the reservoir and spiral from aluminium alloy. A variety of combinations is possible and data collected using modifications to the standard rig may be useful when the standard again comes to be revised.

B.3.3 Exposed surface areas

It is desirable that the surface areas of the metals in the test rig are in a fixed ratio to each other, and to the volume of the test solution used. It is for this reason that the introduction of the solder spiral is necessary. In practice these ratios in actual engines vary considerably but for the purposes of the tests in this standard the ratios given in table B.1 have been adopted.

Table B.1. Ratios of metal surface areas		
Metal	Surface area per litre of test solution mm ²	Proportion of total metal area %
Ferrous metals	33 100	32.5
Aluminium alloys	12 900	12.6
Copper and copper alloys	56 000	54.9

B.3.4 Positioning of test specimens

Two sets of metal test assemblies are introduced into the rig, one in the reservoir and the other in the pipework. The object of this is to provide information on corrosion under varying conditions of coolant flow rate.

B.3.5 Flow rate of test solution

Because of the complexity of the waterways in an engine block and head and the wide variation in design from one engine to another, it is not possible to give an accurate typical figure for the coolant flow rate under service conditions. Similarly, even in the relatively simple design of the test rig, there is considerable flow rate variation, for example between the flow over the metal surface of the reservoir and the flow between the metal test specimens.

The rate of test solution turnover is considered to be the most practicable measurement to use in order to relate the flow rate in the rig to that encountered in practice.

In the rig operating at standard conditions, the flow rate of the test solution is equivalent to 3.6 liquid changes/min, this rate being of the same order as that encountered in vehicle cooling systems (one typical vehicle is known to give 9 coolant changes/min at 55 km/h (35 mile/h) and 13 coolant changes/min at 80 km/h (50 mile/h)).

B.3.6 Temperature

A temperature of 95 °C is chosen because it has been found that operation of this rig at this temperature can simulate the corrosion of hot aluminium alloy surfaces under service conditions. There is no doubt that temperatures of this order are met in service. Engine overheating has been known to arise as a result of radiator deposits of corrosion products; a cold finger is therefore inserted into the rig head tank to provide a relatively cool surface, so that such deposits can be readily detected (the formation of heavy deposits on the cold finger has been demonstrated with solutions of certain products known to produce them in vehicle radiators).

B.3.7 Level of aeration

To provide the test with some measure of acceleration over the best practical conditions, aeration, generally held to be the simplest and most easily controlled method of achieving this, is introduced. In addition, the deliberate introduction of a small amount of aeration has the advantage of swamping any accidential leaks of air into the rig due to faulty assembly.

Since it has been shown that certain coolant problems have arisen in de-aerated cooling systems, it may be useful to employ nitrogen instead of air in additional non-standard experiments to assess the performance of a given coolant under such conditions.

B.3.8 Pressure

It is common practice to use coolant systems pressurized to 70 kPa and this is the value chosen for the rig.

B.3.9 Solution concentration

When the test was first devised it was common practice to use a solution containing one part of product to three parts of water. Nearly all of the test data available from the use of these rigs has therefore been obtained using this concentration. Although current practice favours the use of a higher concentration, e.g. one part of product to two parts of water or 1:1, it was decided to retain the original strength so that direct comparisons with earlier results may still be made.

Non-standard tests at higher or lower

concentrations may, of course, still be run and may be advisable when fully evaluating the behaviour of a product.

A standard test water is used because it represents more closely than distilled water the conditions under which a coolant would be used in the field. Distilled water of grade 3 of BS 3978: 1987 is used, however, for topping up to replace liquid lost by evaporation and leakage. The use of distilled water for this purpose is preferable to the addition of further test solution, which may lead to spurious results because of replenishment of inhibitors that may otherwise reach a critically low concentration. Distilled water is used rather than the standard test water to avoid unnecessary interference with the ionic content of the test solution. Because severe leakage would invalidate corrosion results, the maximum permitted addition of distilled water is 50 ml. Topping up within this limit will not affect significantly the water to product ratio of the test solution.

