COMMISSION OF THE EUROPEAN COMMUNITIES
SAFETY AND HEALTH COMMISSION
FOR THE MINING AND EXTRACTIVE INDUSTRIES
Working Party
Rescue arrangements, fires and underground combustion
Committee of Experts
on
Fire-Resistant Fluids

SIXTH REPORT
on
SPECIFICATIONS AND TESTING CONDITIONS RELATING TO FIRE-RESISTANT HYDRAULIC FLUIDS used for POWER TRANSMISSION (HYDROSTATIC AND HYDROKINETIC) IN MINES

(This report supersedes the "FIFTH REPORT" dated 15th November 1974)

Doc. 2786/8/81 E LUXEMBOURG 1983 Edition

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CONTENTS

Page

PART I - PREFACES 1

Preface to the Sixth Report 2
Preface to the Fifth Report 4
Preface to the Fourth Report 5
Preface to the Third Report 7
Preface to the Second Report 9
Introduction to the Second Report 10

PART II - GENERAL PROVISIONS 12

2.1 Approval/acceptance procedures and testing centres 13
2.1.1 Approval/acceptance procedures 13
2.1.2 Submission of fluid for test 13
2.1.3 Approval/acceptance certificates 14
2.1.4 Publication of results 14
2.1.5 Testing centres 14
2.2 Classification and marking 16
2.2.1 Identification 16
2.2.2 Classification 16
2.2.3 Marking of containers 21
2.3 Storage and use 22
2.4 Testing methods 22
2.4.1 Prescribed tests 23
2.4.2 Discretionary technological tests 24
2.4.3 Summary of test requirements 24
2.5 Behaviour of fluids in service 26
2.6 Quality control tests 26

PART III - TESTS FOR RESISTANCE TO FLAME 27

3.1 General 28
3.2 Spray ignition tests 28
3.2.1 The "Community of six" spray test 28
3.2.2 The United Kingdom spray test 34
3.2.3 The flame length/flow rate flame test 36
3.3 Flame propagation tests 48
3.3.1 Method of test for flame propagation in a fluid/coal dust mixture 48

3.3.2 Method of test for flame propagation along a fluid soaked wick 54

PART IV - HEALTH CRITERIA 58

4.1 Testing Conditions and Criteria for Assessing the Hazard to Health 59

4.1.1 General 59

4.1.2 Assessment of the hazard to health 59

4.2 Methods of Test for the Toxicity of types HFA, HFB and HFC Fluids 59

4.2.1 General 59

4.2.2 Verification of identity 60

4.2.3 Oral toxicity 60

4.2.4 Irritant effect 60

4.2.5 Aerosol toxicity 62

4.2.6 Thermal decomposition products 66

4.2.7 Acceptance criteria 67

4.3 Methods of Test for the Toxicity of type HFD Fluids 72

4.3.1 Chemical composition 72

4.3.2 Verification of identity 72

4.3.3 Oral toxicity 72

4.3.4 Irritant effect 72

4.3.5 Aerosol toxicity 73

4.3.6 Thermal decomposition products 75

4.3.7 Acceptance criteria 76

4.4 Method of Test for Neurotoxic Effects of Phosphoric Esters 78

PART V - PRESCRIBED TECHNOLOGICAL TESTS 79

5.1 Methods of Test for Kinematic Viscosity 80

5.1.1 Method of Test for Kinematic viscosity using capillary type viscometers 80

5.1.2 Method of Test for Kinematic viscosity using drop-ball viscometers 87

5.2 Methods of Test for the Stability of Emulsions 91

5.2.1 Method of Test for Emulsion Stability of type HFA Fluids (excluding type HFAS Fluids) 92

5.2.2 Method of Test for Emulsion Stability of type HFB Fluids at ambient temperature 95

5.2.3 Method of Test for Emulsion Stability of type HFB Fluids at medium temperature 99

5.2.4 Method of Test for Emulsion Stability of type HFB...LT Fluids at low temperature 102
5.3 Ageing and thermal stability 105
5.3.1 Method of Test for ageing properties of type HFC Fluids 106
5.3.2 Method of Test for ageing properties of type HFD Fluids 112
5.4 Method of Test for pH value of types HFA and HFC Fluids 118
5.5 Methods of Test for water content of types HFB and HFC Fluids 121
5.5.1 Method of Test for water content of type HFB Fluids 121
5.5.2 Method of Test for water content of type HFC Fluids 125
5.6 Method of Test for air separation or deaeration capacity 129
5.7 Method of Test for foaming characteristics 133
5.8 Method of Test for shear strength of fluids 136
5.9 Method of Test for corrosion inhibiting properties of fluids 140
5.9.1 Method of Test for corrosion inhibiting properties (all fluids) 141
5.9.2 Method of Test for corrosion inhibiting properties (all fluids) 145
5.9.3 Method of Test for corrosion inhibiting properties of oils used for the preparation of HFA Fluids 148
5.9.4 Method of Test for corrosion inhibiting properties (all fluids) 156
5.10 Methods of Test for compatibility of sealing materials and fluids of types HFA, HFB, HFC and HFD 160
5.10.1 Method of Test for compatibility of sealing materials and fluids of types HFA, HFB, HFC and HFD 161
5.10.2 Method of Test for compatibility of fluids with standard test elastomers 164

PART VI - DISCRETIONARY TECHNOLOGICAL TESTS 172
6.1 Determination of fluid density 174
6.2 Method of Test for the neutralisation number of types HFB and HFD Fluids 175
6.3 Method of Test for viscosity index of type HFD Fluids 184
6.4 Method of Test for pour point 185
6.5 Method of Test for oil content of type HFA Fluids 190
6.6 Method of Test for the filterability of fluids 193
6.7 Methods of Test for the thermal stability of type HFD Fluids 196
6.7.1 Method of Test for thermal stability of type HFD Fluids 197

6.7.2 Method of Test for thermal stability of type HFD Fluids in traction couplings 201

6.7.3 Method of Test for thermal stability of type HFD Fluids in scoop controlled couplings 204

6.8 Methods of Test for the compatibility of coatings for power pack tanks with fluids of types HFA, HFB and HFC 207

6.8.1 Method of Test for compatibility of coatings of power pack tanks with type HFA Fluids and emulsifying oils 208

6.8.2 Method of Test for compatibility of coatings of power pack tanks with types HFB and HFC Fluids 211

6.9 Methods of Test for the lubricating properties of Fluids 216

6.9.1 Method of Test for lubricating properties of fluids by measurement of their effect on rolling fatigue resistance of bearing steel using the rolling 4-ball machine 218

6.9.2 Method of Test for lubricating properties of fluids in sliding contact conditions 223

6.9.3 Method of Test for anti-wear properties using the vickers pump test rig 229

6.10 The closed flash point test 235

Appendix I - List of properties considered by the Committee of Experts on fire-resistant fluids during the preparation of the Sixth Report on criteria and specifications

Units of measurement

In compiling this report, the Committee of Experts sought to base many of its test requirements on established laboratory procedures and on current specifications of national or international organisations whenever these were considered satisfactory for the purpose. As many of these specifications originated at different times and in different countries, the units of measurement and the abbreviations used also differ. To preserve comparability with established methods and to avoid difficulties which may arise from the simple conversion of values, no attempt has been made to adopt a single system of units of measurement (SI system for example). The units used in the original test description have been retained subject to the metrication of Imperial units and the adoption of a uniform system of abbreviations. The principal units used in this report are given in the following table with the SI unit shown underlined :

Table. Units of measurement with multiples and sub-multiples

Quantity	Unit	Symbol
length	metre	m
	centimetre	cm
	millimetre	mm
volume	litre	l
	millilitre	ml (1 ml is also equal to 1 cm^3)
mass	gramme	g (10^{-3} kilogramme)
time	second	s
	minute	min
	hour	h
	day	d
pressure	bar	bar (1 bar is equal to 10^5 Newton per square metre)
kinematic viscosity	centistoke	cSt (numerically equal to the SI unit of mm^2/s)
angular velocity	radians per second	rad/s
	revolutions per minute	rev/min
linear velocity	metres per second	m/s

Note Pressure is also expressed as a head of water or mercury when measurement is by means of a manometer.

PART I

PREFACES

PREFACE TO THE SIXTH REPORT

When, in November, 1974, the Safety and Health Commission for the Mining and Extractive Industries accepted the Fifth Report on the "specification and testing conditions relating to fire-resistant fluids used for power transmission", it recommended that progress be made towards harmonising those specifications and testing conditions and it asked the Experts in the domain to continue their work with a view to reducing the number of standards applied.

This work started at the end of 1977, with a detailed consideration of all the test procedures listed in the Fifth Report, together with others which might have been relevant (see Appendix 1). In this work, a series of basic documents of the technological properties of fluids, prepared by the United Kingdom National Coal Board, were of great value. At the same time, the Health and Safety Executive Explosion and Flame Laboratory at Buxton were engaged in developing a test procedure for comparing some of the fire properties of all types of fluids when emitted from a nozzle under pressure. The methods of assessing the hazards to health posed by the use of fire-resistant fluids in mines were also being reviewed. In this report there is no discussion of any indirect environmental risks which could perhaps be associated with the use of certain fire resistant fluids. This question is currently being considered.

The opportunity has been taken to assemble this work in one volume and publish a Sixth Report which largely responds to the wishes of the Safety and Health Commission, as set out above, indicating, as far as is possible at the moment, harmonised testing procedures. At the same time, this report recognises and takes into account the very considerable experience gained in both the use and the testing of fire-resistant fluids in the mining industries over the last 20 years.

This report is divided into six parts. Part I contains the prefaces to this and previous reports and Part II deals with general provisions including approval procedures and the systems to be adopted for the classification and identification of fluids. Descriptions of the various test methods are to be found in Parts III to VI.

Parts III, IV and V contain, respectively, descriptions of test methods relating to resistance to flame, health criteria and selected technological properties which should form the basis for the acceptance or approval of a fluid for use below ground in mines. The final Part VI of the report contains descriptions of the methods relating to those other technological properties of a fluid which, having regard to the application and conditions of use, may also be essential to a proper assessment of the fluid. For the purpose of this report the technological tests described in Part V are designated as 'prescribed tests' while those in Part VI of the report are termed 'discretionary tests'.

With regard to be criteria to be applied by the testing authorities, these still vary, to some extent, between Member States, but there is a large measure of agreement. It this envisaged that the flammability criteria, in particular, will be established by the authorities, taking into account both the conditions of use and the situations in which the fluid is likely to be used. In the meantime, work should continue to further harmonise the flammability and the technological criteria.

This Sixth Report has been produced by Experts drawn from the coal mining industries of the European Community, who have studied carefully the conditions relating to the application of hydraulic fluids in those industries. It is, nevertheless, for the Authorities of the Member States of the Community to decide in which circumstances and under what conditions the various types of fire resistant fluids will be used.

The Experts of the Community are aware that the previous Reports have been used as a standard for fire-resistant fluids in many other industries, including some outside the Community, and they hope that the present report may be similarly used.

PREFACE TO THE FIFTH REPORT

Since the publication of the Fourth Report on the Specifications and Testing conditions relating to Fire-Resistant Fluids used for power transmission in 1971, the United Kingdom, Denmark and Ireland have joined the Community, whilst at the same time, there has been an increase in the use of fire-resistant fluids in mines in member countries of the enlarged Community.

The Mines Safety and Health Commission is aware of these developments and has decided to publish a Fifth Report, updating the specifications and testing conditions applying to fire-resistant fluids meeting the original 'Community of Six' requirements as given in the Fourth Report, and adding further sections to incorporate the specifications and test procedures used in the United Kingdom.

In the enlarged Community a whole series of fire-resistant fluids and equipment designed for their use, have been developed, and although these developments have been along widely different lines, the results have generally been satisfactory. At this time, different fluids are being used successfully in mining industries throughout the Community, and in order to allow a wide choice of fire-resistant fluids, the Mines Safety and Health Commission has decided to publish the Fifth Report in its present form. It is for the mining authorities in the individual states to specify which parts shall be applied in mines coming under their jurisdiction.

Notwithstanding this, the Commission recommends that continuous progress be made towards harmonising specifications and testing methods, and it instructs the Experts to continue their work with a view to reducing the number of standards, adopting only those which are acceptable to all.

This report has been arranged in eight parts ; Parts I, II, III and IV concern the Types of fluid considered in the Fourth Report, which are those extensively used in the original Community of Six, and meeting their test requirements.

In the United Kingdom, the development of fire-resistant fluids and the machinary in which they are used have been along different lines, and accordingly different tests have evolved. Parts V, VI and VII deal with those fluids used mainly in the United Kingdom and complying with the test requirements of the British mining authorities.

Part VIII contains a list of Experts and once again on behalf of the Mines Safety and Health Commission I wish to thank them and all other persons who have contributed to the production of this Fifth Report.

Luxembourg, 15 November 1974

P.J. HILLERY

Chairman of the Mines Safety
and Health Commission

PREFACE TO THE FOURTH REPORT

In adopting in 1967 the Third report on specifications and testing conditions relating to fire-resistant fluids used for power transmission, the Mines Commission expressed its satisfaction at seeing this report published for the third time by the group of experts.

It was nonetheless mindful of the difficulties involved in handling fire-resistant fluids due to their effects on the working life of hydraulic components. This applies in particular to equipment already in service designed for using mineral oils. In this respect the preface to the last report already mentioned the importance of anti-corrosion and anti-wear properties.

The Safety Commission declared itself in principle in favour of carrying out research and development in this area. It recommended the Commission of the European Communities to award grants for research to determine to what extent machinery in service could, if necessary, be adapted to the use of fire-resistant fluids recommended for safety reasons for use in the mines. It also expressed the hope and desire that the industries concerned would continue their work towards finding a solution to this problem.

Having regard to the situation, it instructed the group of experts to follow developments in this area and examine to what extent test criteria and methods for assessing the characteristics of such products might be relaxed to enable their use underground.

The experts endeavoured to carry out the task assigned to them by the Safety Commission. They first took account of the need for using hydraulic transmission fluids in mines while at the same time looking for safety improvements. In addition, praiseworthy attempts are being made by fluid manufacturers to develop products meeting the criteria of the Third report.

In this connection the experts not only benefited from the findings of research institutes in the Community countries, but also conducted a great many comparative trials, allowing more than had been done in the past for practical conditions underground in both determining test conditions and interpreting results.

In this way the experts revised inter alia the specifications and test conditions for determining flame propagation in mixtures of coal dust and liquids and fixed new limiting values.

The test method for determining wear protection by means of the 4-ball apparatus has been supplemented by the Vickers-type rotary pump method ; but it must be remembered that the determination of the limit values for each of these methods has not yet been completed, so that they are to be regarded until further notice as experimental.

Mention should also be made of theoretical changes in the procedures for determining mining health criteria.

There is no need to go into details on many other modifications here.

I would like once again to thank all who have contributed to improving this report, and assure them of my gratitude ; in particular the experts and research institutes who took a direct part in compiling this report, the mines which are making increased and more frequent use of the fire-resistant fluids approved by the mining authorities for use underground, and the manufacturers of fluids and machinery who by unremitting research and development efforts, working in close liaison with the aforementioned experts and bodies have promoted progress in this area that is hailed with approval by all who are interested in improved safety in the mines.

In adopting this report at its session of 26 March 1971, the Mines Safety Commission indicated that it would have to be reviewed in the light of further progress in this constantly evolving field.

Luxembourg, 26 March 1971

A. COPPE

Chairman of the Mines Safety Commission
Member of the Commission of the European Communities

PREFACE TO THE THIRD REPORT

Having been finally adopted by the Mines Safety Commission on 16 October 1964, the second report on specifications and testing conditions relating to fire-resistant fluids used for power transmission was issued to the mining authorities and collieries, and also to the relevant sectors of the oil, engineering and chemical industries in Community and third countries.

The Safety Commission instructed the working party to devote itself in particular to the following points :

1. Adjustment of the criteria to technical progress

After a period of practical application of the specifications set forth in the Second Report, some of the specifications and test conditions must be reviewed to allow for technical progress.

2. Comparison of test results

If, even though the testing conditions have been meticulously adhered to, there are still differences of opinion as to the merits of a particular fluid - if for instance it is authorised in one country and banned in another - the technical bodies must examine the reasons for these differences.

The extent to which the Second Report's proposals have been followed has in fact varied a good ideal, in the case not only of the member countries' mining authorities but also of the manufacturers of hydraulic fluids and machinery. So far, official regulations making the employment of the Report's specifications and conditions obligatorv have been promulgated in only a few countries ; in others the authorities have provisionally recommended collieries to use only such fire-resistant fluids as are on line with the Report's specifications.

Now, nearly three years after the Report appeared, it is clear that the requirements laid down in it have been of considerable value in influencing developments in fire-resistant fluids and hydraulic plant.

For instance, there are a number of Group A, C and D, fire-resistant fluids which correspond to the Second Report's specifications, and several manufacturers have made changes in the design of their machines to suit the technological characteristics of such fluids.

Moreover, it is becoming evident that the collieries' experience is being turned to account in other industries, with the necessary adaptations for their own particular purposes.

In the meantime, the conclusions drawn from the practical implementation of the Report have enabled a number of improvements to be made, which are taken into account in the present edition.

Fuller data have also recently been secured concerning the importance of anti-corrosion and anti-wear properties, but the findings are not yet sufficiently detailed and conclusive to serve as a basis for recasting the testing conditions. Particulars of progress to date in this connexion can be obtained from the specialised establishments listed in the Report.

I hope very much that the new Third Report will play its due part in raising safety standards in the mines, and I wish to extend sincere thanks to all those who participated in the work and in the preparation of this Report.

Luxembourg, 10 October 1967

A. COPPE

Chairman of the Mines Safety Commission
Member of the Commission of the European Communities

PREFACE TO THE SECOND REPORT

The present Report on specifications and testing conditions relating to fire-resistant fluids used for power transmission does not represent only six years' work by a group of specialists (engineers and medical experts) who are daily occupied with these problems in the coal-mining industry and in its research stations ; it is also the fruit of the continuous collaboration between numerous experts from the oil and chemical industries (the manufacturers of these fluids), from the coal-mining industry (the users) and the engineering industries (which make the machines for which these fluids are intended).

The objective was an ambitious one : namely to ensure that the underground use for fire-resistant fluids for power transmission should be subject, in all Community countries, to the production of a certificate witnessing that they had undergone an identical set of tests.

It was consequently not enough simply to lay down flammability criteria. Other criteria had to be laid down, as a guarantee that the liquids possess the technical characteristics called for by their intended application, and finally, other criteria again ensuring that these two requirements are met without attendant risk to the health of the workers.

In addition, it was necessary to stipulate the methods to be used for determining that each fluid proposed for underground use meets their requirements.

It is this which explains the meticulous detail in which the report has been prepared.

Adopting this report at its Session of 16 October 1964, the Mines Safety Commission expressed the wish that it would be distributed as widely as possible, being certain that it could contribute greatly to the furtherance of safety.

On behalf of the Mines Safety Commission, I thank all the experts who participated in the preparation of this report.

Luxembourg, 16 October 1964

Paul FINET

Member of the High Authority
Chairman of the Mines Safety Commission

INTRODUCTION TO THE SECOND REPORT

The Conference on Safety in Coal Mines summoned on 6 September 1956 by the Council of Ministers at the request of the High Authority of the ECSC, immediately after the Marcinelle disaster- adopted the following recommendation in its final report, Chapter II. "Technical Research", in Recommendation 36 - M (page 136 of the French version of the Conference Report) :

"Research should be continued with the object of developing incombustible fluids to be used in place of inflammable oils for mechanical purposes, e.g. in hydraulic equipment, couplings, balances, props, etc."

The Mines Safety Commission which was set up on the basis of this Conference and at the suggestion of the High Authority, by decision of the Council of Ministers on 9 July 1957, with a view to reducing the dangers attendant on the use of inflammable fluids in the coal industry, instructed its Working Party on Mine Fires to work out criteria for fire-resistant fluids and test methods.

On 23 November 1958, the Working Party decided in the light of the requirements of safety in mines to entrust the study of this problem to a group of experts.

On 20 December 1960, the Working Party was able to submit to the Mines Safety Commission an information report (published on 28 November 1960) regarding the first conclusions which the group of experts had reached since the beginning of its work.

Having examined this report, the Mines Safety Commission agreed to make it available to all interested bodies for their information, in order to keep them in touch with developments in this field and with the work of the group of experts.

In the circular of 24 February 1961 (Doc. 1159/1/61) this information report on the establishment of criteria for fire-resistant fluids used for power transmission and for tests to be carried out was made available to the coal mining industry, the oil and chemical industries and the mechanical engineering industry in the Community countries. In addition, the text of the report was included in the 2nd Report and the Mines Safety Commission published in June 1961.

Since then the group of experts has re-examined in depth the problems of stipulating criteria of inflammability and technological criteria. Comparative tests, carried out in the laboratories of the Technischer Ueberwachungsverein (Essen), the Versuchgruppengesellschaft (Dortmund), the Institut National des Mines (Pâturages) and Houillères du Nord et du Pas-de-Calais (Sin-le-Noble), have made it possible to verify the proposed criteria and to test new experimental apparatus and methods. The group of experts has carefully examined the proposals and suggestions submitted by representatives of the industries consulted. In particular, it has had discussions regarding the solution of various questions with representatives of the oil industry, the chemical industry and the mechanical engineering industry, and also the coal-mining industry. It has also sought to take full account of the most recent American information in this field.

Examination of the health criteria received particular attention. The group of experts was able to call upon medical experts from the Federal Republic of Germany, France and Belgium.

The group of experts is of the opinion that this 2nd Report, which contains the most recent information relating to the establishment of criteria for fire-resistant fluids used for power transmission, provides in its present form information which will be useful not only to the coal-mining industry but to the above-mentioned industries as well.

It hopes thereby to have contributed to the enhancement of safety.

However, it does not consider that its task is complete. At a suitable time this 2nd Report must be re-examined and updated in the light of the latest technical developments. In this connexion, it hopes to receive, as in the past, suggestions and proposals of practical value.

The group of experts wishes to thank all those who have assisted in drawing up its report.

PART II

GENERAL PROVISIONS

2 GENERAL PROVISIONS

2.1 APPROVAL/ACCEPTANCE PROCEDURES AND TESTING CENTRES

2.1.1 Approval/acceptance procedures

Fire-resistant hydraulic fluids for use in mines should be approved or accepted only on evidence that:

a) they have been tested in accordance with the prescriptions contained in Part III to verify that they have an adequate resistance to flame;

b) they have been assessed in accordance with the requirements of Part IV to ensure that they are unlikely to cause a hazard to health under the conditions in which they may be used; and

c) they have been subjected to selected technological tests, see Note 1 below, and assessed to ensure that they possess adequate physical and chemical properties, see Note 2.

Note 1. In producing this report, the Experts considered the various tests available and selected those most appropriate to the measurement of the physical and chemical properties of a fire-resistant fluid. In view of the importance of certain of these properties to performance and safety, some tests were considered an essential prerequisite to acceptance of a fluid. Such tests are designated as 'prescribed technological tests' and are contained in Part V. Other tests not designated as prescribed tests but which nevertheless, may be required by a purchaser or accepting/approving authority to verify the suitability of a fluid for a particular application have been designated 'discretionary technological tests' and have been included in Part VI.

Note 2. Normally, new fluids should be subjected to each of the prescribed technological tests described in Part V and to those of the discretionary tests in Part VI considered appropriate to the proposed application. However, new fluids of a type and formulation very similar to an existing subjected to a particular test provided the approving or accepting authority is satisfied that the available test data for the existing fluid is equally applicable to the new fluid. In such cases proof of the identify of the fluid should be submitted to the above authority.

2.1.2 Submission of fluid for test

A fluid presented for approval or acceptance should be identified by a precise and original name which differs from all previously used names for fluids of the same type.

The quantity of fluid submitted for test should be taken from a single manufactured batch of fluid and should be sufficient to provide test samples for all the tests which may be required to be carried out, including those for resistance to flame, hazards to health and technological properties.

Where the series of tests involves more than one laboratory, the approving or accepting authority shall be resonsible for allocating to the submitted fluid a single identification number and for arrangements whereby the necessary quantities are distributed to the various laboratories.

2.1.3 Approval/acceptance certificated

Following the completion with satisfactory results of the series of tests comprising those specified in Parts III, IV and V of this report and the appropriate discretionary technological tests from Part VI, the approving or accepting authority (or testing centre) may issue a certificate of approval or acceptance.

The nature of the certificate or notification of approval or acceptance, its period of validity, the terms on which it may be renewed or extended, etc, are for the authority to determine but, in any case, the approval/acceptance shall be revoked if service experience or the results of quality control tests (see section 2.) show this to be necessary.