B.3.10 Operating cycle

While continuous operation of the rig might be simpler, a series of regular shut-down periods is used to reproduce the effects of start-up and shut-down common to all vehicles in service. The cycle of shut-downs is a compromise between actual service conditions and the simplest test procedure. The typical cycle described in appendix C of BS 5117 : Section 2.3 : 1985 includes a series of short shut-downs and one long shut-down per week. The operation of the rig overnight and at weekends gives a maximum running time in a given total test period and permits servicing of the rig during the daytime. Provided the cycle is followed in all other respects there is no reason why a run should not start on any week day.

B.3.11 Duration of test

The operating period, 6 weeks, is considered to be a reasonable but arbitrary compromise between brief exposures in a grossly accelerated test and longer exposure under more realistic conditions. In the light of past experience this period can be regarded as being comparable to 1 year of moderately heavy use in a road-vehicle.

B.4 Static engine test

(See BS 5117 : Section 2.4 : 1985)

B.4.1 Engine preparation

Ideally a new engine should be used for each test, but this is not thought to be practicable. Therefore before each test the metal surfaces in the engine used are brought to a standardized condition by chemical cleaning.

B.4.2 Duration of test

This corresponds to a running distance of about 30 000 km (18 750 miles) which is considered to represent the average life of a coolant. The tolerance on the total test time allows for periods of engine stoppage for maintenance but eliminates the possibility that the engine might stand cold for protracted periods.

B.4.3 Test specimens

The assemblies are similar to those used in the glassware and recirculating rig tests. Further assemblies are not introduced into the system during the test as it is considered that the exposure of fresh metal surfaces would be unrealistic. Three assemblies are used to allow evaluation to be made after three time intervals. This is the minimum necessary to give some indication of change in corrosiveness with time. It is thought that the use of more assemblies would introduce complications in terms of the operation of the test.

B.4.4 Coolant samples

The total volume of coolant withdrawn for test during the run is limited so that any necessary replacement does not materially replenish the inhibitor content.

B.4.5 Conditions which affect test validity

An attempt has been made to strike a balance between the need to standardize conditions as closely as possible and the practical difficulties of the test. The conditions have been chosen on the basis of limited practical experience.

B.5 Field test

(see BS 5117 : Section 2.5 : 1985)

B.5.1 General

The recommendations given in BS 5117: Section 2.5: 1985 are based on the assumption that the engines to be tested are in road vehicles; conditions for engines used in other ways are so varied as to require individual treatment in each case.

B.5.2 Statistical conditions

These are based on the assumption that the test is of the simple pass or fail type. It may be argued that fewer vehicles need to be used if quantitative data from the test specimens are available. There are two main reasons for rejecting this view. First, it is thought that the use of specimens would introduce an element of simulation into an essentially service test, as the quantitative data from specimens might not represent certain types of damage to the waterways, e.g. the incidence of crevice attack or cavitation damage, even though these can cause the engine to fail. Secondly, even if this is not the case, the experimental scatter of the data derived from field tests is likely to be so large as to make the evaluation still essentially of the pass or fail type.

B.5.3 Relationship between the number of vehicles and the significance of the test

The figures given in table B.2 illustrate a simple method of calculating the significance of field tests involving various numbers of vehicles. It is assumed that the test is of the simple pass or fail type and is required to be reasonably certain of revealing an unacceptably high failure rate in the whole population of vehicles from which the test vehicles are selected. For the sake of simplicity, it

is also assumed that sampling is random and that the whole population is homogeneous, i.e. that failures are distributed evenly. As, in practice, these conditions cannot be achieved fully, the figures are optimistically low.

The required degree of certainty is a matter of individual choice but it is common practice in scientific experimentation to aim at 95 % or 99 %. Because of the inherent difficulties of conducting this test, experience has shown that a margin of at least 50 % is required to allow for vehicles that do not complete the test for extraneous reasons.

B.5.4 Engine cleaning

Ideally tests should be carried out on new engines, but this is not always possible in practice. If used engines have to be tested, only those which respond to the normal cleaning treatment should be employed to ensure that all the waterways have similar surfaces.

Table B.2. Significance of field tests			
Fraction of total population that will fail in service	Number of vehicles tested	Probability that there will be no failures in the vehicles tested	Percentage certainty of not missing failure
p	n	$(1-p)^n$	$\begin{bmatrix} 100[1-(1-p)^n] \\ \% \end{bmatrix}$
0.1	5	0.591	41.0
(i.e. 1 in 10)	10	0.349	65.0
10)	20	0.122	88.0
	30	0.042	96.0
	40	0.015	98.5
	50	0.005	99.5
0.01	10	0.905	10.0
(i.e. 1 in 100)	50	0.605	40.0
100)	100	0.366	63.0
	200	0.134	87.0
	300	0.049	95.0
	400	0.018	98.0
	500	0.007	99.0
0.001	10	0.990	1.0
(i.e. 1 in 1000)	100	0.906	9.0
1000)	500	0.610	39.0
	1000	0.372	63.0
	2000	0.138	86.0
	3000	0.051	95.0
	4000	0.019	98.0
	5000	0.007	99.0

B.5.5 Sampling

It is recommended that samples be taken from the top of the radiator, in order to obtain a sample representative of the circulating coolant.

B.5.6 Conditions which affect test validity

The permitted variation of coolant concentration is based on limited experience of the variability to be expected even from well-controlled tests.

B.6 Test for corrosion of cast aluminium alloys under heat-transfer conditions

(see BS 5117: Section 2.6: 1992)

NOTE. This test method is based upon that contained in ASTM D 4340-89 [2] Method for corrosion of cast aluminium alloys in engine coolants under heat-transfer conditions.

B.6.1 Heat-transfer corrosion cell temperature and pressure

The cell used in this test has been designed to simulate the conditions in a typical cylinder head arrangement. Likewise, the conditions of temperature and pressure have been chosen as being typical of engine operation at the time of issue of the standard.

B.6.2 Test solution

Standard test water, of grade 3 of BS 3978: 1987, is used with the addition of sodium chloride to prepare the test solution. A suitable concentration is achieved by diluting the coolant with three times its volume of this water.

B.6.3 Duration of test

The test is run continuously over a period of 168 h, which has been found to be a sufficient length of time in which to distinguish between coolant performances.

B.6.4 Cleaning of test specimens

It has been found that, with deterioration of a specimen surface, the rate of corrosion during cleaning often increases. To ensure uniformity and fairness in testing, it was considered necessary to stipulate the use of uncorroded specimens in determining a blank correction.

B.6.5 Precision

Repeatability and reproducibility figures obtained with this test are given in 10.2 of BS 5117: Section 2.6: 1992. Although it is not a requirement of the test that such data be obtained, they should, if they are obtained, be within the values stated.

List of references (see clause 2)

Informative references

BSI standards publications

BRITISH STANDARDS INSTITUTION, London

BS 3978: 1987	Specification for water for laboratory use
BS 5117:	Testing corrosion inhibiting, engine coolant concentrate
DC 5117 . David 1 .	('antifreeze')
BS 5117 : Part 1 :	Methods of test for determination of physical and chemical properties
BS 5117: Section 1.1: 1985	Ancillary procedures
BS 5117: Section 1.2: 1985	Determination of boiling point
BS 5117 : Section 1.3 : 1985	Determination of freezing point
BS 5117 : Section 1.4 : 1985	Determination of foaming characteristics
BS 5117: Section 1.5: 1992	Coolant hard water stability test
BS 5117 : Part 2 :	Methods of test for corrosion inhibition performance
BS 5117: Section 2.1: 1985	General procedures
BS 5117: Section 2.2: 1985	Glassware tests
BS 5117 : Section 2.3 : 1985	Recirculating rig test
BS 5117: Section 2.4: 1985	Static engine test
BS 5117: Section 2.5: 1985	Field test
BS 5117 : Section 2.6 : 1992	Test for corrosion of cast aluminium alloys under heat-transfer conditions
BS 6580 : 1992 ¹⁾	Specification for corrosion inhibiting, engine coolant concentrate ('antifreeze')

Other references

[1] ASTM D 1384-80 ²⁾	Method for corrosion test for engine coolants in glassware
[2] ASTM D 4340-89 ²⁾	Method for corrosion of cast aluminium alloys in engine coolants
	under heat-transfer conditions

Referred to in the foreword only.
 Copies may be obtained from the ASTM European Office, 27-29 Knoll Piece, Wilbury Way, Hitchin, Herts, SG4 0SX, England.

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