2.1.4 Publication of results

The laboratories listed in section 2.1.5 should, at regular intervals, exchange information on the testing and service experience of fluids including details of approvals/acceptances granted and withdrawn (unless this contradicts the procedures in the individual countries, and also subject to the agreement of the manufacturer).

2.1.5 Testing centres

The laboratory tests on fire resistant fluids should be carried out at one or more of the following testing centres :

Federal Republic of Germany

- Rheinisch-Westfälischer Technischer Ueberwachungsverein, Essen/Ruhr (Leitendes Fachinstitut).
- Versuchgrubengesellschaft mbH, Dortmund.
- Pharmakologisches Institut der Universität Hamburg, Hamburg.
- Hygiene-Institut des Ruhrgebiets, Gelsenkirchen.

Belgium

- Institut National des Industries Extractives, Pâturages/Hainaut.

France

- Laboratoire du Centre d'Etudes et Recherches des Charbonnages de France, Verneuil en Halatte.

Luxembourg

Italy

Netherlands

United Kingdom

- National Coal Board, East Midlands Regional Scientific Department, Mansfield Woodhouse.

- Health and Safety Executive Explosion and Flame Laboratories, Buxton (Flame tests only).
- Mining Research and Development Establishment, National Coal Board, Ashby Road, Stanhope Bretby, Burton-on-Trent, Staffordshire, DE 15 0QD.
- Chief Medical Officer, National Coal Board, Hobart House, Grosvenor Place, London, SW1X 7AE.
- Other Nationally accredited laboratories.

2.2 CLASSIFICATION AND MARKING

2.2.1 Identification

Fire-resistant hydraulic fluids for use in hydrostatic and hydrokinetic power transmissions in mines are to be identified by the letters:

HF

followed by further symbols to indicate their general fluid type, their viscosity and other characteristics in accordance with the system of classification described in section 2.2.2.

2.2.2 Classification

2.2.2.1 Classification by type

Fire-resistant hydraulic fluids are to be classified and designated on the basis of their general type as follows:

HFA	Emulsions of the oil-in-water type containing low concentrations of emulsifying oil (not more than 20%). Fluids currently in common use contain between 1 and 5% by volume of emulsifying oil.
HFAS	Solutions of fluid concentrates in water which are designed for use in applications similar to those for which HFA fluids are used. The proportion of the fluid concentrate is usually not more than 10% by volume.
HFB	Emulsions of the water-in-oil type containing, typically, about 40% of water by volume.
HFC	Solutions of polymers in water, normally containing more than 35% of water by volume. The polymers are usually polyglycols.
HFD	Fluids that do not contain water.

2.2.2.2 Classification by viscosity

With the exception of certain existing fluids which should be dealt with as described below, the classification of hydraulic fluids by viscosity should be based on their kinematic viscosity at 40°C in accordance with the system specified in the International Standard ISO 3448 (see note 1).

As the kinematic viscosity of types HFA and HFAS fluids having low concentrations of emulsifying oil and fluid concentrate, respectively, is very close to that of water and is not normally measured, these made-up fluids should be given the viscosity designation 1. (This is not an ISO viscosity grade number, see note 1).

For existing fluids, some of which were developed before the ISO system of viscosity grading was devised and whose measured viscosities fall outside the viscosity bands of the ISO system, the requirements of ISO 3448 should be modified to provide a continuous grading system. For such fluids, the viscosity classification is to be based on the manufacturer's stated value of the kinematic viscosity measured at 40°C and the fluid shall be designated by the ISO grade number which is nearest to the value of viscosity declared by the manufacturer.

The viscosity grade designations of the various types of fluids currently in use should fall, normally, within the ranges given in table 1, or such other grades as prescribed in ISO standard 3448.

Table 1. Viscosity grade designations for various fluids

Fluid type	Viscosity grade designations
HFA	1, 10, 15, 22
HFAS	1
HFB	32, 46, 68, 100
HFC) HFD)	15, 22, 32, 46, 68, 100

Note 1

The ISO system of viscosity grading defines 18 kinematic viscosity bands, the mid points of which form a logarithmic series in the range 2.2 to 1500 cSt measured at 40°C. The width of each viscosity band represents a tolerance of ± 10% of the mid point value. As a consequence the ISO system of grading is discontinuous with gaps between the viscosity bands. It was devised, primarily, to rationalise future production and use of oils and hydraulic fluids. The ISO viscosity bands relevant to the types of fluids dealt with in this report are given in table 2.

Table 2. ISO Viscosity classifications

ISO viscosity grade No.	Mid-point kinematic viscosity cSt at 40°C	Kinematic viscosity limits cSt at 40°C	
		Min.	Max.
10	10	9.0	11.0
15	15	13.5	16.5
22	22	19.8	24.2
32	32	28.8	35.2
46	46	41.4	50.6
68	68	61.2	74.8
100	100	90.0	110.0

2.2.2.3. Classification by constituents

In the case of type HFD fluids, the nature of the constituents should be indicated using the following designation:

HFD R **	fluid based on phosphate esters
HFD S **	fluid based on chlorinated hydrocarbons
HFD T **	fluid based on mixtures of phosphate esters and chlorinated hydrocarbons
HFD U **	fluid based on other compounds

Note: In practice the symbol ** will be replaced by the viscosity classification number, see section 2.2.2.2.

2.2.2.4. Suitability for use at low temperatures

Those type HFB fluids which have superior emulsion stability properties at low temperatures (and which are, therefore, more suitable for storage and use under low temperature conditions) should be given designation LT.

2.2.2.5. Summary of classification

A hydraulic fluid for use in mines should be designated by the relevant symbols arranged in the order in which they appear in sections 2.2.2.1. to 2.2.2.4.

Examples of complete classifications are as follows:

HFA 1	Emulsion type HFA fluid with a viscosity classification of 1.
HFAS 1	Solution type HFAS fluid with a viscosity classification of 1.
HFB LT 68	Type HFB fluid with a viscosity classification of 68 and suitable for use at low temperatures.
HFC 46	Type HFC fluid with a viscosity classification of 46.
HFD S 68	Type HFD fluid based on chlorinated hydrocarbons with a viscosity classification of 68.

2.2.2.6. Classification of emulsifying oils and fluid concentrates

Type HFA and type HFAS fluids are normally made by mixing emulsifying oil or fluid concentrate, respectively, with water on site. The fire-resistance of the resulting fluids is largely due to the high concentration of water. The emulsifying oils and fluid concentrates, therefore, should not be given the designation HF but should be otherwise suitably identified by the manufacturer and accepted by the approving or accepting authority according to their suitability for use with specified waters, see the table below. Where an emulsifying oil or fluid concentrate is compatible with more than one of the specified waters, the designation of the hardest water should be given.

Type designation of emulsifying oil or fluid concentrate	Type of water with which the emulsifying oil or fluid concentrate is suitable for use.
Type X	Water with a composition similar to and a hardness not greater than type X test water
Type Y	Water with a composition similar to and a hardness not greater than type Y test water
Type Z	Water with a composition similar to and a hardness not greater than type Z test water

2.2.2.6.1. Test waters

In addition to the requirements of the above classification system (section 2.2.2.6), certain methods of test described in this report, such as the determination of emulsion stability of type HFA fluids (section 5.2.1) require the provision of clearly defined test waters with which to make up the emulsion.

For the purposes, three test waters are specified each of which corresponds to one of the classifications given in section 2.2.2.6. The test waters specified should be made up using chemicals of analytical quality.

a) Type X test water (NCB No 18)

The following salts are added to distilled water:

308 mg/l of $MgSO_4$, $7H_2O$

215 mg/l of $CaSO_4$, $2H_2O$

330 mg/l of NaCl

It should be noted that calcium sulphate ($CaSO_4$) is soluble only with difficulty and even with shaking it may take up to two days for it to dissolve completely. The di-hydrate is usually supplied as a fine powder but when it is not it is advantageous to grind it thoroughly.

b) Type Y test water (DIN 24320 (provisional))

Approximately 100 g of pulverised calcium carbonate ($CaCO_3$) are added to 5 litres of distilled water. Carbon dioxide (CO_2) is passed through the solution from a pressurised container at a vigorous flow rate of approximately 2 to 2.5 l/min for about 1 h. The solution is then filtered until clear and stored in a sealed vessel as a stock solution.

The stock solution is filtered until clear immediately before use and the carbonate hardness is determined in accordance with DEV J3 or equivalent standard. A quantity of the filtrate calculated to produce 10 litres of test water with a carbonate hardness of 8.5°d is poured into a suitable vessel and diluted with distilled water to 5 litres. The following salts are added and dissolved by stirring:

418.0 mg/l of $MgCl_2 , 6H_2O$

89.5 mg/l of NaCl

221.8 mg/l of Na_2SO_4

27.4 mg/l of $NaNO_3$

The solution is then topped up to 10 litres.

As a result of its free dissolved carbonic acid content, the test water has a pH value of less than 6.5. By adding an appropriate quantity of caustic soda solution, still stirring continuously, the pH value is adjusted to the required value of 7.0 to 7.5.

c) Type Z test water (NCB No 19)

The following salts are added to distilled water:

924 mg/l of $MgSO_4 , 7H_2O$

645 mg/l of $CaSO_4 , 2H_2O$

330 mg/l of NaCl

It should be noted that calcium sulphate ($CaSO_4$) is soluble only with difficulty and even with shaking it may take up to two days to dissolve completely. The di-hydrate is usually supplied as a fine powder but when it is not it is advantageous to grind it thoroughly.

The properties and characteristics of the three test waters are compared in the table below.

The stability of an emulsion made with a particular emulsifying oil will depend primarily on the hardness of the water used. This hardness, which arises from the dissolved salts of calcium and magnesium, is expressed in the table in columns 2 to 5. Firstly, it is expressed as an equivalent amount of calcium carbonate ($CaCO_3$) in parts per million ; secondly, as an equivalent amount of calcium oxide (CaO) in parts per 100,000 (°d) ; thirdly, as an equivalent amount of calcium carbonate expressed in parts per 100,000 (°TH) ; and finally, as the milli-equivalent.

The salinity of water which arises from sodium has a lesser effect on the stability of the emulsion.

Table 1. Properties of test waters

Type of test water	Total hardness					Ion concentration ppm					
	$CaCO_3$ equiv. ppm	n Mols/l	°d	°TH	Milli-equiv.	Ca	Mg	Na	Cl	SO_4	NO_3
X (NCB 18)	250	2.5	$14^{(1)}$	25	5	50	30	130	200	240	0
Y (320)	357	3.57	$20^{(2)}$	35.7	7.14	61	50	115	200	150	20
Z (NCB 19)	750	7.5	$42^{(1)}$	75	15	150	90	130	200	720	0

(1) Permanent hardness
(2) 11.5° permanent hardness, 8.5° temporary hardness

Note: Types X and Z waters have the same type of hardness. Type Y water contains $NaNO_3$.

2.2.3 Marking of containers

To ensure that containers of fire-resistant fluid delivered to a mine can be readily identified, they should be clearly and legibly marked in a colour different from white (e.g. yellow or black) with:

the name of the manufacturer;

the name of the fluid;

the batch number of the fluid;

the date by which the fluid should be used; and the nature of any restrictions or limitations on storage or usage imposed by the manufacturer.

In addition for containers normally stacked in the horizontal position the designations determined in accordance with sections 2.2.2.1 to 2.2.2.4 should be clearly and legibliy marked on both ends of the container. In the case of containers of 205 litres capacity or more, the letters should be not less than 50 mm high.

Either the two ends of the container or the central band of the container should, for a width of not less than 200 mm, be coloured in accordance with the colour code given in the following table:

Type of fluid	Colour
Emulsifying oil for type HFA fluid	Pink
Fluid concentrate for type HFAS fluid	Pink with white S
Type HFB fluid	Black
Type HFC fluid	Green
Type HFD R .. fluid	Blue with white R
Type HFD S .. fluid	Blue with white S
Type HFD T .. fluid	Blue with white T
Type HFD U .. fluid	Blue with white U

2.3 STORAGE AND USE

All fluids should be supplied in sound containers, clearly marked and identified in the manner specified in section 2.2.3. Any restrictions or limitations concerning either the storage or subsequent use of the fluids should be declared by the manufacturer.

The life of types HFB, HFC and HFD fluids and that of the emulsifying oils and fluid concentrates from which types HFA and HFAS fluids, respectively, are made should be at least two years from the time of manufacture when the fluid is stored in a container under the conditions recommended by the manufacturer.

Advice on suitable operating temperatures for the four main types of fire resistant hydraulic fluids was given in the Fifth Report (section 1.1) published in 1974. This information is reproduced below but it must be emphasised that it should be regarded as a general guide only. Developments in fluid technology and service experience with particular fluids have not been taken into account.

Type of fluid	Operating temperature range
HFA	+ 5 to + 55°C
HFB	+ 5 to + 60°C
HFC	- 20 to + 60°C
HFD	- 20 to + 150°C

Trials are currently being made with HFC fluids in hydraulic systems with pressurised tanks or special valves. These might lead to higher temperatures being permitted in the future.

2.4 TESTING METHODS

The general philosophy of testing fire-resistant hydraulic fluids for their suitability for use in mines is declared in section 2.1. The tests enumerated in sections 2.4.1 and 2.4.2 are those which are recommended in this Sixth Report as being relevant to those fluids. Fluids of types B, C and D are tested as supplied, except where other concentrations are specified in the test procedures. Types A and AS are tested at the manufacturers recommended concentration for general use, or at a concentration specified by the accepting/approving authority. Certain Toxicological tests on Types A and AS Fluids are carried out at higher concentrations as specified in

Part IV of this report or by the accepting/approving authority.

2.4.1 'PRESCRIBED TESTS'

In this report, the term 'prescribed' is used as a general designation for those tests which form the essential basis for the approval or acceptance of a fluid (see section 2.1.1).

In general, fluids should be subjected to one or other of the alternatives offered in each of the groups of tests for resistance to flame described in Part III, and should be assessed for their possible hazard to health in accordance with Part IV. In addition, fluids should be subjected to each of the prescribed technological tests described in Part V unless the approving or accepting authority is satisfied that, for a particular property, sufficient test data relevant to the fluid exists (see section 2.1.1).

In the case of each prescribed test, the method by which the results are to be assessed and the acceptance criteria to be achieved are specified.

2.4.1.1 Tests for resistance to flame

a) Spray ignition tests

Either : the Community of Six spray test (Fifth Report, Part III)

or : the United Kingdom spray test (Fifth Report, Part VI)

Note : A new test designated the flame length/flow rate spray test has been developed and is currently being evaluated. It is not yet intended that this test be used as an alternative to the above tests but its description has been included to achieve a uniformity of method and conditions of test in the various laboratories.
It is recommended that laboratories exchange test data until definitive acceptance criteria have been determined by the competent authorities.

b) Flame propagation tests

Either : the coal-dust/fluid mixture test (Fifth Report, Part III)

or : the wick test (Fifth Report, Part VI)

2.4.1.2 Tests for the hazard to health

The tests for the hazard to health should include tests for:

a) oral toxicity;
b) irritant effect on the skin;
c) irritant effect on the eye;
d) aerosol toxicity;
e) thermal decomposition products; and
f) neurotoxic effects (phosphoric esters only).

2.4.1.3 Prescribed technological tests

These tests include tests for:

a) kinematic viscosity;
b) stability of emulsions (HFA and HFB fluids only);
c) ageing and thermal stability (HFC and HFD fluids only);
d) pH value (HFA and HFC fluids only);
e) water content (HFB and HFC fluids only);
f) air separation or deaeration ability (HFC only);
g) foaming characteristics (HFA, HFC and HFD only);
h) shear strength (HFB, HFC and HFD only);
i) corrosion inhibiting properties; and
j) compatibility with sealing and packing materials.

2.4.2 'DISCRETIONARY' TECHNOLOGICAL TESTS

The term 'discretionary' denotes those tests which, while not being considered as an essential part of the approval or acceptance test programme, may, nevertheless, be required by a purchaser or accepting/approving authority to verify the suitability of a fluid for a particular application (see section 2.1.1).

In the case of these tests, the criteria to be satisfied are not specified but will normally be the subject of agreement between the manufacturer and the purchaser. In certain cases, however, acceptance criteria are given but this is for information only and guidance on the interpretation of the results.

The discretionary technological tests included in the report comprise tests for :

a) air separation (HFA, HFB and HFD only);
b) shear strength (HFA only);
c) fluid density;
d) neutralisation number (HFB and HFD fluids only);
e) viscosity index;
f) pour point;
g) oil content (HFA fluids only);
h) filterability;
i) thermal stability (HFD fluids for hydrokinetic applications);
j) compatibility with power pack coatings;
k) lubrificating properties; and
l) closed flash point (emulsifying oils).

2.4.3 SUMMARY OF TEST REQUIREMENTS

A detailed summary of the test requirements listed, in general terms, in sections 2.4.1 and 2.4.2 showing their relevance to the different types of fluids is given in the following table:

2.4.3 Table 1. Summary of prescribed and discretionary tests :

Test requirement	Section of report	Type of fluid					
		Emulsifying oils or fluid concentrates	HFA HFAS	HFB	HFC	HFD	Page no.
Tests for resistance to flame							27
Spray ignition (Two alternative methods)	3.2.1) 3.2.2)	-	-	P	P	P	28 28
Flame propagation (Two alternative methods)	3.3.1) 3.3.2)	-	-	P	P	P	48 54
Tests for hazard to health							59
Toxicity $^{(1)}$	4.2	-	P	P	P	-	59
Toxicity $^{(1)}$	4.3	-	-	-	-	P	72
Neurotoxic effects	4.4	-	-	-	-	P	78
Technological tests							79
Kinematic viscosity (Two alternative methods)	5.1.1) 5.1.2)	P	P$^{(2)}$	P	P	P	80 87
Emulsion stability at :							
low temperature (-10°C)	5.2.4	-	-	P$^{(3)}$	-	-	102
ambient temperature (20°C)	5.2.2	-	-	P	-	-	95
medium temperature (70°C)	5.2.3	-	-	P	-	-	99
medium temperature (50/70°C)	5.2.1	-	P$^{(4)}$	-	P	-	92
Ageing	5.3.1	-	-	-	P	-	106
Ageing	5.3.2	-	-	-	-	P	112
pH value	5.4	-	P	-	P	-	118
water content	5.5.1	-	-	P	-	-	121
water content	5.5.2	-	-	-	P	-	125
Air separation	5.6	-	D$^{(2)}$	D	P	D	129
Foaming	5.7	-	P$^{(2)}$	-	P	P	133
Shear strength	5.8	-	D$^{(2)}$	P	P	P	136
Corrosion inhibiting properties (Two alternative methods)	5.9.1) 5.9.2)	-	P	P	P	P	141 145
Corrosion inhibiting properties	5.9.3	P$^{(5)}$	-	.	-	-	148
Compatibility with sealing and packing material	5.10	-	P	P	P	P	160
Fluid density	6.1	D$^{(5)}$	D	D	D	D	174
Neutralisation number	6.2	-	-	D	-	D	175
Viscosity index	6.3	-	-	-	-	D	184
Pour point	6.4	-	D$^{(2)}$	D	D	D	185
Oil content	6.5	-	D$^{(4)}$	-	-	-	190
Filterability	6.6	D	D	D	D	D	193
Thermal stability	6.7.1	-	-	-	-	D$^{(5)}$	197
Thermal stability in couplings	6.7.2) 6.7.3)	-	-	-	-	D$^{(5)}$	201 204
Compatibility with power pack tank coatings	6.8.1))	D	D	-	-	-	208
	6.8.2)	-	-	D	D	-	211
Lubricating properties	6.9	-	D	D	D	D	216
Closed flash point	6.10	D	-	-	-	-	235

In the above table, P denotes a prescribed test (for definition see section 2.4.1) and D denotes a discretionary test (for definition see section 2.4.2).

Notes:
(1) These requirements may be met by test or by assessment (see section 4.1.2).

(2) Not normally carried out on type HFA fluids with a viscosity below 10 cSt.

(3) Applicable to type HFB LT .. fluids only.

(4) Excluding type HFAS fluids.

(5) Only in the U.K.

2.5 BEHAVIOUR OF FLUIDS IN SERVICE

Practical use over a period of time may reveal faults in a fluid or an incompatibility of a fluid with certain operating conditions that could not be foreseen from the results of the laboratory tests carried out in connection with the initial approval/acceptance of the fluid. It would appear essential, therefore, for some service experience to be obtained before a definitive approval or acceptance is granted. The length of the testing period shall be determined by the competent authority.

In this connection, it should be noted that there are a great variety of operating factors such as pressure, temperature, fluid velocity, dynamic effects, etc., which can adversely affect the behaviour of a fluid and thus each apparent service or fluid abnormality should be investigated thoroughly. Such investigation is sometimes made more difficult by the fact that fire-resistant fluids are being used increasingly by industry under conditions which may differ significantly from those at mines and about which mining authorities may have little or no information. It follows, therefore, that there should be a continuous exchange of information between the laboratories of Member States on their experience and evaluation of fluids in service.

2.6 QUALITY CONTROL TESTS

Quality control tests should be carried out periodically to ensure that the composition and properties of a fluid have not deviated significantly from those declared by the manufacturer.

It is strongly recommended that quality control tests are carried out at regular intervals of not more than two years (when they may be considered as reacceptance tests) and at random intervals on new deliveries of fluid.

The approving or accepting authority may require quality control tests to be carried out especially where the properties of a fluid are thought to have changed.

PART III

TESTS

FOR

RESISTANCE TO FLAME

3 TESTS FOR RESISTANCE TO FLAME

3.1 GENERAL

Fire-resistant hydraulic fluids for use in power transmission systems in mines are normally required to be subjected to two types of test:

a) a test of the flammability of a spray of the fluid when it is emitted from a nozzle under a nominal pressure of 70 bar. (The new flame length flow rate spray test under development and assessment utilises a spraying pressure of 50 bar, see section 3.2.3); and

b) a test of the liability of the fluid to propagate flame either when mixed with coal dust or when it has impregnated a wick.

3.2 SPRAY IGNITION TESTS

Fluids in use in the Member States of the Economic Community are currently tested in accordance with the prescriptions of the Fifth Report, that is to say the Community of Six test procedure described in Part III, section 3.1 and reproduced in section 3.2.1 of this report, or the United Kingdom test procedure described in Part VI, section 6.15 of the Fifth Report and reproduced here in section 3.2.2.

A new test procedure has been developed to measure some of the fire-resistant properties of a spray of fluid emitted under pressure and to be applicable to the full range of hydraulic fluids used in mines and in general industrial applications. This test has been designated the 'flame length/flow rate spray test and is described in section 3.2.3. The test is under assessment and evaluation in several laboratories in the Community but the work is not yet complete. For the time being, therefore, the two current test methods, namely the 'Community of Six' spray test or the United Kingdom spray test should continue to be used.

To expedite the evaluation of the new test and the determination of agreed acceptance criteria, it is suggested that, whenever possible, tests be made with the new method in parallel with tests by one or other of the current methods and that the test data so obtained be communicated between the various laboratories engaged in the assessment.

3.2.1. THE 'COMMUNITY OF SIX' SPRAY TEST

3.2.1.1. Principle

The flame of an oxyacetylene torch is directed on to an atomized jet of the fluid under test. The effect of the flame on the jet is observed.

3.2.1.2. Apparatus

a) A container for the test fluid, with a minimum capacity of 1 litre.

b) Pressurizer for atomizing the fluid (See figure 1 or 2)

Figure 1

A cylinder of compressed nitrogen with a pressure-reducing valve set to 70 ± 3 bar. This cylinder is linked by a high-pressure hose to a fluid container which can be heated electrically, from which a closable heat-insulated pipe leads to the spray nozzle.

The temperature of the test fluid can be measured by means of a copper-constantan thermocouple in the container or immediately in front of the atomizing spray nozzle.

Figure 2

A pressure generator, consisting of a pressure pump connected to the spray nozzle via a metal pipe on which are mounted the pressure gauge and a pressure regulator, which discharges excess fluid back into the tank.

The pressure regulator is calibrated to 70 ± 3 bar. The spray nozzle is connected directly to a valve.

c) A test spray nozzle as shown in drawing (see figure 3)

This consists of a hard steel disc with a 0.4 mm diameter discharge orifice in the centre; the edges of this orifice are sharp. The disc must present a smooth surface of 10 mm diameter to the test fluid; the plate with the 0,4 mm orifice is 1,6 mm thick. To ensure maximum security against blockage of the jet, a $\leqslant$ 0,4 mm mesh sieve (144 meshes per cm^2), with a minimum diameter of 100 mm, must be fitted upstream of the jet. The distance between the jet and the sieve should be about 20 mm.

d) An oxyacetylene welding torch which must burn with a 100 mm long conical white flame, edged with blue, and which meets the requirements set out below (see sketch, figure 4).

Each of the two gases leaves its container under pressure and passes through a pressure regulator which is generally placed between a high-pressure manometer and a low-pressure manometer, after which it reaches a precision pressure gauge and passes thence into the torch via a flowmeter.

The indicating range of the precision pressure gauge for oxygen is from 0 to 1,6 bar; the range for acetyle-e manometer is 0 to 1,6 bar.

The RHN type flowmeters are supplied by Rota of Oeflingen (Baden).

The oxyacetylene welding torch, Rex N. 1 type, manufactured by Charledave of Paris, is fitted with a 750 nozzle.

The pressure regulator and the needle valves of the torch are so adjusted that 13 ± 1 l/min of oxygen and 15 ± 1 l/min of acetylene are discharged, at a pressure of 5,0 bar, for the oxygen and 1,0 bar for the acetylene.

So as to avoid frequent readjustment of the gas pressure during a series of tests, it is advisable to fit a tap between the pressure regulators of the pressurized containers and the precision pressure gauges.

e) A metal plate 75 cm wide and 100 cm high, set up at right angles to the jet at a distance of 175 cm from the nozzle, so that its middle-point falls approximately on the axis of the spray nozzle.

3.2.1.3. Procedure

The test must be carried out at an air flow rate of 0.2 to 0.3 m/s in the same direction as the atomized jet.

When the temperature of the fluid is 65 ± 5°C if the apparatus shown in figure 1 is used, or 65 ± 2°C with the apparatus in figure 2, and, when its pressure has been set at 70 ± 3 bar, the spray nozzle valve is opened. An attempt is made to ignite the atomized jet by means of the oxyacetylene flame. For this purpose, the flame is moved along the jet of fluid, at right angles to it, so that the tip of the cone travels along the axis of the jet from the spray nozzle to a point 1,20 m away from the nozzle, so far as possible at a steady speed of 0.04 m/s. The flame is then kept stationary for 5 s at the maximum distance of 1.20 m. Thus the whole test takes 35 s. Five consecutive tests must be carried out.

Before running a new test with a different fluid, the fluid containers and the various parts of the spray nozzle must be rinsed several times in boiling water to which a cleansing agent has been added.

In the case of figure 2, the test apparatus must be flushed carefully with the fluid under test.

3.2.1.4. Results

The results are expressed as follows :

a) the jet of fluid does not ignite = (1)

b) the jet of fluid ignites, but the flame does not reach the screen = (2)

c) the jet of fluid ignites, and the flame reaches the screen = (3)

However, it should be noted that the "Rating 2" may also be given if occasional flame peaks reach the screen when the burner is 1,20 m. from the spray nozzle.

3.2.1.3. Acceptance criteria

The fluid must achieve a rating of 1 or 2 in five consecutive tests.

Note : This method is based on the American method AMS - 3150 C. The atomizing pressure and the dimensions of the nozzle are as defined therein.

DETERMINATION OF IGNITABILITY OF FLUID ATOMIZED UNDER PRESSURE

Diagram A

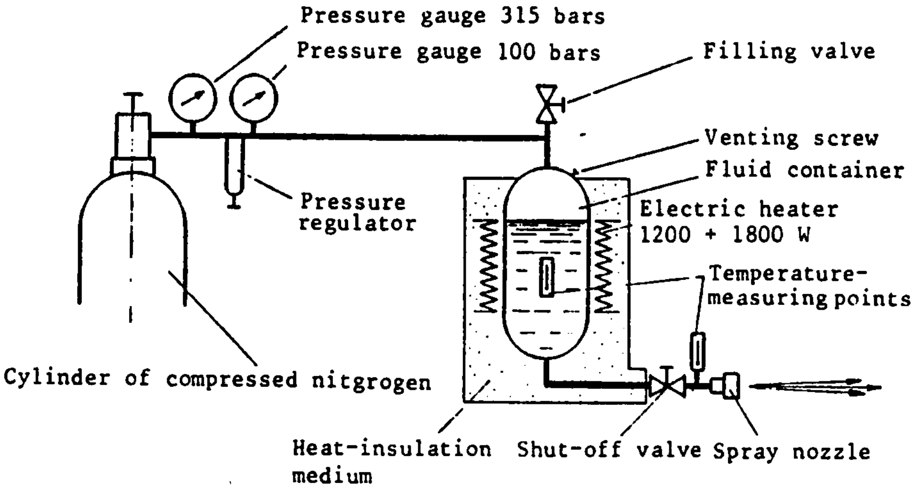


Figure 1. Pressurizer using a nitrogen cylinder

Diagram B

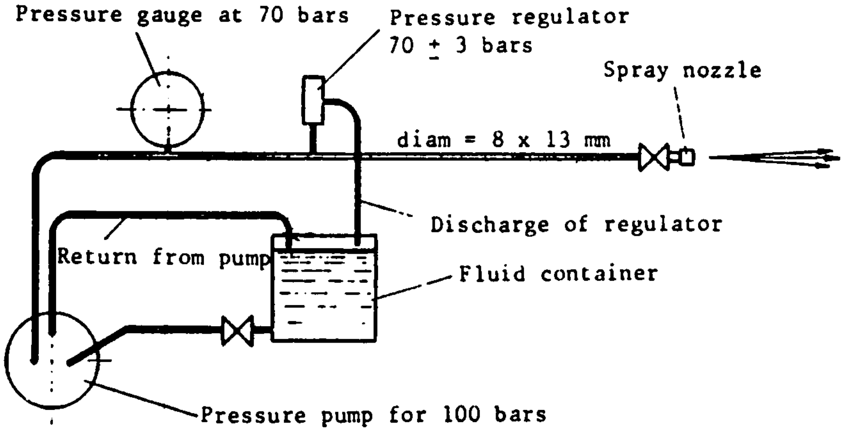


Figure 2. Pressurizer using a pressure pump

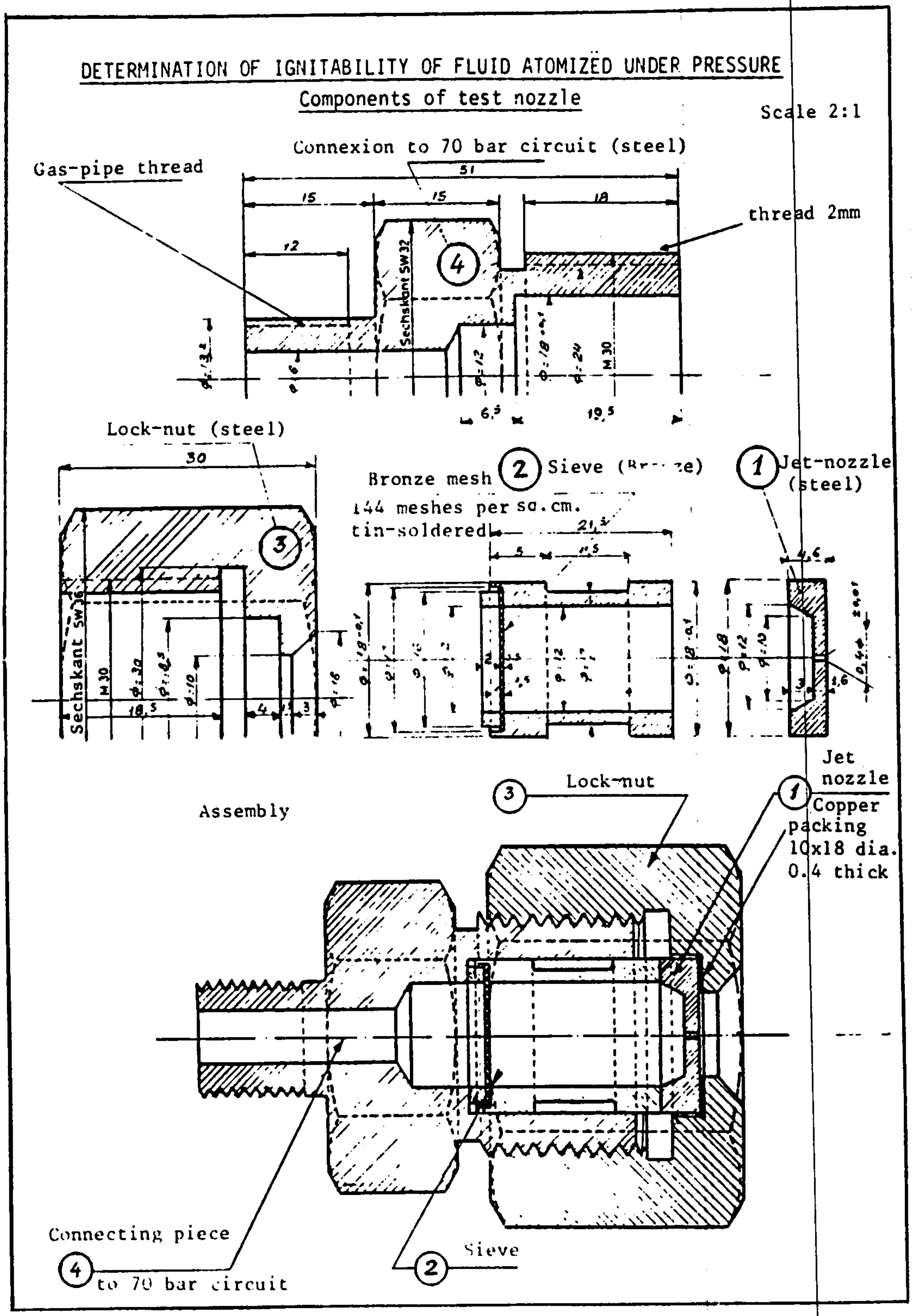


Figure 3. Test spray nozzle

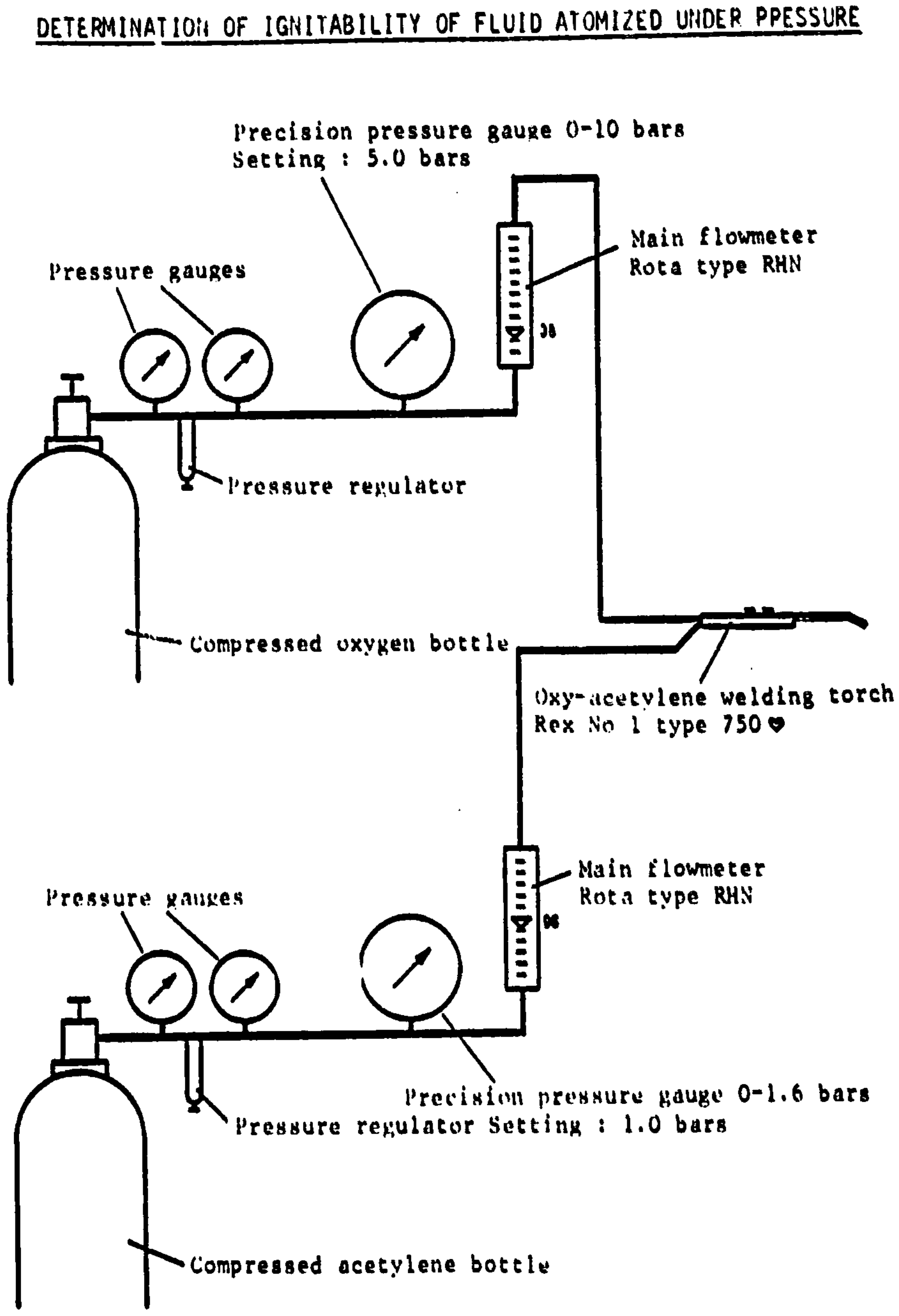


Figure 4. Igniting torch

3.2.2. THE UNITED KINGDOM SPRAY TEST

3.2.2.1. Scope

This method serves to determine selected fire properties of fluids used in hydraulic systems. It may be applied to all types of fluids.

3.2.2.2. Principle

The fluid for testing is atomised under a pressure of 70 ± 3 bar. The spray is directed on to a precisely defined oxygen/acetylene flame. After ignition of the spray the igniting flame is withdrawn and the persistence of burning of the spray is recorded.

3.2.2.3. Apparatus

The fluid, heated to the test temperature as specified in section 3.2.3.4. below, shall be sprayed from an 80° hollow-cone spray oil-burning nozzle type F 80° 2.5 PL manufactured by Monarch and supplied by PP Controls Ltd., Cross Lances Rd, Hounslow, Middlesex. This nozzle is rated at 9.5 l/h at 7 bar presure. Throughout the test a spraying pressure of 70 ± 3 bar is maintained by pressurising with nitrogen from a cylinder. The igniting flame shall be an oxyacetylene flame from a welding blowpipe (either high pressure model DH with a No 10 nozzle with both gases at 0.2 bar or "Saffire" lightweight model with a No 10 nozzle with both gases at 0.3 bar). The igniting system parts are supplied by British Oxygen Co. Ltd.

3.2.2.4. Preparation

The fluid shall not be stored under temperatures less than 0°C nor in excess of 30°C. Care shall be taken to ensure that the fluid is properly mixed before samples are withdrawn for test and that the samples are representative of the whole.

A sample of 1100 ml shall be placed in a 1500 ml glass beaker and heated to the test temperature at a rate of approximately 3 ± 0.5°C/min. For water based fluids or emulsions containing water, the test temperature shall be 65°C ; for other fluids the temperature shall be 85°C.

Any change in composition of the fluid as a result of heating at the above rate will be observed and a note made of any such change.

3.2.2.5. Procedure

The pressure vessel of the apparatus with the water jacket filled so that the vessel is completely immersed, shall be raised to the appropriate test temperature. When the sample has been heated to its test temperature as described in section 3.2.2.4., it shall immediately be transferred to the pressure vessel and the test carried out. When the spray is established the igniting flame shall be repeatedly applied to and withdrawn from different positions along the length of spray and the persistence of burning after each withdrawal shall be noted.

3.2.2.6. Results

When the fluid has been tested in accordance with the procedure in section 3.2.2.5. the maximum recorded persistence of burning shall be noted.

3.2.2.7. Acceptance criteria

The maximum persistence of burning of the flame in the spray after withdrawal of the igniting flame shall not exceed 30 seconds.

3.2.3 THE FLAME LENGTH/FLOW RATE FLAME TEST

Note : This method of test is being assessed and the acceptance criteria have yet to be determined, see section 3.2.

3.2.3.1 Scope

This method of test serves to determine some of the fire-resistant properties of fluids used in hydraulic power transmission systems in mines. It may be applied to all types of fluid.

3.2.3.2 Principle

An atomised spray of the fluid, heated to the specified test temperature, is produced in a test chamber in which the air movements are closely controlled. The flame from an oxygen/propane burner is directed into the spray at right angles to the axis of spraying and the maximum horizontal length of flame extension from the point of ignition is noted during a measured period. The mass throughput of the fluid is measured and the test result is expressed as a ratio of mean flame length to mean rate of mass throughput.

3.2.3.3 Apparatus

The test chamber is 3.75 m long, 2.5 m wide and 2.5 m high. The chamber is constructed from sheet steel and is fitted with a door, an observation window a chimney and two ventilation inlets. The ventilation is supplied by two Vent Axia 305 mm window type fans running at 1,325 rev/min, or other similar fans, to provide the prescribed air flow (see section 3.2.3.5). Details of the construction of the chamber are shown in figures 1 and 2.

The fluid spray equipment consists of a cylinder containing 6 litres of the test fluid pressurised with nitrogen to 50 ± 1 bar. The cylinder is heated by electric band heaters and the temperature of the fluid is controlled to 65 ± 2°C. The fluid flows through a solenoid valve to an oil spray nozzle (Steiner Type 1.0, 80° hollow cone, rated at 3.8 l/h). The temperature of the fluid is checked by a thermocouple immediately prior to ejection from the nozzle. Details of the spray equipment are shown in figure 3.

Ignition of the spray is effected by using an oxygen/propane torch (British Oxygen Company - Part Nos. 143456, 143758, 143813 and 157554). Oxygen is supplied at 22 ± 1 l/min at a pressure of 0.14 bar and propane is supplied at 10 ± 0.5 l/min at a pressure of 0.14 bar. The igniter is positioned in relation to the spray nozzle as shown in figure 4.

The flame length measuring system consists of two bars, one graduated, attached to the side of the test chamber. These support a sliding pointer system with a mask to minimise dazzle when the main hody of the flame is very bright. The pointer system incorporates a vertical sighting wire which, when used in conjunction with the vertical edge of the mask, eliminates parallax effects, see figures 5 and 6.

(Fully detailed specifications of the apparatus are available from the Secretariat of the Safety and Health Commission for the Mining and Extractive Industries, Luxembourg).

3.2.3.4 Preparation

Care must be taken to ensure that the fluid is properly mixed before samples are withdrawn for test and that the samples are representative of the whole. The apparatus should be cleaned between tests as described in section 3.2.3.8.

Before a testing session, air movements in the spray zone, must be checked using a remote reading low velocity anemometer. It is convenient to use a hot wire anemometer to measure the air velocities but if a directional anemometer is used both horizontal and vertical air movements should be measured and both should be within the specified range. During these air velocity measurements, the door of the test chamber should be tightly closed and persons should not remain in the chamber.

The air velocities shall be within the range 0.15 to 0.30 m/s at the positions indicated in figure 7. The velocities at other positions at the sides, above and below the spray cone may also be measured but experience during the development of the test indicated that this is not necessary. (This may be checked during the commissioning tests with the chamber).

3.2.3.5 Procedure

Immediately before and after each test, the fluid mass throughput must be measured over a one minute period by collecting the ejected fluid in a preweighed vessel, the fluid being pressurised to 50 bar and at a temperature of 65°C.

Flame length is defined as the separation between two parallel planes normal to the axis through the spray nozzle, the first plane being that containing the axis of the igniter flame and the second plane being that further along the nozzle axis in the direction of spraying that is just reached by flame at any point in the plane. Maximum flame length is the instantaneous measured maximum separation of these planes observed during a twenty second period, having excluded the first two seconds after initiation of the flame.

During the twenty second measuring period, the pointer is slid along the graduated bar to coincide with the greatest length of flame observed, this length being recorded to the nearest centimetre. A total of 50 such measurements concludes the test.

While flame length measurements are being made, the door of the chamber shall be tightly closed and no person shall remain within the chamber.

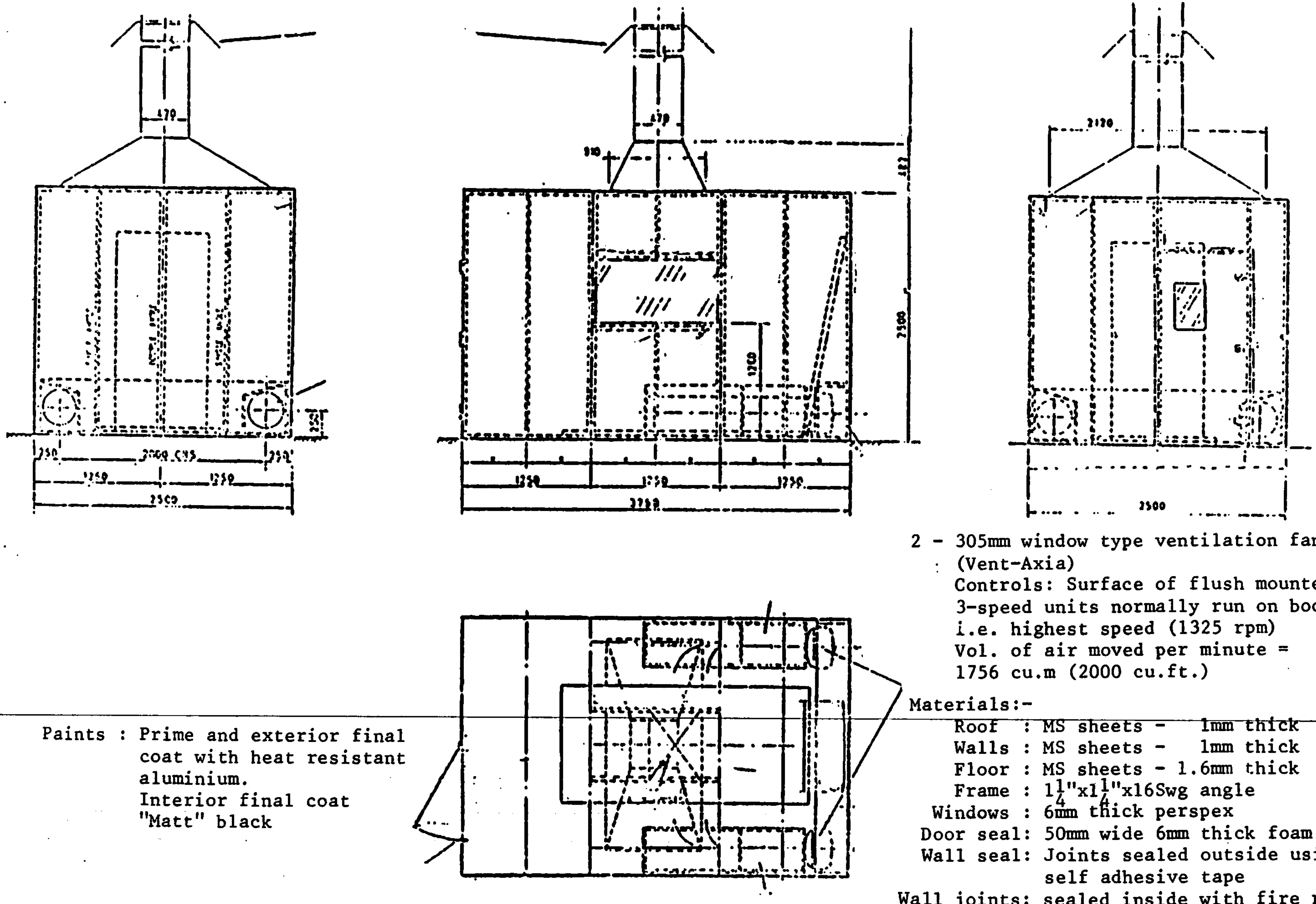


Figure 1 General arrangement of test chamber

2 - 305mm window type ventilation fans
(Vent-Axia)
Controls: Surface of flush mounted
3-speed units normally run on boost
i.e. highest speed (1325 rpm)
Vol. of air moved per minute =
1756 cu.m (2000 cu.ft.)

Materials:-
Roof : MS sheets - 1mm thick
Walls : MS sheets - 1mm thick
Floor : MS sheets - 1.6mm thick
Frame : $1\frac{1}{4}$"x$1\frac{1}{4}$"x16Swg angle
Windows : 6mm thick perspex
Door seal: 50mm wide 6mm thick foam strip
Wall seal: Joints sealed outside using self adhesive tape
Wall joints: sealed inside with fire resistant gaskets

Paints : Prime and exterior final coat with heat resistant aluminium.
Interior final coat "Matt" black

PART III 3.2.3.

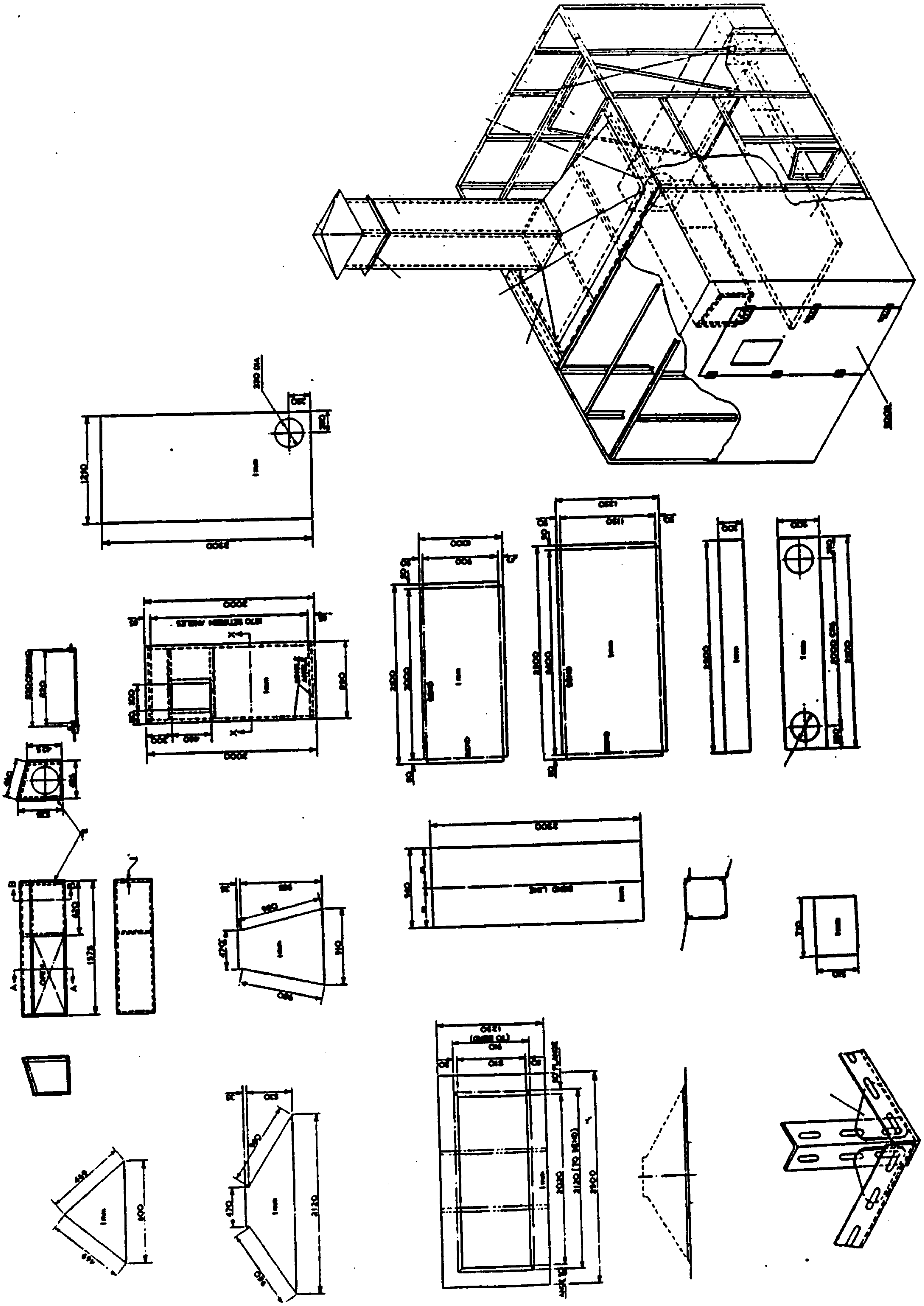


Figure 2. Component parts of test chamber

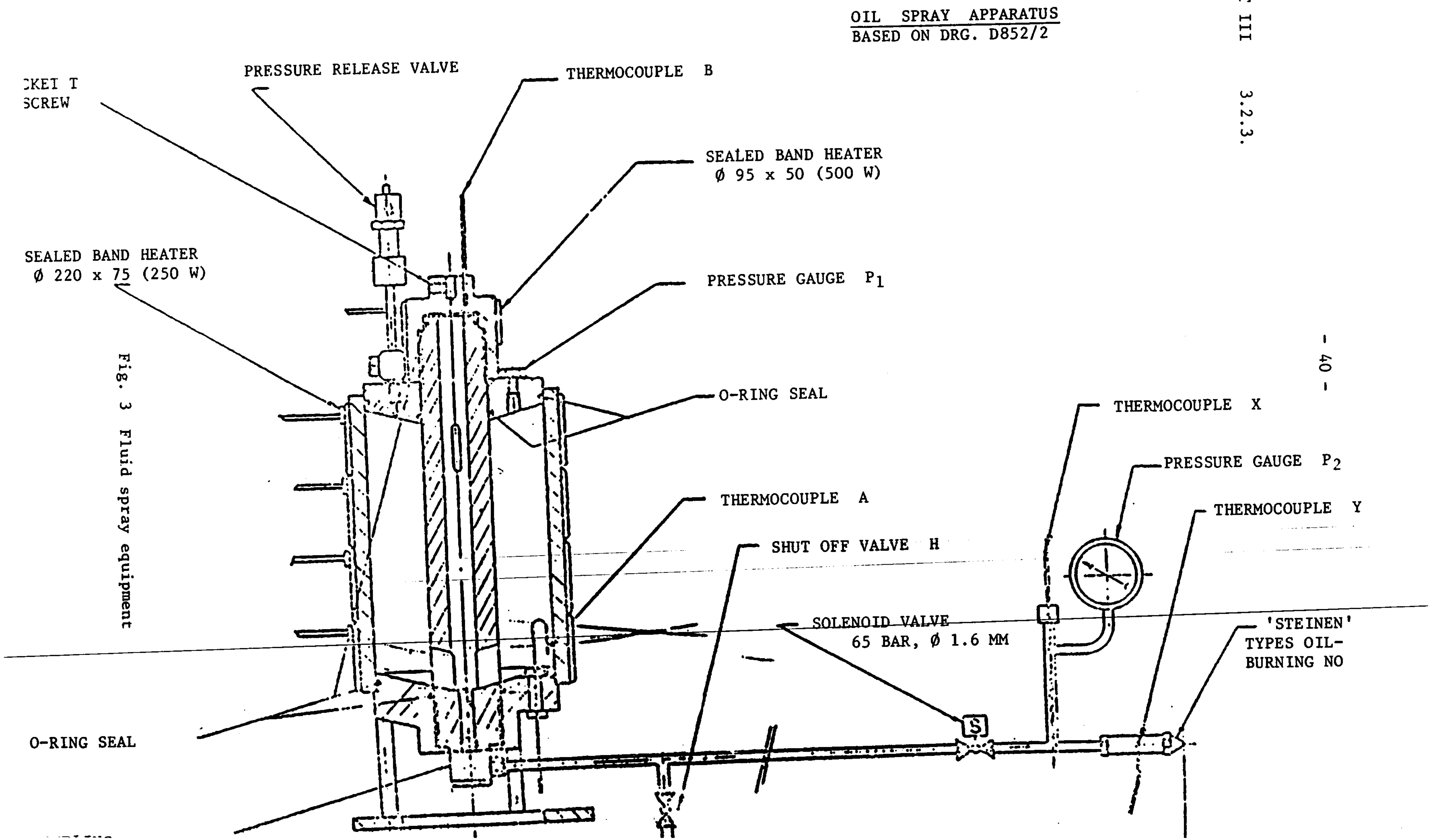
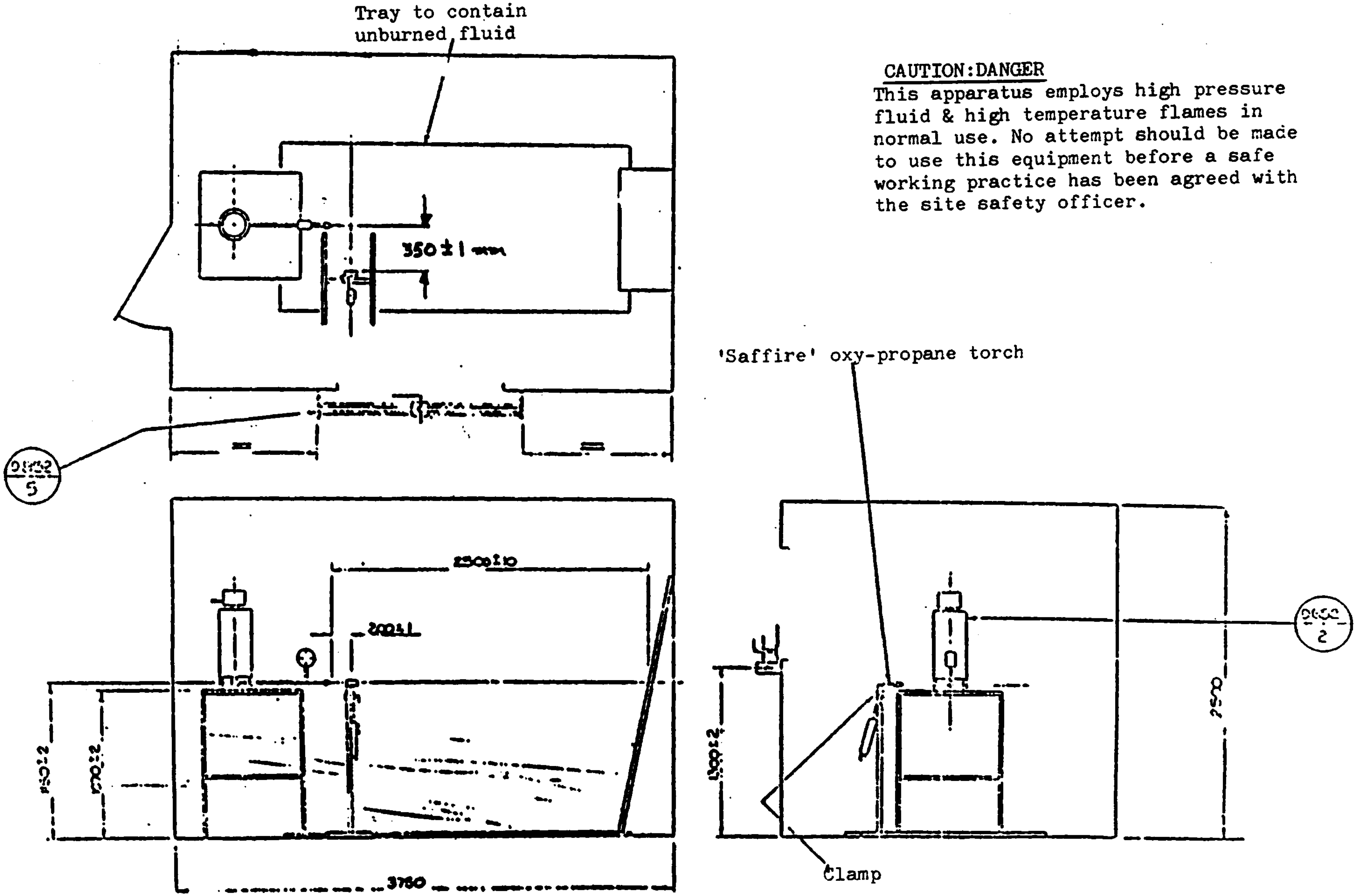


Fig. 3 Fluid spray equipment



CAUTION:DANGER

This apparatus employs high pressure fluid & high temperature flames in normal use. No attempt should be made to use this equipment before a safe working practice has been agreed with the site safety officer.

Fig. 4 Position of igniter

PART III 3.2.3.

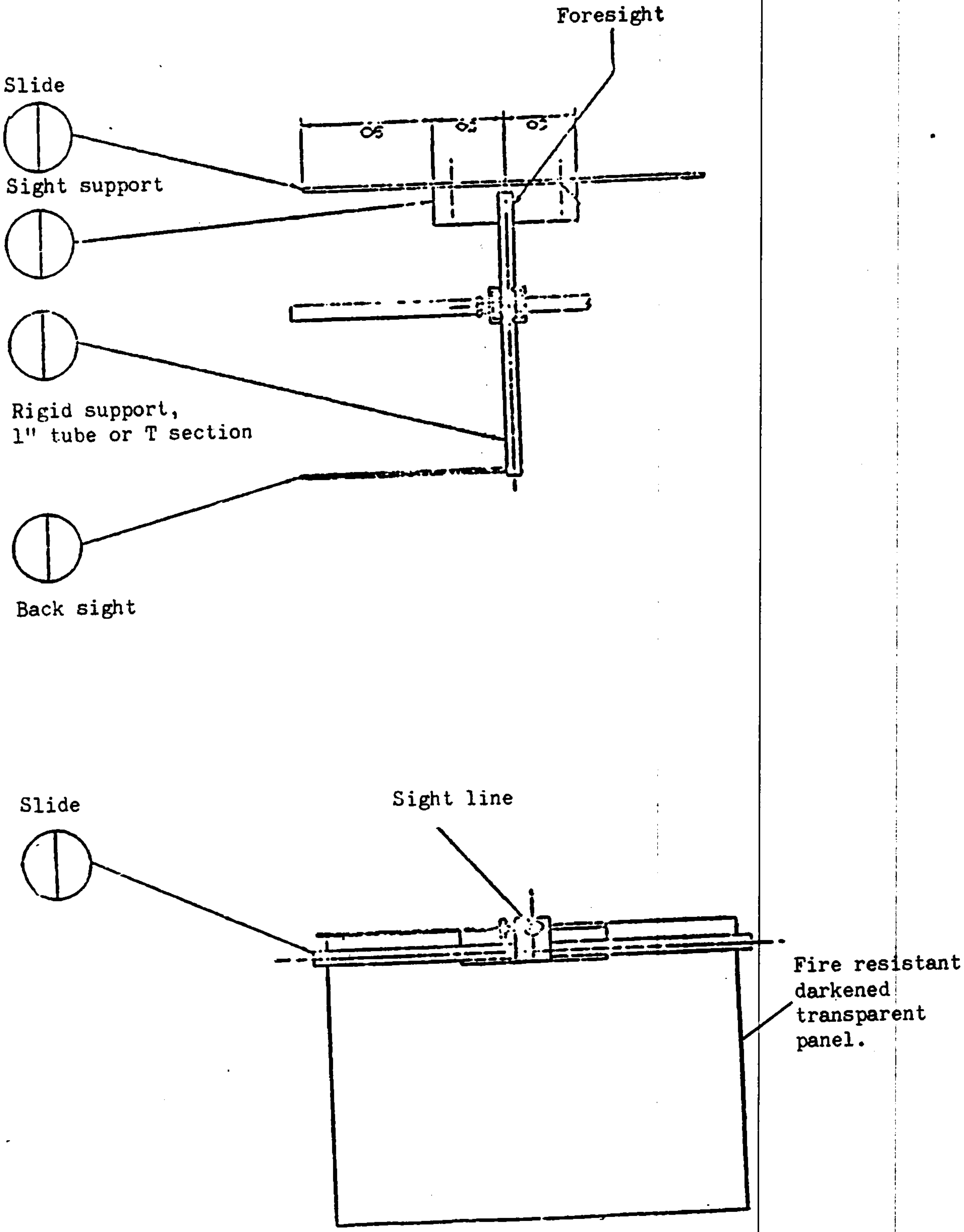


Fig. 5 Runner sight assembly

PART III 3.2.3.

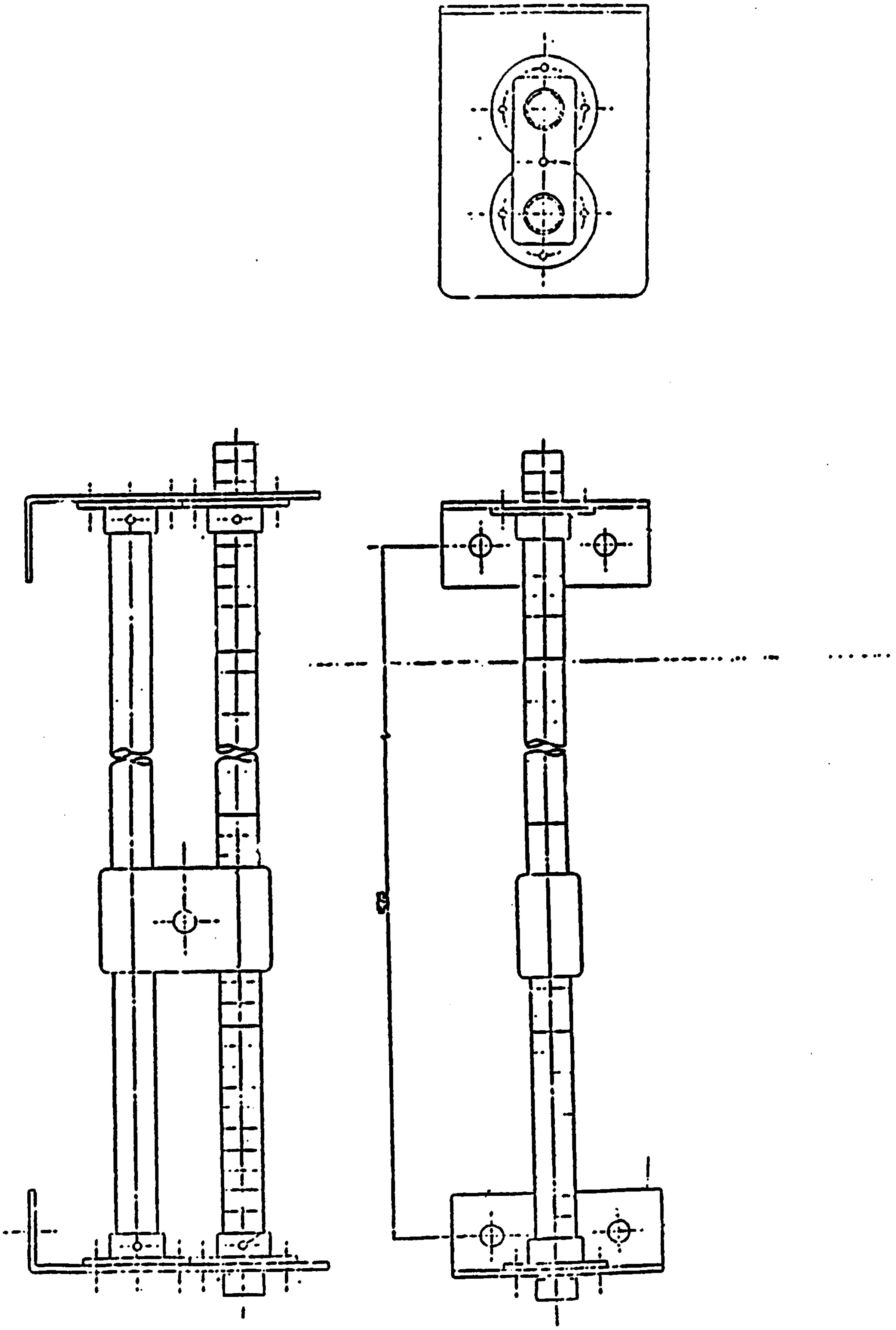
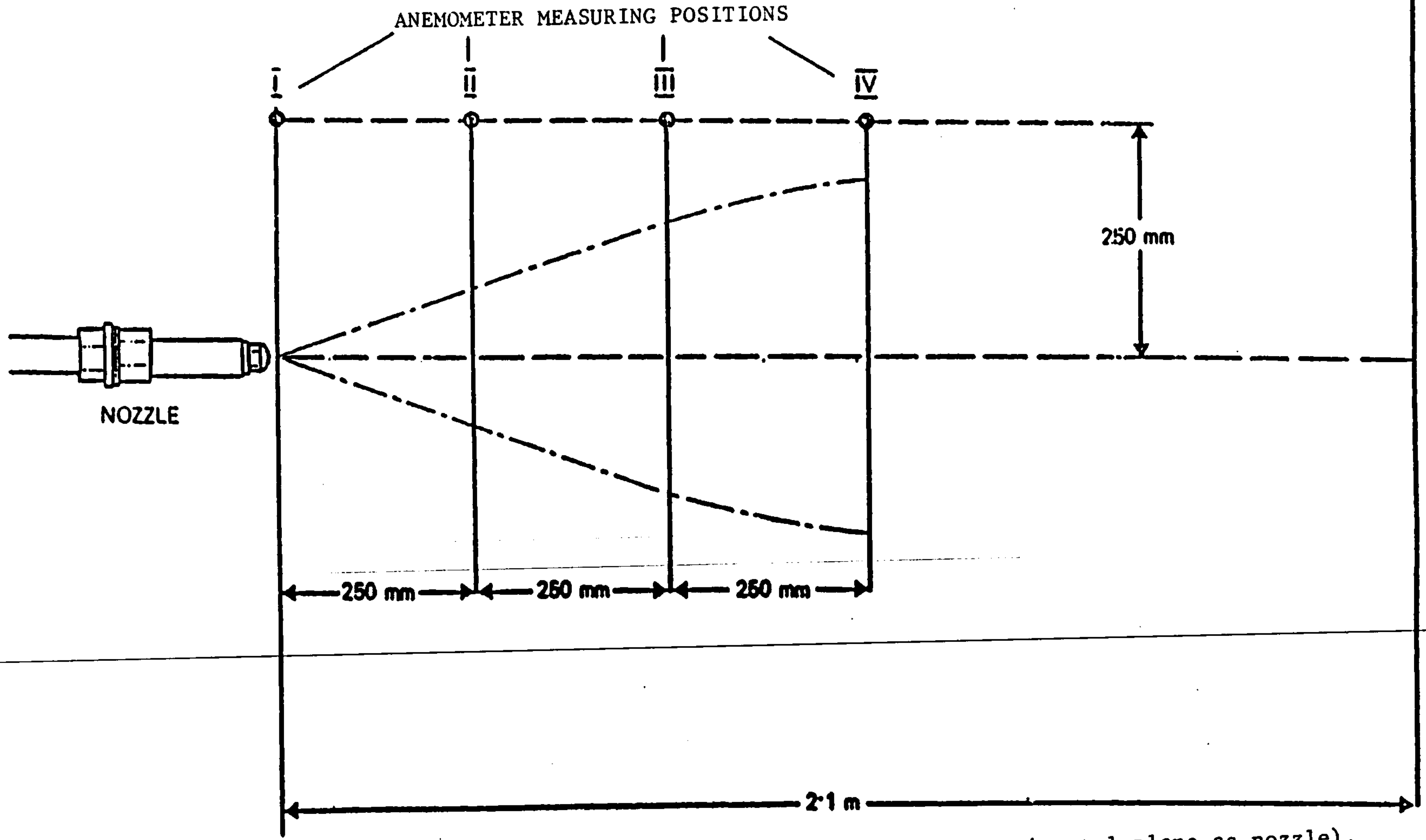


Fig. 6 Runner sight assembly



Anemometer recording positions (all in same horizontal plane as nozzle).

Fig. 7 Anemometer recording positions

3.2.3.6. Results

After 50 measurements of the maximum flame length, each recorded over a twenty second period, the arithmetic mean is calculated. The mean flame length per unit mass throughput of fluid is then calculated and the result expressed in cm/g/min. (see Note 1).

Note 1 : During the assessment of this method of test, the volume throughput of the fluid may also be determined and the results expressed in $cm/cm^3/min$.

3.2.3.7. Acceptance criteria

As this method of test is still under development and evaluation in the Member States, definitive acceptance criteria cannot be specified. It is anticipated, therefore, that each Member State will determine its own criteria for the time being.

3.2.3.8. The cleaning of spray equipment between tests

To avoid contamination which might invalidate test results the following cleaning procedure must be carried out between tests.

Fluid remaining in the pressure vessel from the previous test must be ejected from the hand operated valve H in figure 3.

A polythene wash bottle filled with a suitable solvent (see end of this note) must be used to wash down the inside surface of the pressure vessel until it appears clean. Soiled solvent will pass out of valve H. After removing the nozzle the wash bottle delivery tube must be pushed into the flow tube and solvent forced through the flow tube, solenoid valve S, and out through valve H. The cleaning process must continue until clear solvent is seen to be issuing from valve H.

The cleaning procedure is best carried out immediately after a test has been completed whilst the equipment is still hot. In this case any solvent remaining after cleaning will quickly evaporate. Otherwise the equipment will need reheating to remove traces of solvent.

No material other than solvent must be used in the cleaning procedure because the use of other materials might subsequently cause blockages of the nozzle.

The nozzle and its strainer must be cleaned thoroughly as follows: separate the strainer from the nozzle. Place both in a beaker and cover with Toluene solvent. Manipulate the beaker so that swirling solvent frees any residue of fluid. Replace the toluene and repeat the process. The nozzle and strainer should now be fairly clear of fluid residue. The nozzle must next be more thoroughly cleaned by forcing solvent under pressure through its orifice and swirl chamber. This is most easily achieved by using a special vessel as illustrated in figure 8.

The vessel should be filled with solvent, the nozzle fitted in place and the vessel hung up in a fume cupboard and air pressure at about 3.5 bar applied to it.

A spray of a mixture of hydraulic fluid and solvent having a cloudy appearance will first appear and this should quickly clear to become a spray of clear solvent. The process should be continued until a clear spray is obtained. The nozzle should then be quite clear of hydraulic fluid, and the residual solvent will soon evaporate leaving the nozzle ready for re-use.

Brushes or other fibrous materials must not be used for cleaning the nozzle or strainer because of the possibility of a fibre finding its way into the nozzle orifice and affecting its spraying behaviour.

List of parts : Wash bottle with delivery tube, capacity 500 ml, from any laboratory equipment supplier.

Solvents (1) : For cleaning the pressure vessels :

Acetone (Minimum assay 99%) or similar degreasing fluid.

For cleaning the nozzle and strainer :

Toluene (Sulphur free).

Note 1.

Toluene is a toxic, volatile hydrocarbon which is absorbed by inhaling the vapour or through the skin by contact with the liquid. Use adequate ventilation and avoid skin contact. Acetone is highly flammable and must only be used under conditions of adequate ventilation.

PART III 3.2.3.

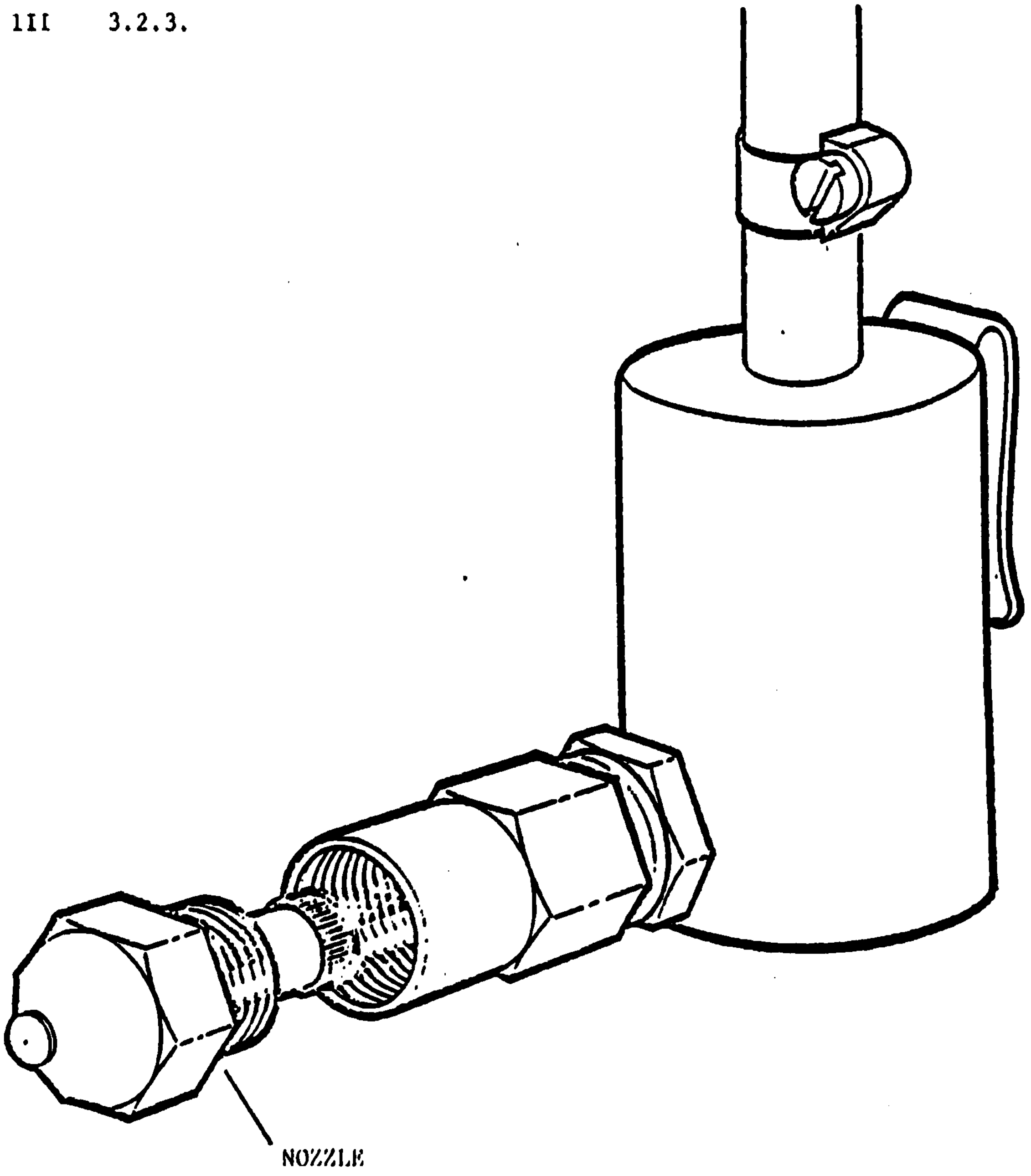


Figure 8 Small pressure vessel for cleaning nozzle

3.3. FLAME PROPAGATION TESTS

Fluids for use in the Member States of the Economic Community are currently tested for their ability to propagate flame by either the fluid/coal-dust mixture test described in section 3.3.1. or the wick test described in section 3.3.2.

3.3.1. METHOD OF TEST FOR FLAME PROPAGATION IN A FLUID/COAL DUST MIXTURE

3.3.1.1. Principle

The propagation of a flame is measured in a mixture of 75 g of coal dust and 37.5 ml of fluid. The test is carried out in an enclosure at ambient temperature without artificial ventilation.

3.3.1.2. Apparatus (see figures 1 to 3)

1) Standard propane gas burner of "Heintz" type, with controls for both air and gas.
2) Propane gas cylinder (1).
3) A standard base plate with mounts for the various parts of the apparatus (support for the test piece plate, support for the thermocouples and support for the graduated scale).
4) Support for the test piece plate, held in position only at both ends.
5) Test piece plate.
6) Pyrometer couple.
7) Graduated scale.
8) Rotameter of type RLT or L, Apparate und Maschinenbau Dr. Hennig KG., D-7867 Oeflingen.
9) Water-filled U-shaped tube manometer, 1 m difference in level.
10) A precision propane reducing valve.
11) Metal jigs for making the test pieces.
12) Slides for making the test pieces.
13) Porcelain mortar and glass or porcelain pestle weighing about 200 g.
14) Timer.
15) Watch-glass.

Note: (1) The propane cylinder must not be emptied of more than 80% by weight of the gas contained in it.

3.3.1.3. Coal Dust

Dortmund Standard Dust is now used (in place of the previous Montramber dust). The characteristics of this are as follows :

Moisture	0.46 %	(tolerance ± 56.5% of 0.46%)
Ash, dry basis	4.7 %	(tolerance ± 13.4% of 4.7 %)
Volatile matter	23.5 %	(tolerance ± 4.4% of 23.5%)
Particle size	0 to 50 nm	Median size of 94%, 18-20 nm

3.3.1.4. Preparation

a) The test mixture

The test mixture must be in proportions of 75 g coal dust to 37.5 ml of the fluid to be tested. Three mixtures are prepared, each sufficient for 10 determinations.

In principle a porcelain mortar and a glass or porcelain pestle should be used. First, all the coal dust is placed in the mortar, then the fluid is gradually added while the contents are stirred. The mixture should be stirred for 15 min. Each test mixture is covered by a watch-glass.

b) The test piece

An interval of 1 hour should be allowed between completing the test mixture and beginning the preparation of the test pieces. Before starting the latter, the test mixture must be stirred once again. The test piece is 20 mm wide, 3 mm thick and at least 150 mm long. The test piece plates bear a datum mark 150 mm from the end to indicate the starting point of the test piece. The lateral clearances are obtained by means of slides. 10 pieces are produced from each test mixture.

3.3.1.5. Procedure

The gas pressure is set at 650 mm water gauge and its flow at 26 l/h corresponding to 60 divisions of the rotameter scale. The air intake is adjusted so that the free height of the flame is about 14 cm and that of its blue cone approximately 35 mm. Under these conditions, the temperature of the flame, measured 5 mm below an empty sheet steel plate, is 1,000 ± 30°C.

The various adjustment rings are locked in position by suitable means. The adjustments are checked before the test, 1/2 hour after the burner is first lit. The empty steel plate is then replaced by the test piece plate.

The centre of the burner is positioned vertically below the point of origin of the test piece, as shown on the assembly drawing, figure 1. The tip of the burner is 45 mm away from the under-surface of the steel plate. A timer is started as soon as the steel plate is fitted, and after a heating period of 5 minutes, the burner is removed.

The following are noted :

a) the farthest distance in mm travelled by the tip of the flame ;

b) the time taken for the flames on the test piece to die out ;

c) any anomalies : glowing after extinction of the flame, extinctions followed by renewed ignition, etc.

Each test with a given fluid consists of 2 x 10 measurements, each test piece being used only once.

3.3.1.6. Results

For each of the mixtures, the result is expressed as the arithmetic mean of the 10 distances measured in mm. If the difference between the two means does not exceed 10 mm the final result will be the mean of both results. If the difference exceeds 10 mm, a third test will be carried out consisting of 10 determinations with a new mixture.

Notes :

a) In preparing the test piece care must be taken to press the paste uniformly.

b) The steel plates for the test pieces are refurbished by scraping and cooling. The remaining traces are removed by scraping with an abrasive cloth of suitable grade.

c) After ten or so determinations, it is advisable to renew the datum mark on each support by means of a metal scriber.

3.3.1.7. Acceptance criteria

The flame in the fluid/coal dust mixture must not extend appreciably the field of action of the burner flame. The arithmetic mean of the two sets of 10 measurements must not exceed 100 mm, and 95% of the individual measurements (i.e. 19 out of 20) shall be less than 130 mm. The anomalous measurement may be eliminated from the calculation of the mean. Furthermore the mean values of the two test mixtures must not differ by more than 10 mm. If a third series of tests is required, the following method of assessing the fluid is applied.

The mean is taken of the three test series. It shall not exceed 100 mm and 28 of the 30 individual results shall be less than 130 mm. The difference between the means is not taken into account in this case.

PART III 3.3.1.

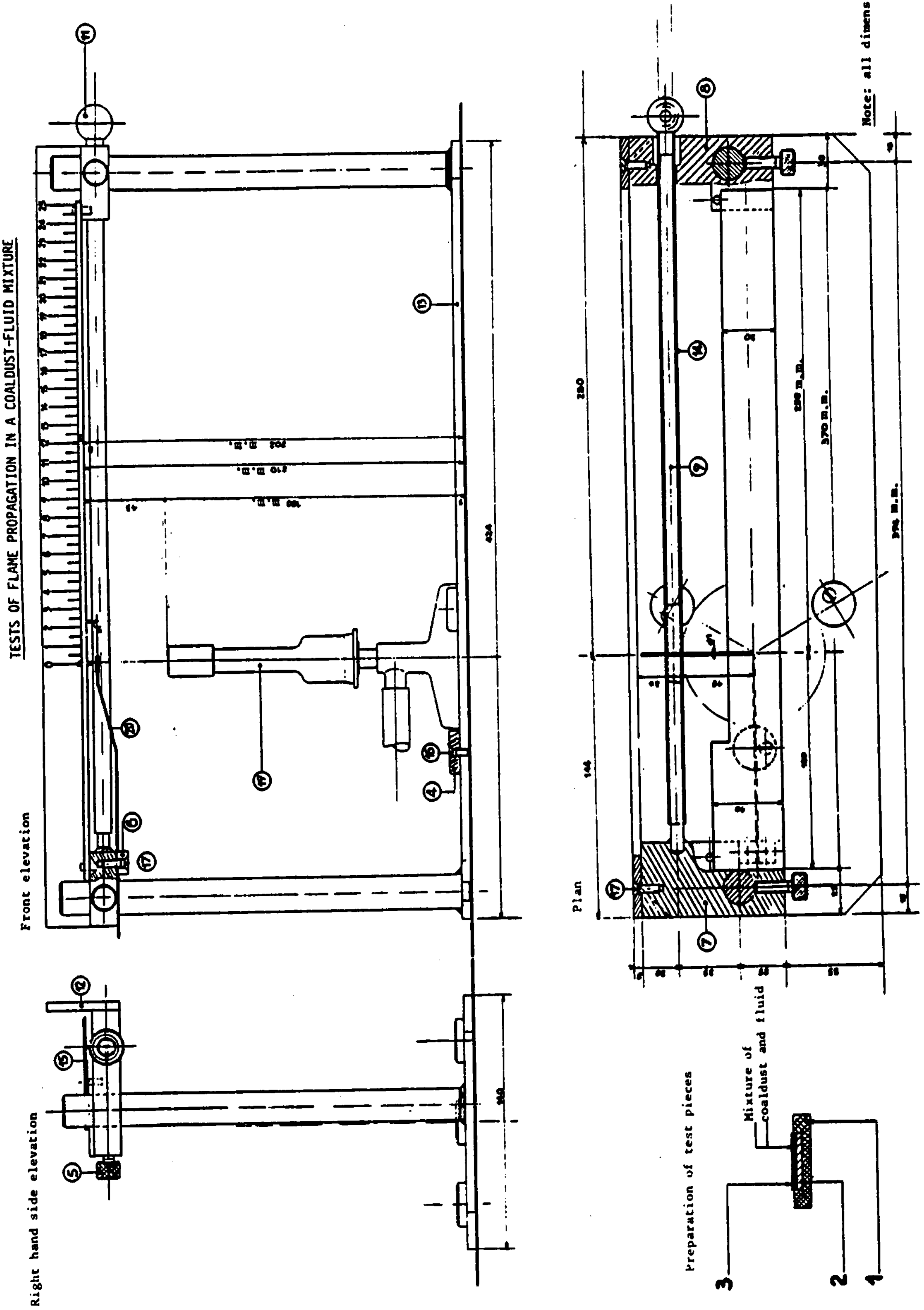
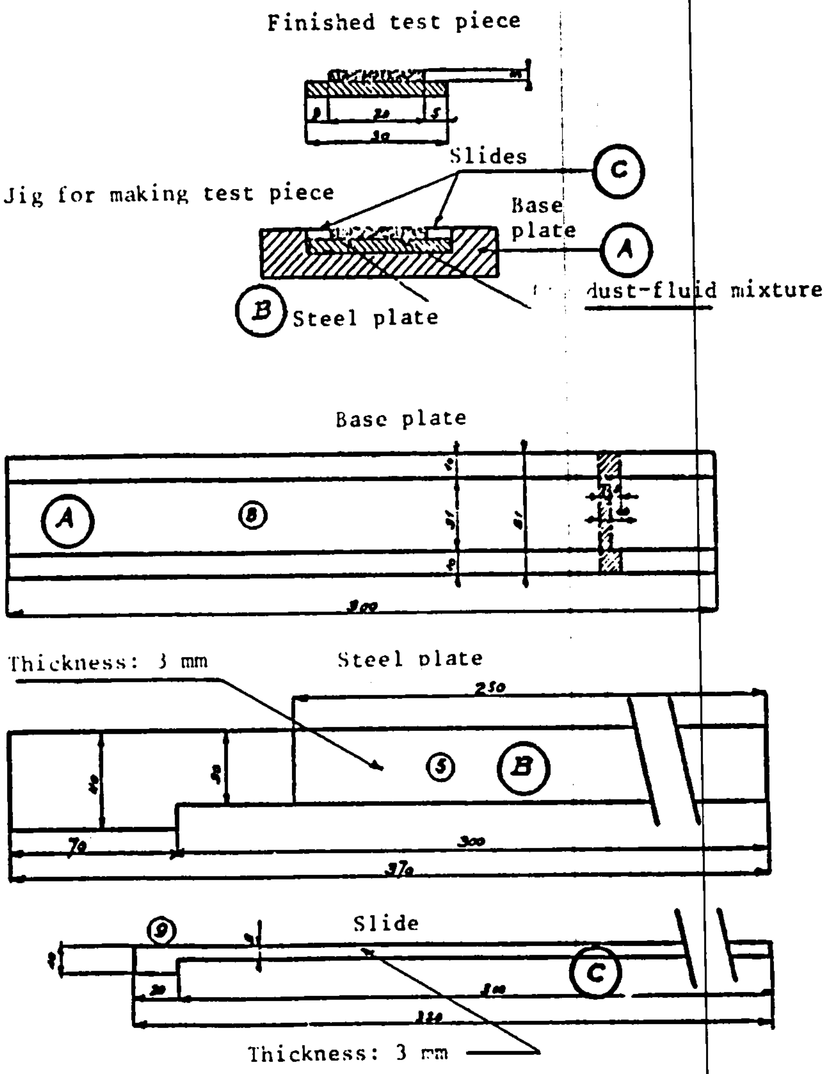


Figure 1 Assembly of apparatus

PART III 3.3.1.

MAKING OF TEST PIECE

Scale 1:1 and 1:2



B

Note: all dimensions in mm.

Figure 2 Test piece

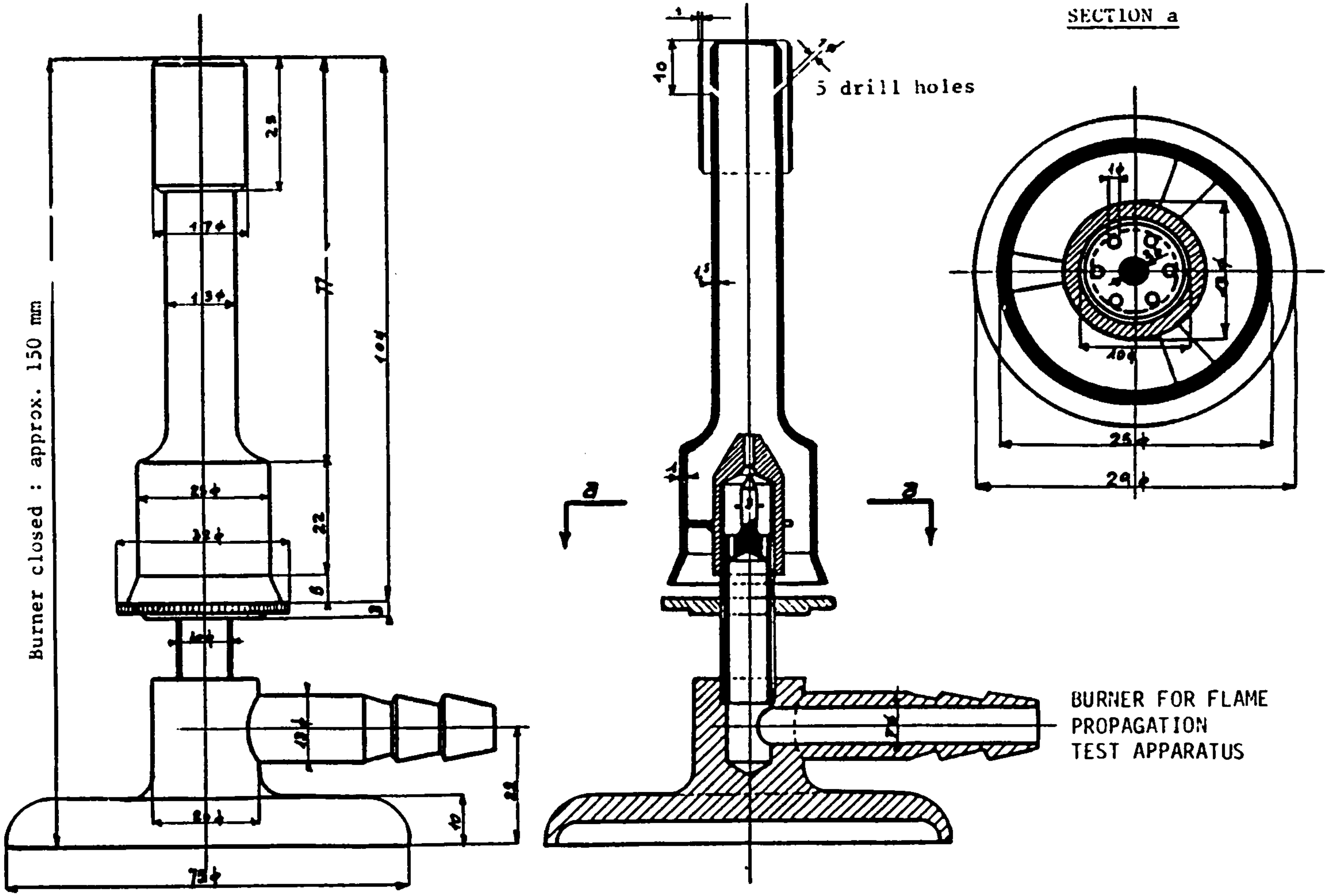


Figure 3 The burner

Note: all dimension in mm.

3.3.2. METHOD OF TEST FOR FLAME PROPAGATION ALONG A FLUID SOAKED WICK

3.3.2.1. Principle

A length of woven asbestos tape shall be soaked in the fluid under test and then placed in a reservoir of fluid with one edge exposed, forming a wick. A small igniting flame shall be applied to the exposed edge of the wick and the persistence of flame on the wick after removal of the igniting flame shall be measured. Six repeat measurements shall be carried out for each of five different periods of application of the igniting flame.

3.3.2.2. Apparatus

The tape shall be of woven asbestos of nominal width 25 mm and nominal thickness 0.40 mm, complying with BS 1944/73 and approved by the National Coal Board. Approved tape will normally be kept by the NCB for purchase by recognised fluid suppliers only. Orders for tape should be for one or two coils (not more, as each coil contains about 33 m of tape) and should be sent to Purchasing and Stores Department, NCB Mining Research and Development Establishment, Ashby Road, Stanhope Bretby, Burton-on-Trent, Staffordshire.

The reservoir shall be 25 mm wide and 19 mm deep as shown in figure 1. A clip for holding the wick shall be fixed to each end of the reservoir and the distance between the inner edges of the clips shall be 178 mm. Each clip shall have a reference mark at a height of 28.5 mm above the base of the reservoir.

The burner is equiped with a British Oxygen Co, No 1 welding nozzle, and may be fabricated from a length of tube, one end of which is threaded to accomodate the nozzle, as shown in figure 2.

The burner shall be connected by means of flexible tubing to a cylinder of liquid propane through a suitable regulating valve. Suitable valves are usually incorporated in Bunsen burners designed for use with liquid propane. To utilise such a valve the Bunsen burner chimney should be removed and the flexible tubing connected directly to the burner jet. The flame at the nozzle is not aerated.

The testing jig is shown in figure 2 and the burner is mounted on a pivot bar so that it may be moved between the igniting position and the setting position.

The jig provides adjustment and positive location for the reservoir and the burner such that the wick and the nozzle are in their correct relative positions when the burner is in the igniting position. A stop is provided so that the burner may be positively located in the correct setting position for setting the height of the flame. The burner can be moved along the pivot bar so that it may be adjusted to any position along the length of the wick.

The soaking bath is approximately 200 mm long, 30 mm wide and 40 mm deep.

A stop-watch is required, reading to 0.1 s.

3.3.2.3. Preparation

a) Fluid sample

A sample of not less than one litre shall be supplied for test. Care shall be taken to ensure that the sample supplied for test is representative of the bulk.

b) Wick

Each wick of length 190 mm with edges free from knots shall be suspended in still air with the long edges vertical, and shall be ignited by applying a small flame to the lower edge. The flame shall be allowed to spread vertically so that the whole of the wick is burned free of combustible material. It is recommended that fifteen wicks should be prepared. No wick shall be used for test purposes until at least one hour after preparation.

c) Jig

To facilitate adjustment of the testing jig a dry wick shall be mounted in the clips in the reservoir with the top of the wick as defined by the top of the uppermost warp thread, in line with the reference marks on the clips. The wick shall be lightly tensioned. The reservoir shall be level, the plane of the wick shall be vertical and the top of the wick shall be horizontal. The testing jig shall be adjusted so that when the burner nozzle is horizontal and level with the top of the wick to within 1.5 mm (Figure 3). The horizontal distance between the tip of the nozzle and the top of the wick shall be 6.5 ± 1.5 mm as shown in figure 3. This adjustment shall be maintained for all positions of the burner along its pivot bar. The testing jig shall also be adjusted so that when the burner is in the setting position the axis of the nozzle is vertical.

d) Flame

With the burner in the setting position the igniting flame shall be adjusted to a height of 25.5 ± 1.5 mm. The flame height should be checked between tests and adjusted as necessary.

3.3.2.4. Procedure

The tests shall be carried out in still air. This can be conveniently achieved in a fume cupboard with the ventilator closed and the door open only by an amount sufficient to allow access to the apparatus. The tests shall be carried out at a temperature of 20 ± 2°C.

The sample of fluid shall be thoroughly mixed and the soaking bath filled to a depth of approximately 30 mm. A batch of at least five wicks shall immediately be immersed in the fluid in the soaking bath. The wick shall remain immersed for at least 10 minutes and until required for test. No further wicks shall be immersed in this fluid. Normally, a test can be completed with five wicks out under some circumstances as many as fifteen may be required. Should it be necessary to soak a further batch of wicks, the soaking bath shall be emptied and cleaned then refilled with fluid from the freshly mixed sample.

The reservoir shall be filled with fluid from the freshly mixed sample to a level 12.5 ± 1.5 mm below the reference marks on the clips. The fluid shall be maintained at this level throughout the test.

A soaked wick shall be taken from the soaking bath and mounted between the clips in the reservoir. The wick shall be handled carefully within 20 mm of each end using forceps. The top of the wick, as defined by the top of the uppermost warp thread, shall be in line with the reference marks on the clips and the tape shall be lightly tensioned so that its exposed edge lies in a straight line between the clips.

The wick shall be allowed to drain in this position for at least one minute and the igniting flame then applied, to the mid-point of the wick edge for the required exposure time. The time of persistence of flame on the wick after removal of the igniting flame shall be measured to the nearest 0.1 s.

If the flame on the wick reaches the clips this shall be reported together with the test result and no further tests shall be carried out on that wick edge.

If the burned portion of the wick does not extend more than 20 mm from the point of ignition two further tests may be carried out on that wick edge applying the igniting flame at positions 50 mm from the mid point of the wick edge. Should any residual flame on the wick traverse a previously burned section of the wick, or should the flame reach a supporting clip, that result shall be discarded. No test shall be made after the draining time exceeds five minutes.

The wick may then be inverted and again allowed to drain for one minute. Further tests may then be made following the above procedure. No test shall be made after the draining time in the inverted position exceeds five minutes.

Further tests may be carried out with a fresh wick following the procedure described above.

The exposure time shall be 2, 5, 10, 20 and 30 seconds, and six observations of the persistence of flame on the wick shall be obtained for each of these exposure times. If all the tests have not been completed within a period of two hours the reservoir shall be emptied and refilled with fluid from the freshly shaken sample.

3.3.2.5. Results

All six of the results for each of the five exposure times shall be reported. The averages of each set of six results shall be reported and the largest of these averages shall be taken as the mean persistence of flame.

3.3.2.6. Acceptance criteria

When a sample of fluid is tested in accordance with section 3.3.2.4. the mean persistence of flame shall be less than 60 seconds.

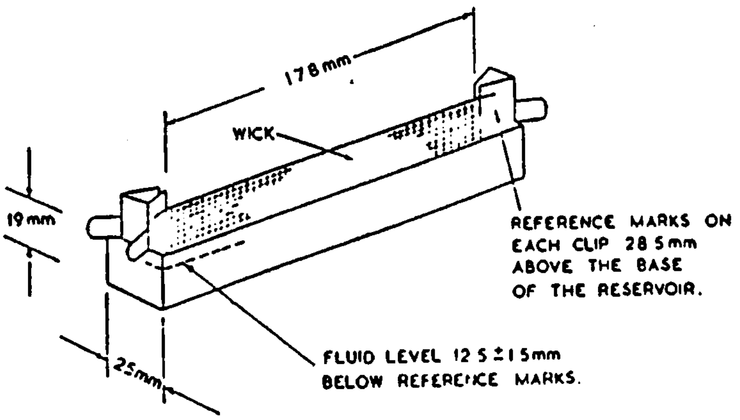


Fig. 1 Reservoir

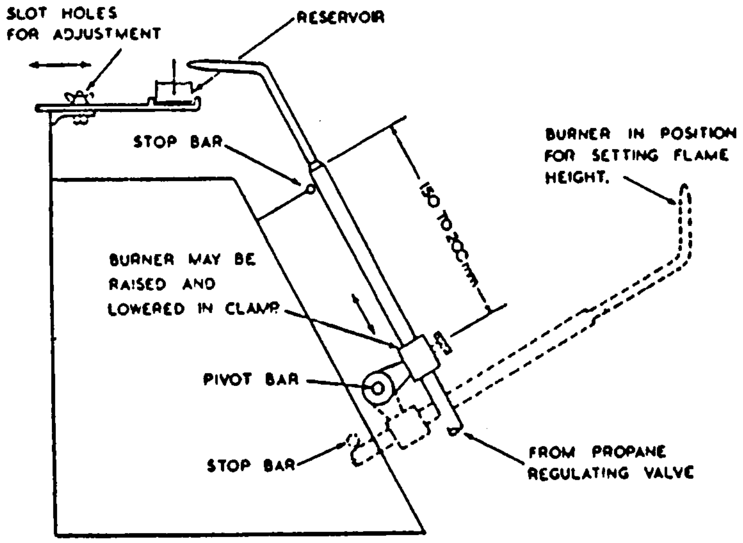


Fig. 2 Test rig

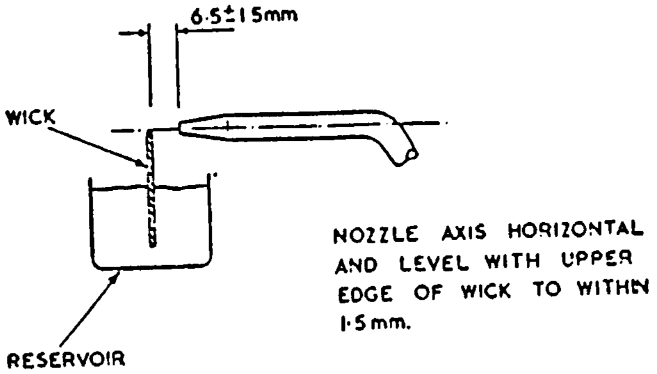


Fig. 3 Burner nozzle in igniting position

Figure 3 Relative positions of nozzle and wick

PART IV

HEALTH CRITERIA

4. HEALTH CRITERIA

4.1. TESTING CONDITIONS AND CRITERIA FOR ASSESSING THE HAZARD TO HEALTH

4.1.1. General

Fire-resistant hydraulic fluids should satisfy the criteria specified in this part of the report with respect to :

- acute toxicity, irritation of skin and mucous membranes, and toxicity of aerosols and thermal decomposition products.

The minimum procedures are described in sections 4.2 and 4.3.. However, whatever may be the results of this assessment, it cannot predict deleterious effects of allergic or other origin, in the long term or upon repeated exposure. Hence, approval/acceptance based on these testing procedures should only be provisional and any final approval/acceptance should be given only after a period of operational use. In this report there is no discussion of any environmental risks which could perhaps be associated with certain fire resistant fluids. This question is currently being considered.

4.1.2. Assessment of the hazard to health

Member countries shall make such arrangements as may be necessary to ensure that fluids satisfying the criteria set out below are only approved/accepted after:

review of the available toxicological data on all ingredients in the fluids or formed in the fluid by interaction of the components or by hydrolysis, oxidation or thermal decomposition;

examination of the results of all toxicological tests carried out on the fluid and its reaction and decomposition products when it has been found necessary to perform these because of inadequate data;

examination of the reports of the effects on health of human exposure of those who have been in contact with the fluid during its manufacture and testing.

The requirements of sections 4.2 or 4.3, whichever is appropriate, constitute the minimum which will normally be applied to any new fluid proposed for an application in mines. In certain countries and for certain fluids additional requirements may be proposed.

4.2. METHODS OF TEST FOR THE TOXICITY OF TYPES HFA, HFB AND HFC FLUIDS

4.2.1. General

The manufacturer of the fluid to be tested must supply the chemical composition in sufficient detail to enable an assessment of its likely toxicity to be made. In particular, in the case of fluids containing mineral oils, the content of aromatic constituents must be specified.

The nature and content of any additives and impurities must also be specified.

4.2.2. Verification of identity

The organic phase is separated by an appropriate method (extraction or centrifugation) and dried.

The IR spectrum of the organic phase is determined. This IR spectrum is the reference for the identity of the product.

In the case of type HFC fluids (aqueous solutions of glycols containing not less than 35% water) it is not possible to separate the organic phase. Instead of an IR spectrum, an HPLC chromatogram of the original fluid may be taken using a differential refractometer for the detection of the glycols. A parallel registered HPLC chromatogram with UV detection can monitor the pattern of additives.

Unless the fluids can be assessed as acceptable for use on the basis of present knowledge of the chemical and physical properties and the biological behaviour of components and impurities, the following tests shall be carried out at the concentration in which the fluid is normally used. For the purposes of tests, the diluent shall be distilled water.

4.2.3. Oral toxicity

Determination of oral toxicity (LD50, i.e. the dose which causes the death of 50% of the animals) is performed on Wistar SPF rats, weighing 150 to 200 g on an equal number of males and females. The LD50 is expressed in g/kg body weight of the animal.

The fluid (at the concentration in which it is normally used) is administrated to the rats by buccal probe. The observation period is 14 days. The number of dead animals for each dose is expressed as a percentage. The LD50 is determined by the usual methods of calculation.

The results are scored in accordance with the following scale:

$\mathrm{LD50} > 2\ \mathrm{g/kg}$ 0

$2\ \mathrm{g/kg} \geq \mathrm{LD50} > 0.2\ \mathrm{g/kg}$ 3

$0.2\ \mathrm{g/kg} \geq \mathrm{LD50} > 0.02\ \mathrm{g/kg}$ 5

$0.02\ \mathrm{g/kg} \geq \mathrm{LD50}$ 10

4.2.4. Irritant effect

Tests are done on the fluid at the concentration in which it is normally used.

'For types HFA and HFAS fluids, the skin test is additionally carried out with the undiluted emulsifying oil or fluid concentrate respectively.

4.2.4.1. Irritant effect

Skin irritation is examined on three male albino rabbits, each weighing about 2 kg, using a patch test which is carried out as follows:

A patch of skin, 80 x 80 mm on the animal's flank is clipped, care being taken not to injure the epidermis.

Next day the dressing is placed in position. This consists of a piece of cotton wool, 40 x 40 mm, soaked in 2.5 ml of the fluid, enclosed in a strip of inert material with a 20 x 20 mm, window demarcating the surface which comes into contact with the skin. The dressing is kept in position with a bandage.

After 24 hours, the dressing is removed and the state of the skin examined daily for the next week. The maximum effect on each animal is noted.

The results are scored as follows:

No reaction	0
Slight, barely visible erythema	1
Moderate erythema	2
Severe erythema	3
Erythema with slight oedema	4
Erythema with moderate oedema	5
Erythema with severe oedema	6
Limited necrosis	7
Widespread necrosis	8-9
Total necrosis of the surface	10
Death of one or more animals	10

The average of the maximum score obtained for each of the three animals is calculated. If one animal gets a score of 10 then the product is not accepted.

4.2.4.2. Irritant effect on the eye

The test is carried out on three male albino rabbits, but not those used for the skin test.

Using sterile equipment, 0.1 ml of the fluid to be tested is placed in the conjunctival sac of the right eye. The eye must be kept closed with the hand for about 5 s after administration.

The animals must be isolated for the duration of the tests.

The state of the right eye and its adnexae is compared with that of the left eye once a day for a week. The maximum effect on each animal is noted.

The results are scored as follows:

No reaction	0
Slight Lacrimation	1
Intense lacrimation	2
Conjunctiva injected	3
Bright red conjunctiva	4
Purulent exudate	5
Partial opacity of the cornea, according to intensity	6-9
Total cornea opacity and/or loss of sight	10

The average of the maximum score obtained for each of the three animals is calculated . If one animal is given a score of 10, then the product is not accepted.

4.2.5. Aerosol toxicity

The test is carried out on 6 male Wistar rats using the apparatus shown in figure 1. The details of the different parts are shown in the diagram. The fluid, at the concentration at which it is normally used is placed in an aerosol generator (Type HFA fluids: figure 2 and types HFB and HFC fluids: figure 3). The temperature of the generator is maintained at 20°C for type HFA fluids and at 70°C for types HFB and HFC. The temperature is regulated by means of a thermocouple (H) the hot joint of which is placed in the aspiration opening of the nozzle. The compressed air feed pressure of the aerosol generator is measured with a manometer (C). The generator should dispense from 20 to 30 ml of fluid an hour. Delivery of air should be approximately 1 m^3 an hour.

After leaving the generator the aerosol mist passes into a stainless steel U tube (l) which has a collector (J) at its lowest point, and then passes through flexible tubing (K) into an inhalation chamber (L) made of transparent plastic material, with a volume of approximately 140 l. The recommended dimensions for this chamber are : length 800 mm, width 350 mm. height 500 mm. The aerosol enters the chamber through a lateral opening (M) sited 120 mm from the bottom and leaves it by an opening (N) sited on the opposite wall at the same height. The front face of the chamber consists of a door to enable the animals to be placed in the chamber. This door has a port so that atmospheric monitoring samples can be taken. The rats are placed in two cages (P) the top and sides of which consist of bars or netting. The cages must be placed centrally in the chamber with the bottom of the cages being 70 mm above the floor. The concentration by weight of the aerosol in the inhalation chamber is determined by drawing a sample of the atmosphere from the immediate proximity of the cages through a filter (Poelman scaphander type, ref. 3205-51 or equivalent) which is weighed before and after sampling. The details of the method can be obtained from the Secretariat of the Safety and Health Commission for the Mining and Extractive Industries.

The whole installation must be airtight. A depression (approximately 1 mm water gauge) is maintained in the inhalation chamber by means of an aspiration device (Q) inserted at the exit. Pressure is measured by means of a tilted manometer (R). The temperature of the interior of the inhalation chamber is monitored with a thermometer (S).

Three male Wistar rats, each weighing approximately 200 g, are placed in each of the cages, where they are exposed to the aerosol for one hour.

After this exposure time the development of body weight is determined daily for a period of 14 days and compared with that of a control group of 6 untreated rats of the same weight and age; missing values being estimated by interpolation. The averages of the daily weight differences (x control - x test = mD) are calculated and scored as follows:

No reaction observed and average difference in weight of less than 5 g			0
5	mD	10	2
10	mD	15	3
15	mD	20	4
20	mD		5

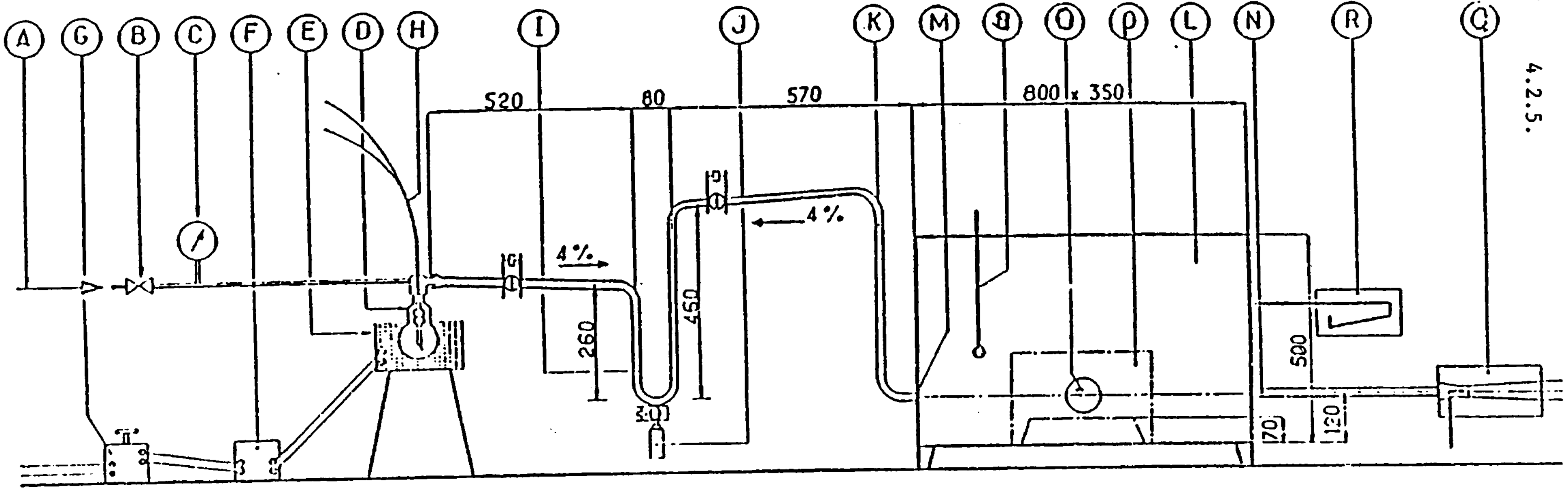
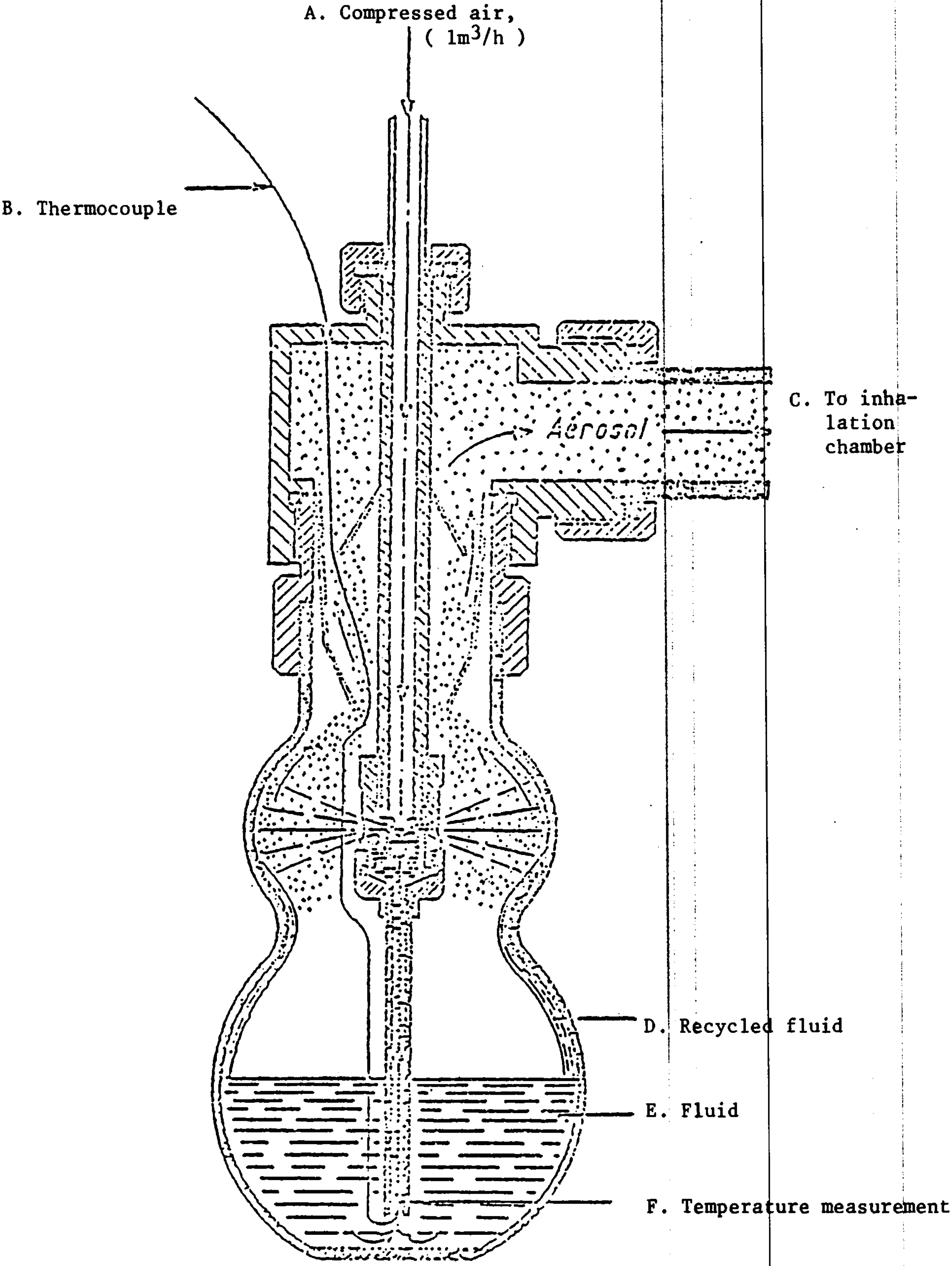


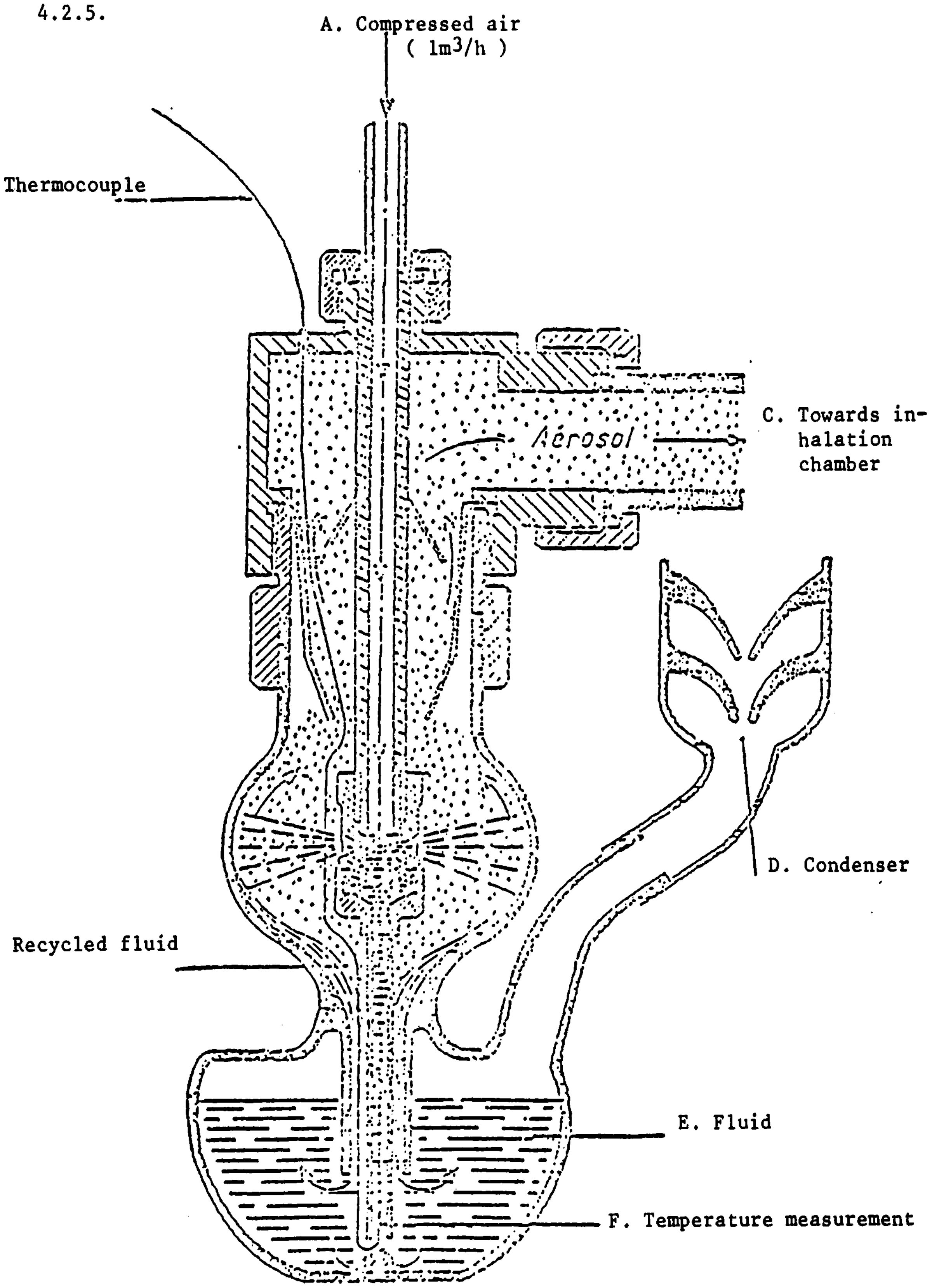
Figure 1 Apparatus for the aerosol toxicity test - overall design

A. Compressed air feed
B. Pressure-reducing valve
C. Manometer for regulating compressed air pressure
D. Cerchar - Type A1 aerosol generator with interchangeable nozzles for types A, C and D fluids
E. Electric flask heater
F. Temperature regulator
G. Potentiometric recorder
H. Fer-constantan thermocouple
I. Condenser, int. diam. 15 mm, connected to aerosol generator by spherical Rotulex 29/15 valve
J. Collector, vol. 25 ml, connected to condenser by spherical valve
K. Flexible connecting tubing, diam. 15 mm.
L. Inhalation chamber, 140 litres, Internal dimensions : L - 800 mm, W - 350 mm, H - 500 mm
M. Lateral opening for aerosol entry, diam. 15 mm
N. Lateral opening for aerosol exit, diam. 15 mm
O. Sampling opening
P. Animal cage. Dimensions : L - 320 mm, W - 250 mm, Height with feet - 230 mm
Q. Aspirator
R. Inclined monometer
S. Thermometer with an accuracy of 0.50°C



SCALE 1

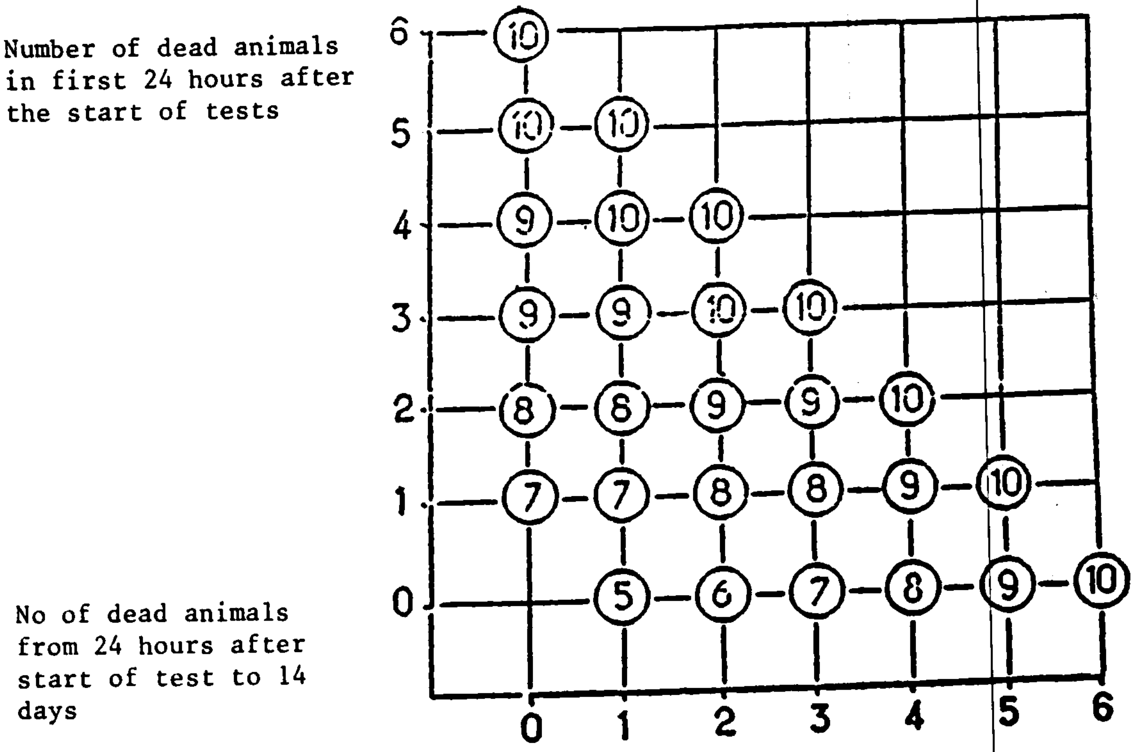
Figure 2 Aerosol generator for type HFA (20°C) and type HFD (50°C) fluids



SCALE 1

Figure 3 Aerosol generator for types HFB and HFC (70°C) and type HFD (150°C) fluids

In the event of the death of one or more of the animals, the toxicity is assessed according to the following diagram:



At the end of the 14 day observation period the animals are killed and the organs examined macroscopically.

4.2.6. Thermal decomposition products

The test is carried out only on types HFB and HFC fluids diluted to the concentration at which they are normally used, projected on to a plate heated to 700°C. The apparatus is shown in figures 4 and 5. The details of the different parts are shown in the diagram.

Thermal decomposition of the fluid to be tested is carried out in a closed metal chamber (A). The fluid is placed in a graduated flask connected to a variable flow high pressure membrane pump (B). The pump supplies the fluid to a Diesel injector (C) placed at one end of the chamber (Cone angle of the injector 45°) at a pressure of 100 bar and flow of 1 ml/min. The atomised liquid is projected onto a metal plate (D), 60 mm in diameter, placed 115 mm from the opening of the nozzle. The plate is heated electrically by means of a device consisting of four heating units buried in a metal block (E). The temperature is regulated by means of a thermocouple (F), the hot joint of which is 1 mm from the surface of the plate, and is kept constant throughout the test to ± 10°C.

The resulting aerosol is carried in the primary air flow of 10 l/min which enters the chamber by two lateral openings (G). A vertical partition (H), forming a baffle between the injector and entry of the air, prevents direct aspiration of the injected product before it reaches the heating plate. The chamber is tilted downwards and has a collector (I) at its lowest point.

At the exit from the chamber, the aerosol passes through a condensation apparatus consisting of a stainless steel U tube (J) which has a collector at its lowest point (K).

At the end of the U-tube, the aerosol is diluted by a secondary air flow of 20 l/min. This air is injected in the current direction at an angle of 30° by means of a Y-shaped tube (L). The primary and secondary air flow is supplied by a compressed air source and the flows are measured by means of rotameters (M) and (N). The tubing slopes 4% to ensure that the condensed fluid flows towards the collector.

The diluted aerosol is passed through flexible tubing (O) into the inhalation chamber (P) of transparent plastic material, with a volume of approximately 140 litres. The dimensions recommended for this chamber are : length 800 mm, width 350 mm, height 500 mm. The aerosol enters the chamber through a side opening (Q) 120 mm from the bottom and leaves it by an opening (R) on the opposite side at the same level. The front of the chamber consists of a door, so that the animals can be placed inside. This door has a port (S) so that atmospheric monitoring samples can be taken. The rats are placed in two cages (T), the top and sides of which consist of bars or netting. The cages must be placed centrally in the chamber with the bottom of the cages being 70 mm above the floor. The concentration of the aerosol in the inhalation chamber is determined by drawing a sample of the atmosphere in the immediate proximity of the cages through a filter (Poelman scaphander type, ref. 3205-51 or equivalent) which is weighted before and after removal. The details of the method can be obtained from the Secretariat of the MSHC.

The whole installation must be airtight. A slight depression (1 mm water gauge approximately) is maintained in the inhalation chamber by means of an aspiration device (U) inserted at the exit. Pressure is measured by means of a tilted manometer (V). The temperature inside the inhalation chamber is monitored by means of a thermometer (W).

The procedure to be adopted and the scale of values are described in section 4.2.5.

4.2.7. Acceptance criteria

Each product tested is given a rating from 0 to 180 determined by adding the scores obtained in each of the above tests, and weighted by coefficients as follows:

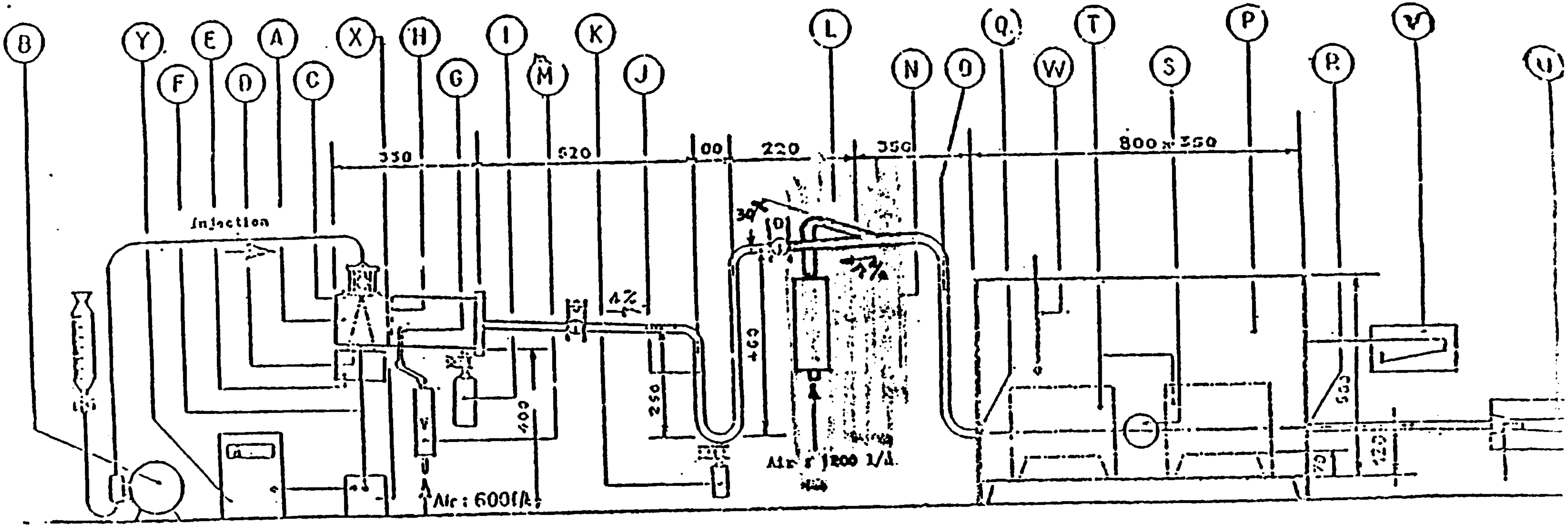
		Type HFA	Types HFB and HFC
Oral toxicity	LD50	2	2
Irritant effect on the skin -			
made-up fluid		5	5
emulsifying oil or fluid concentrate		2	-
Irritant effect on the eye		5	5
Aerosol toxicity		4	4
Thermal decomposition toxicity		-	2

With the exception of the thermal decomposition test, a fluid which scores 10 in any of the tests before weighting or a total score after weighting of 40 should be rejected. However, in the test reports of fluids obtaining high scores in the thermal decomposition test, note should be made of the fact that this fluid may give rise to dangerous fumes on thermal decomposition.

Figure 4

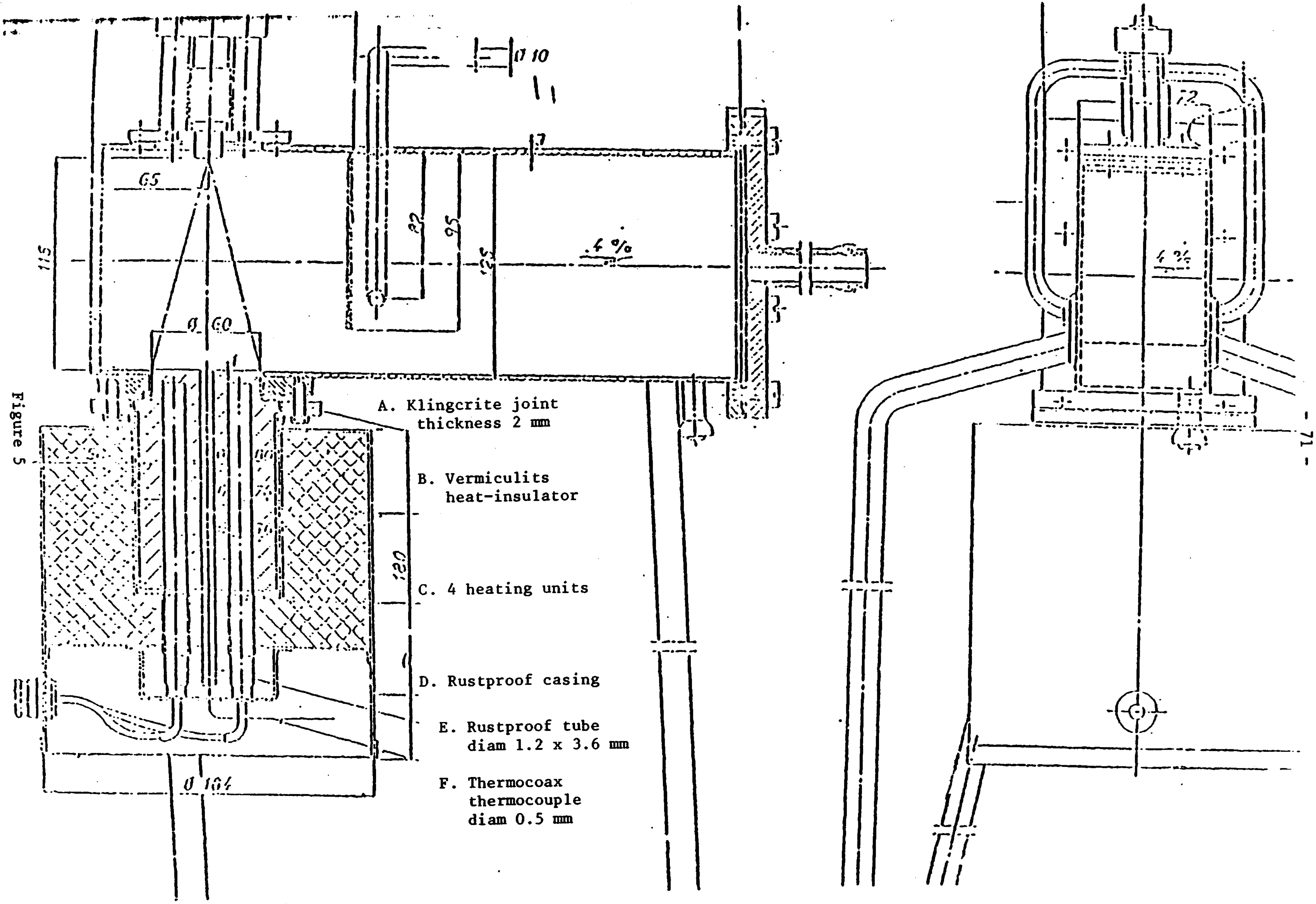
- APPARATUS FOR THERMAL DECOMPOSITION TEST -

OVERALL DESIGN



PART IV 4.2.5. Figure 4. LEGEND

A	Metal chamber (internal dimensions):L 350 mm, W 66 mm, H 115 mm
B	Variable flow membrane pump, ORLITA type DMP AE 10.4
C	Bosch injector (0434, 200, 024 - DN 30 S3)
D	Stainless steel heated plate.
E	Electrical heating for plate. 4 heating units, diam. 12.7 L 165.1 LC 117.3, P 400 W, Load 9W/cm2, total power, 1,600 W. Exit L 250.
F	Thermocouple (Thermocoax Chromel-Alumel, diam. 0.5 mm)
G	Lateral openings for air entry, int. diam. 10 mm.
H	Partition : H 95 mm, W 66 mm.
I	Collector vol 50 ml, connected to chamber by sperical Rotulex 19/9 valve.
J	Condenser, int. diam 15 mm, connected to chamber exit tubing by sperical Rotulex 29/15 valve.
K	Collector, vol. 25 ml, connected to condenser by sperical valve.
L	Y-shaped diluter, int. diam of air entry tube 8 mm, angle 30°.
M-N	Flowmeters (Rotameters).
O	Flexible connecting tubing, int. diam. 15 mm.
P	Inhalation chamber, 140 l. Internal dimensions ; L 800 mm, W 350 mm, H 500 mm.
Q	Lateral opening for aerosol entry, diam. 15 mm.
R	Lateral opening for aerosol exit diam. 15 mm.
S	Sampling opening.
T	Cage for animals. Dimensions L 320 mm, W 250 mm, Height with feet 230 mm.
U	Aspiration apparatus.
V	Inclined manometer.
W	Thermometer, 0 - 50°C.
X	Temperature regulator.
Y	Potentiometer recorder.



A. Klingcrite joint
thickness 2 mm

B. Vermiculits
heat-insulator

C. 4 heating units

D. Rustproof casing

E. Rustproof tube
diam 1.2 x 3.6 mm

F. Thermocoax
thermocouple
diam 0.5 mm

Figure 5

4.3. METHODS OF TEST FOR THE TOXICITY OF TYPE HFD FLUIDS

4.3.1. Chemical composition

The manufacturer of the fluid to be tested must supply the chemical composition in sufficient detail to enable an assessment of its likely toxicity to be made. The nature and amount of any additives and impurities must also be specified.

4.3.2. Verification of identity

An IR spectrum is prepared. This is used as a reference for the identity of the product.

Note : The following sections relate to tests on all type HFD fluids. However, in the case of phosphate esters, the tests specified in section 4.4. will be carried out first before the additional tests in sections 4.3.3. to 4.3.6. are carried out.

4.3.3. Oral toxicity

Determination of the oral toxicity (LD50, i.e. the dose which causes the death of 50% of the animals) is performed on SPF Wistar rats, weighing 150 to 200 g on an equal number of males and females. The LD50 is expressed in g/kg body weight of the animal.

The fluid is administrated to the rats by buccal probe. The observation period is 14 days.

The number of dead animals for each dose is expressed as a percentage. The LD50 is determined by the usual methods of calculation.

The results are scored according to the following scale:

LD50	$>$	2 g/kg			0
2 g/kg	$\geq$	LD50	$>$	0.2 g/kg	3
0.2 g/kg	$\geq$	LD50	$>$	0.02 g/kg	5
0.02 g/kg	$\geq$	LD50			10

4.3.4. Irritant effect

4.3.4.1. Irritant effect on the skin

Skin irritation is examined on three male albino rabbits each weighing about 2 kg, using a patch test which is carried out as follows:

A patch of skin 80 x 80 mm, on the animal's flank is clipped, care being taken not to injure the epidermis.

Next day, the dressing is placed in position. It consists of a piece of cotton wool, 40 x 40 mm, soaked in 2.5 ml of the fluid, enclosed in a strip of inert material with a 20 x 20 mm window which demarcates the surface of contact with the skin. The dressing is kept in place with a bandage.

Twenty-four hours later, the dressing is removed and the state of the skin examined daily for the next week. The maximum effect on each animal is noted.

The results are scored as follows :

No reaction	0
Slight, barely visible erythema	1
Moderate erythema	2
Severe erythema	3
Erythema with slight oedema	4
Erythema with moderate oedema	5
Erythema with severe oedema	6
Limited necrosis	7
Extensive necrosis	8-9
Total necrosis of the surface	10
Death of one or more animals	10

The average of the maximum score obtained for each of the three animals is calculated. If one animal is given a score of 10, then the product is not accepted.

4.3.4.2. Irritant effect on the eye

The test is carried out on three male albino rabbits, but not those used for the skin test.

Using sterile equipment, 0.1 ml of the test fluid is placed in the conjunctival sac of the right eye. The eye must be kept closed with the hand for about 5 s after administration.

The animals must be isolated for the duration of the tests.

The state of the right eye and its adnexae is compared to that of the left eye once a day for a week. The maximum effect on each animal is noted.

The results are scored as follows :

No reaction	0
Slight lacrimation	1
Intense lacrimation	2
Conjunctiva injected	3
Bright red exudate	4
Purulent exudate	5
Partial opacity of the cornea according to intensity.	6-9
Total opacity of the cornea and/or loss of vision ...	10

The averages of the maximum score obtained for each of the three animals is calculated. If one animal is given a score of 10, then the product is not accepted.

4.3.5. Aerosol toxicity

4.3.5.1. Cold aerosol

The test is carried out on six male Wistar rats, using the apparatus shown in figure 1. The details of the different parts are shown in the diagram.

The fluid, which must not be diluted, is placed in an aerosol generator (figure 2). The temperature of the generator is maintained at 50°C. The temperature is regulated by means of a thermocouple (H) the hot joint of which is placed in the aspiration opening of the nozzle. The compressed air feed pressure for the aerosol generator is measured by a manometer (C). The generator should dispense from 10 to 15 ml fluid an hour. The air flow should be approximately 1 m3 an hour.

On leaving the generator, the aerosol mist passes into a stainless steel U tube (I) which has a collector (J) at its lowest point, and then passed through flexible tubing (K) to an inhalation chamber made of transparent plastic material (L), with a volume of approximately 140 litres. The dimensions recommended for this chamber are : length 800 mm, width 350 mm, height 500 mm. The aerosol enters the chamber by a side opening (M) 120 mm from the bottom and leaves by an opening on the opposite side at the same level. The front of the chamber consists of a door so that animals can be placed inside. This door has a port (O) so that atmospheric monitoring samples can be taken. The rats are placed in two cages (P) the top and sides of which consist of bars or netting. The cages must be placed centrally in the chamber, the bottom of the cages being 70 mm above the floor. The concentration of the aerosol in the inhalation chamber is determined by drawing a sample of the atmosphere from the immediate proximity of the cages through a filter (Poelman scaphander type, ref. 3205-51 or equivalent) which is weighed before and after sampling. The details of the method can be obtained from the Secretariat of the MSHC.

The whole installation must be airtight. A depression of approximately 1 mm water gauge is maintained in the inhalation chamber by means of an aspiration device at the exit. Pressure is measured by means of a tilted manometer (R). The temperature inside the inhalation chamber is monitored with a thermometer (S).

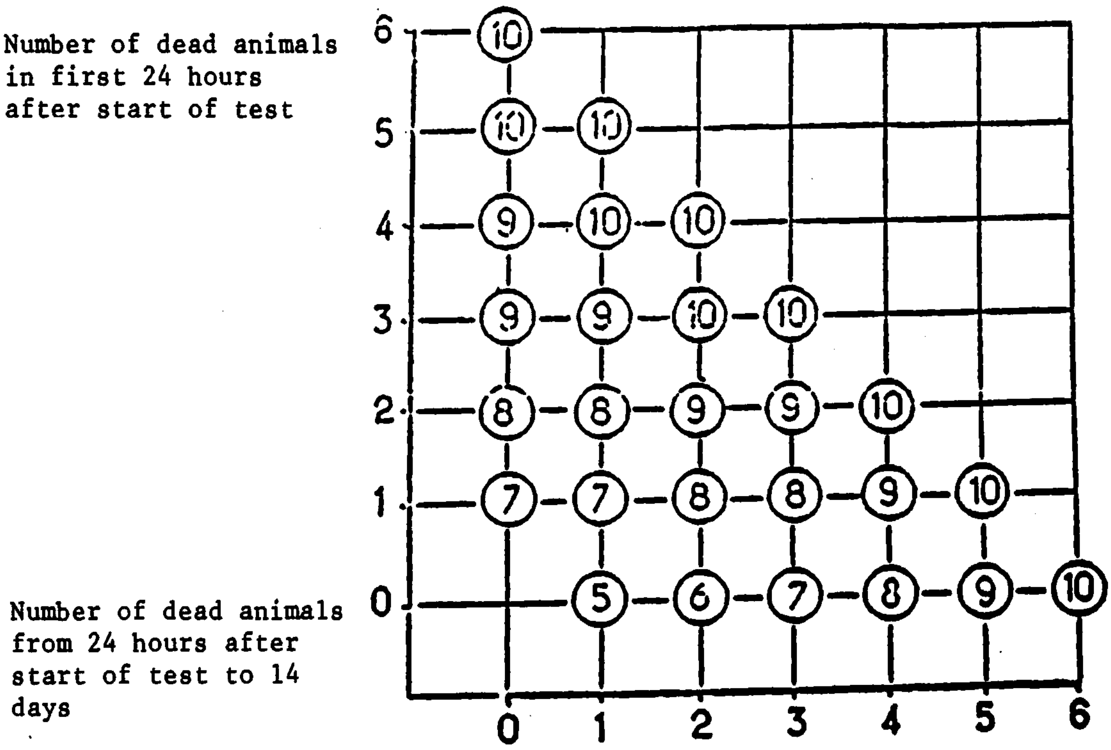
Three male Wistar rats each weighing approximately 200 g are placed in each of the cages, where they are exposed to the aerosol for one hour.

Six other rats of the same weight and age are kept as a control sample.

After this exposure, their development of body weight in determined daily for a period of 14 days and compared with that of a control group of 6 untreated rats of the same weight and age; missing values being estimated by interpolation. The averages of the daily weight differences ($\bar{x}$ control - $\bar{x}$ test = mD) is calculated and scored as follows:

No reaction observed and average difference in weight of less than 5 g			0
5	mD	10	2
10	mD	15	3
15	mD	20	4
20	mD		5

In the event of the death of one or more of the animals, the toxicity is assessed according to the following diagram:



At the end of the 14 day observation period, the mode of toxic action will be examined by means of macroscopic examination.

4.3.5.2. Hot aerosol

The test described above is repeated on a fresh group of animals with the temperature of the generator fluid at 150°C, using the aerosol generator shown in figure 3. The results are assessed in accordance with the same scale as for cold aerosols.

4.3.6. Thermal decomposition products

The test is carried out with the fluid sprayed on to a plate heated to 700°C. The apparatus is shown in figures 4 and 5. The details of the different parts are shown in the diagram.

Thermal decomposition of the test fluid occurs in a closed metal chamber (A). The fluid is placed in a graduated flask connected to a variable flow high pressure membrane pump (B) which sends the fluid to a Diesel injector having a cone angle of 45° placed at one end of the chamber. At a pressure of 100 bar the flow is 1 ml/min. The atomised liquid is projected onto a metal plate (D) 6 cm in diameter, placed 115 mm from the opening of the nozzle. The plate is heated electrically by means of an apparatus consisting of four heating units buried in a metal block (E). The temperature is regulated by means of a thermocouple (F), the hot joint of which is 1 mm from the surface of the plate, and is kept constant for the duration of the test, to + 10°C.

The resulting aerosol is carried in the primary air flow of 10 l/min which enters the chamber by two side openings (G). A vertical partition (H), forming a fabble between the injector and the air flow, prevents direct aspiration of the injected product before it reaches the heating plate. The chamber is tilted downwards and has a collector (I) at its lowest point.

When it leaves the chamber, the aerosol passes through a condensation apparatus consisting of a stainless steel U tube (J) which has a collector (K) at its low point.

At the end of the U-tube, the aerosol is diluted by a secondary air flow of 20 l/min. To ensure a good mixture, this air is injected in a counter current direction at an angle of 30° by means of a Y-shaped tube (L). The primary and secondary air is supplied by a compressed air source and the flown are measured by means of a rotameters (M) and (N). The tubing slopes 4% to ensure that the condensed fluid flows towards the collector.

The diluted aerosol is passed through flexible tubing (O) into the inhalation chamber (P) made of transparent plastic material, with a volume of approximately 140 litres. The dimensions recommended for this chamber are : length 800 mm, width 350 mm, height 500 mm. The aerosol enters the chamber through a side opening (Q) 120 mm from the bottom and leaves by an opening (R) on the opposite side at the same height. The front of the chamber consists of a door so that the animals can be placed inside. This door has an opening (S) so that atmospheric monitoring samples can be taken. The rats are placed in two cases (T) the top and sides of which consist of bars or netting. The cages must be placed centrally in the chamber the bottom of the cages being 70 mm above the floor. The concentration of the aerosol in the inhalation chamber is determined by drawing a sample of the atmosphere in the proximity of the cages through a filter (Poelman scaphander type, ref. 3205-51 or equivalent) which is weighed before and after sampling. The details of the method can be obtained from the Secretariat of the MSHC.

The whole installation must be airtight. A slight depression (approximately 1 mm water gauge) is maintained in the inhalation chamber by means of an aspiration apparatus (U) inserted at the exit. Pressure is measured by means of an inclined manometer (V). The temperature inside the inhalation chamber is monitored by means of a thermometer (W).

The procedure to be adopted and the scale of values are described in section 4.3.5.1.

4.3.7. Acceptance criteria

Each product tested is given a rating between 0 and 180, determined by adding the scores obtained in each of the above tests, after weighting with the following coefficients :

Oral toxicity	LD50	2
Irritant effect	skin	5
	eye	5
Aerosol toxicity	cold 50°C	2
	hot 150°C	2
Thermal decomposition toxicity	700°C	2

With the exception of the thermal decomposition test, a fluid which scores 10 before weighting in any test or a score of 40 after weighting will be rejected. However, in the test reports of fluids obtaining high scores in the thermal decomposition test, note should be made of the fact that this fluid may give rise to dangerous fumes on thermal decomposition.

4.4 METHOD OF TEST FOR NEUROTOXIC EFFECTS OF PHOSPHORIC ESTERS

If the type HFD fluid is a mixture of phosphoric esters and other components (e.g. PCB's) the test has to be carried out with the phosphoric esters only and the manufacturer of the fluid has to submit a sample of the ester.

The tests are carried out on hens weighting approximately 2 kg at least 10 months old. The hens are housed so that they can move about freely. For one week before the tests the hens are observed so that any which show anomalies of gait can be rejected. Sixteen hours before the start of the test the hens' normal diet is withhold, but water is given freely.

The test fluid, which is undiluted, is administered by stomach tube at a dose of 5 g per kg body weight per day to 6 hens for 5 consecutive days. In this way each animal receives a total dose of 25 g/kg body weight.

The observation period is 21 days from the end of dosing during which defects of gait are observed. The first symptoms of poisoning appear in the form of motor instability. Walking with the feet apart is typical of advancing paralysis ; finally, the hens can no longer stand up and squat on the metatarsal bones. There is a tendency for the animals to peck at each other and for cannibalism.

When symptoms of poisoning as severe as that described above appear in even one hen, the fluid is considered to be neurotoxic.

To provide a positive test of neurotoxic reaction a control group of 2 hens is given a single dose of 1 g/kg triorthocresylphosphate (TOCP). This dose usually causes severe paralysis and related histological findings in all the animals treated within 14 to 21 days. Together with the two groups of treated hens (test fluid, TOCP), 6 hens of the same weight and age are also kept under the same conditions to act as a control sample.

If the fluid is shown to be neurotoxic in the above tests there is no point in proceeding to the other tests prescribed for this type of fluid.

PART V

'PRESCRIBED'

TECHNOLOGICAL TESTS

5. 'PRESCRIBED' TECHNOLOGICAL TESTS

The test methods described in this part of the report relate to those technological properties of a fluid which, together with the tests for resistance to flame in Part III and the tests for the hazard to health in Part IV, form the essential basis of an acceptance or approval of the fluid. Any quality control tests should be selected as appropriate from those in this and the following Part of this report (see section 2.4.1).

In the case of each prescribed technological test the criteria to be achieved are specified.

Where alternative test methods are described for the same property, it is for the approving/accepting or testing authority to decide which of the methods of test is to be used.

5.1. METHODS OF TEST FOR KINEMATIC VISCOSITY

Note : Two methods of test are described, one using capillary type viscometers and the other for use with drop-ball viscometers. It is for the accepting or approving authority to decide which method is to be used.

The kinematic viscosity should be measured at the temperatures given in the Table below :

Table - Temperatures at which the kinematic viscosity should be measured

Fluid type	Temperature in °C					
Emulsifying oil for type HFA fluids			20			
HFA (other than type HFA 1)			20	40	50	
HFB			20	40	50	
HFC	-20	0	20	40	50	
HFD		0	20	40	50	100

5.1.1. METHOD OF TEST FOR KINEMATIC VISCOSITY USING CAPILLARY TYPE VISCOMETERS

Note : This method is based on the test procedure given in International Standard ISO 3104. Similar test procedures are also given in the following Standards : BS 4708, AFNOR NFT 60-100, DIN 51562, ASTM D 445-74 and IP 71/75.

5.1.1.1. Scope

The determination of the kinematic viscosity of all types of hydraulic fluids using a glass capillary type viscometer.

5.1.1.2. Principle

The time is measured, in seconds, for a fixed volume of fluid to flow under gravity through the capillary of a calibrated viscometer under a reproducible head and at a closely controlled temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer.

(Kinematic viscosity is the ratio between the dynamic viscosity and the density of a fluid).

The kinematic viscosity coefficient has the dimension $L^2\ T^{-1}$, where L is a length, T is a time. In the SI system of units, the unit of kinematic viscosity is the m^2/s, but for practical use a submultiple is more convenient. The centistoke (cSt) is $10^{-6}\ m^2/s$ and is customarily used.

5.1.1.3. Apparatus

a) A viscometer of the glass capillary type. The viscometer shall be chosen from the types listed in table 2.

For transparent Newtonian fluids of types HFA, HFC or HFD, any of the viscometers listed in table 2 may be used.

For opaque Newtonian fluids of types HFA, HFC or HFD, any of the reverse flow types of viscometer may be used.

For all HFB fluids and other non-Newtonian fluids, a BS/IP reverse flow U-tube viscometer shall be used.

In all cases the size of viscometer shall be chosen to give the shortest flow time consistent with the specified minimum.

b) Viscometer holders to enable the viscometer to be suspended in a similar position as when calibrated. The proper alignment of vertical parts may be confirmed by use of a plumbline.

c) Viscometer thermostat and bath. Any transparent liquid bath may be used provided that it is of sufficient depth that at no time during the measurement will any portion of the sample of fluid in the viscometer be less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath.

d) A temperature control such that for the range of 15°C to 100°C, the temperature of the bath medium does not vary by more than 0.01°C over the length of the viscometer, or between the position of each viscometer, or at the location of the thermometer.

e) Temperature measuring device. Standardised liquid-in-glass thermometers (see table 3) having an accuracy after correction of 0.02°C can be used, or any other thermometric device of equal accuracy.

f) Timing device. Any timing device may be used provided that the readings can be taken with a discrimination of 0.2 s or better, and that it has an accuracy within 0.07% when tested over intervals of 15 min.

Electrical timing devices may be used if the current frequency is controlled to an accuracy of 0.05% or better. AC currents, as provided by some public power systems, are intermittently rather than continuously controlled. When used to activate electrical timing devices, such control can cause large errors in viscosity flow measurements.

5.1.1.4. Sampling

A sample of not less than 100 ml shall be supplied for test. Care shall be taken to ensure that this sample is representative of the bulk.

5.1.1.5. Calibration

Viscometers :

Only calibrated viscometers with calibration constants measured and quoted to the nearest 0.1% of their value should be used.

Note : Procedures for the calibration of viscometers are given in ISO 3105 - Glass capillary kinematic viscometers - Specification and operating instructions.

The calibration constant C is dependent upon the gravitational acceleration at the place of calibration and this must, therefore, be supplied by the calibration laboratory together with the instrument constant. Where the acceleration of gravity, g, in the two locations differs by more than 0.1%, correct the calibration constant as follows :

$$C_2 = \frac{g_2}{g_1} \times C_1$$

where the subscripts 1 and 2 indicate respectively the calibration laboratory and the testing laboratory.

Thermometers

Liquid-in-glass thermometers shall be checked to the nearest 0.01°C by direct comparison with a suitable standardised thermometer. Kinematic viscosity test thermometers shall be calibrated at total immersion, which means immersion to the top of the mercury column, with the stem and expansion chamber at the top of the thermometer exposed to room temperature; the expansion bulb should not be immersed. It is essential that the ice point of calibrated thermometers be determined periodically and that the certified correction be adjusted to conform to any change in the ice point.

Timers

Standard time signals are available in some countries and may be used for checking the accuracy of timing devices.

Viscosity standards (1)

These may be used as confirmatory checks on the procedure in the laboratory. If the measured kinematic viscosity does not agree within ± 0.35% of the certified value, each step in the procedure should be rechecked, including thermometer and viscometer calibration, to locate the source of error.

Note (1) : Viscosity oil standards having the approximate viscosities shown in table 4 are available in certain countries from National laboratories or other authorised sources. Certified kinematic viscosity values are compared by annual cooperative tests by a number of laboratories.

5.1.1.6. Procedure

The specific details of operation vary for the different types of viscometers listed in table 2. In all cases, however, proceed in accordance with the following :

Maintain the bath at the test temperature within the limits given in section 5.1.1.3. Apply the necessary corrections, if any, to all thermometer readings. Ascertain that the thermometer has been checked recently ; a new thermometer may require checking every week.

Select a clean, dry, calibrated viscometer having a range covering the estimated viscosity (i.e. a wide capillary for a very viscous liquid and a narrower capillary for a more fluid liquid). The flow time should not be less than 200 s. When the temperature of the test is below the dew point, fit loosely packed drying tubes onto the open ends of the viscometer to prevent water condensation. Drying tubes must fit the design of the viscometer and not restrict the flow of the sample under test by pressures created in the instrument.

Viscometers used for fluids which are difficult to remove by the use of a cleaning agent, shall be reserved for the exclusive use of those fluids except when calibrating. Such viscometers shall be subjected to calibration checks at frequent intervals.

Charge the viscometer in the manner dictated by the design of the instrument, this operation being in conformity with that employed when the instrument was calibrated.

Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Because this time will vary for the different instruments and for different temperatures, establish a safe temperature equilibrium time by trial (30 min. should be sufficient). Where the design of the viscometer requires it, adjust the volume of the test sample after the sample has reached temperature equilibrium. One bath is often used to accomodate several viscometers. Never add or withdraw a viscometer while any other viscometer is in use for measuring flow time.

Use suction (if the sample contains no volatile constituents) or pressure to adjust the head level of the test sample to a position in the capillary arm of the instrument about 5 mm ahead of the first timing mark. With the sample flowing freely, measure in seconds, to within 0.2 s (see section 5.1.1.3), the time taken for the meniscus to pass from the first timing mark to the second. If this flow time is less than the specified minimum, select a viscometer with a capillary of smaller diameter and repeat the operation.

For modified Ostwald and suspended-level types, repeat the procedure described above to make a second measurement of flow time. The two measurement of flow time. The two measurements should agree within 0.2%.

For reverse flow types, duplicate determinations must be made ; flow times should agree within 0.35%.

Cleaning of viscometer

Between successive determinations, clean the viscometer thoroughly by several rinsings with an appropriate solvent miscible with the sample, followed by a completely volatile solvent. Dry the tube by passing a slow stream of filtered dry air through the viscometer for 2 min. or until the last trace of solvent is removed.

Periodically clean the instrument with chromic acid to remove organic deposits, rinse thoroughly with distilled water and acetone, and dry with clean dry air. Inorganic deposits may be removed by hydrochloric acid treatment before the use of cleaning acid, particularly if barium salts are suspected.

5.1.1.7. Results

Calculate the kinematic viscosity v from the measured flow time t and the instrument constant C by means of the equation :

$$v = C.t$$

where :

v is the kinematic viscosity, in centistokes ;
C is the calibration constant of the viscometer, in centistokes per second ;
t is the flow time, in seconds.

The report shall include the following :

a) the results of the two determinations of kinematic viscosity rounded to the nearest 0.01% of their value ;

b) the average of the two results for kinematic viscosity ;

c) the temperature of test ;

d) the type and size of the viscometer used for the test.

5.1.1.8. Acceptance criteria

The kinematic viscosity of emulsifying concentrates for type HFA fluids shall not exceed 350 cSt at 20°C. (This requirement is imposed to ensure that the emulsifying concentrate can be satisfactorily mixed by an injector).

The manufacturer shall state the kinematic viscosity in cSt for types HFA (other than type HFA$_1$), HFB, HFC and HFD fluids at the temperatures specified in table 1. When such fluids are tested at a temperature of 40°C, the average value of all measurements of viscosity shall lie within ± 10% of the stated value for that temperature.

The manufacturer's stated value for viscosity should, where practicable, lie within one of the viscosity ranges which are permitted by the viscosity grading system of ISO 3448 i.e. within ± 10% of the viscosity grade designations specified in section 2.2.2.2. Fluids may be exempted from this requirement by agreement between the user and the manufacturer. Exemption may be granted where it is impossible or impracticable to manufacture a fluid to a predetermined level of viscosity.

Table 2. Viscometer types

Viscometer identification	Kinematic viscosity range [1] cSt
A. Modified Ostwald types for transparent liquids :	
1 Cannon-Fenske routine[2]	0.5 to 20 000
2 Zeitfuchs	0.6 to 3 000
3 BS/U-tube[2]	0.9 to 10 000
4 BS/U/M miniature	0.2 to 100
5 SIL[2]	0.6 to 10 000
6 Cannon-Manning semi-micro	0.4 to 20 000
7 Pinkevitch[2]	0.6 to 17 000
B. Suspended level types for transparent liquids :	
1 BS/IP/SL[2]	3.5 to 100 000
2 BS/IP/SL(S)[2]	1.05 to 10 000
3 BS/IP/MSL	0.6 to 3 000
4 Ubbelohde[2]	0.3 to 100 000
5 FitzSimons	0.6 to 1 200
6 Atlantic[2]	0.75 to 5 000
7 Cannon-Ubbelohde, Cannon-Ubbelohde dilution[2]	0.5 to 100 000
8 Cannon-Ubbelohde semi-micro	0.4 to 20 000
C. Reverse-flow types for transparent and opaque liquids :	
1 Cannon-Fenske opaque	0.4 to 20 000
2 Zeitfuchs cross-arm	0.6 to 100 000
3 BS/IP/RF U-tube reverse flow	0.6 to 300 000
4 Lantz Zeitfuchs	60 to 100 000

1) Each range quoted requires a series of viscometers. To avoid the necessity of making a kinematic energy correction, these viscometers are designed for a flow time in excess of 200s except where noted.

2) In each of these series, the minimum flow time for the viscometers with lowest constants exceeds 200s.

NOTE:

Specifications and operating instructions for all these viscometers have been assembled in ISO 3105.

Table 3 - Kinematic viscosity test thermometers

Thermometer number		For tests at °C	Subdivisions °C
ASTM	IP		
		- 20	0.05
	33C	0	0.05
44C	29C	20 and 21.1	0.05
120C		40	0.05
46C	66C	50	0.05
	32C	98.9 and 100	0.05
121C		100	0.05

(1) The essential requirements for these thermometers are given in ASTM specification E1, for ASTM thermometers, and in the specification for IP standard thermometers.

Table 4 - Typical viscosity oil standards (1)

Viscosity standard designation	Approximate kinematic viscosity, mm^2/s. at								
	-53.89°C	-40°C	20°C	25°C	37.78°C	40°C	50°C	98.89°C	100°C
3	300	80	4.6	4.0	3.0	2.9	-	1.2	1.2
6	-	-	11	8.9	6.0	5.7	-	1.8	1.8
20	-	-	44	34	20	18	-	4.0	3.9
60	-	-	170	120	60	54	-	7.4	7.2
200	-	-	640	450	200	180	-	17	17

(1) The actual values for these standards are established and reaffirmed annually by co-operative tests.

5.1.2. METHOD OF TEST FOR KINEMATIC VISCOSITY USING DROP-BALL VISCOMETERS

Note : This method is based on the specification and method of use of the Höppler drop-ball viscometer ,to DIN 53 015. A similar procedure can also be adopted for the IFC viscometer which, although to a different design, operates on the same principle.

5.1.2.1. Principle

A calibrated tube with two references marks is filled with the test fluid. A calibrated ball is then introduced into the tube and the time taken for the ball to fall between the two reference marks is measured. This time together with the values of the densities of the fluid and the ball respectively and the calibration constant for the viscometer is used to calculate the viscosity.

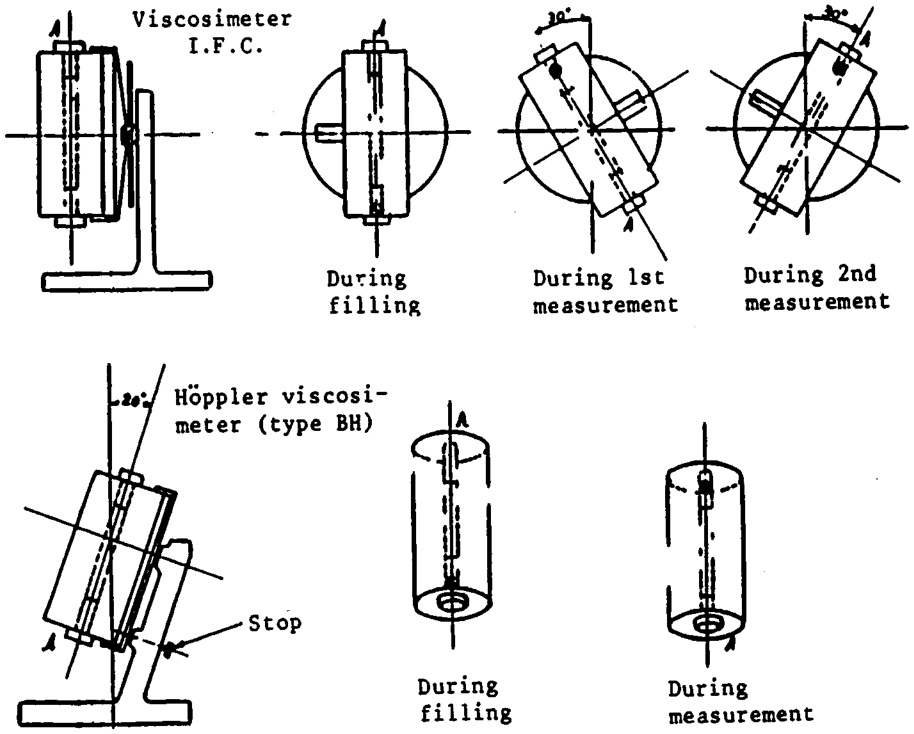
5.1.2.2. Apparatus

a) Viscometer

The measurements can be carried out with two types of apparatus based on the same principle, but of different design :

- The Höppler viscometer, type BH, with which measurement is carried out in a tube inclined at an angle of 20° to the vertical ;
- the IFC viscometer, with which the measurement is carried out in a tube inclined at 30° to the vertical.

MEASUREMENT OF KINEMATIC VISCOSITY



Since the number of measurement operations possible depends on the number of balls used in each type of instrument, the possible measuring ranges are given in the following table :

Balls	Höppler, type BH	IFC
1	0.3 to 3 cSt	1 to 50 cSt
2	3 to 30 cSt	50 to 2,500 cSt
3	25 to 250 cSt	2,500 to 25,000 cSt
4	250 to 2,500 cSt	
5	2,500 to 25,000 cSt	
6	8,000 to 80,000 cSt	

Steel balls are used in the IFC instruments, while in the Höppler type BH apparatus they are either steel or glass.

b) Thermostats

The accuracy of the measurements depends on the exactitude and constancy of the test temperature. It is in practice necessary to use a thermostat, whose temperature must be kept constant to within :

± 0.05°C at measurement temperatures below + 20°C and

± 0.10°C at measurement temperatures above + 20°C.

For a measurement range from - 20°C to + 100°C it is preferable to use two different bath fluids according to the measurement temperature :

- purified kerosene (or aviation spirit) for temperature below + 20°C ;
- pure glycerine or white vaseline oil for temperature above + 20°C ;

c) Description of apparatus and method of use

The Höppler type BH and IFC instruments are illustrated in the accompanying sketches. Detailed descriptions are given in the instructions for use issued by the manufacturers, in the French Standard (AFNOR-T 42-011) and in the corresponding German Standard (DIN 53015).

5.1.2.3. Procedure

The fluid is poured into the calibrated tube ; the ball is then inserted and the instrument re-closed, once any bubbles which may have formed during filling have gone. The fluid is brought to the test temperature ; and six successive measurements are taken. The time t quoted in the formula given in section 5.1.2.4 is the arithmetic mean of the times recorded during these six measurements. These times should lie between 25 and 500 seconds.

This condition requires the use of different balls for the same fluid, in particular for measurements at temperatures below 50°C. The calibrated tube (40 ml with the Höppler BH and 30 ml with the IFC instrument) must be emptied each time balls are changed ; in this particular case it is not necessary to clean the interior of the calibrated tube.

Note : Cleaning the calibrated tube and the balls

The tube is cleaned with solvents and rinsed with pure toluene. In the case of aqueous solutions, clean with a hot solution of soda containing 5% concentrated ammonia to remove any grease, and then rinse with distilled water.

The balls are cleaned with solvents and then toluene, and handled with clean tongs to avoid contact with the fingers.

5.1.2.4. Results

The arithmetic mean of the times recorded during the six measurements made in accordance with section 5.1.2.3. is used to derive the viscosity by the following calculation :

Where :

na = absolute viscosity of the fluid in centipoises;

nc = kinematic viscosity of the fluid in centistokes;

K = calibration constant for the calibrated tube/ball assembly used;

t = falling time in seconds;

D = density of ball material at measurement temperature;

d = density of the test fluid at measurement temperature:

$$na = t \times (D - d) \times K$$

$$nc = \frac{na}{d} = t \times \frac{D - d}{d} \times K$$

Notes : Density

The densities in this calculation are those at the temperature of measurement ; they are normally given for + 15°C, referred to water at + 4°C. The necessary corrections can be made only with homogeneous substances, and this applies only in certain special cases with fire-resistant fluids. For these fluids direct determination of the density is necessary.

Conversion of the density :

c = correction coefficient for density, for a difference of 1°C

d_o = density at + 15°C

e = difference between measurement temperature and + 15°C, in degrees C

$$d = d_o \pm c \times e$$

The correction is positive if the measurement temperature lies below + 15°C and negative if above.

Constant K

The constants K are independent of the test temperature. They are determined for a given calibrated tube and a specific inclination of the tube (30° for the IFC and 20° for the Höppler BH instrument). When a calibrated tube or balls are changed, or when measurements are undertaken at an angle other than 30° (using the IFC apparatus) these constants must be determined anew, using a standard reference fluid.

These constants must be quoted in the test reports for each instrument.

Thixotropic fluids

In the case of thixotropic fluids the measured falling times will decrease progressively from the first measurement onwards, until they reach a stable minimum value at the end of measurement. The value to be accepted is the minimum time for disappearence of the gel. The difference between the first falling time and the minimum time is an index of the thixotropy of the test fluid.

5.1.2.5. Acceptance criteria

The kinematic viscosity of emulsifying oils for type HFA fluids shall not exceed 350 cSt at 20°C. (This requirement is imposed to ensure that the emulsifying concentrate can be satisfactorily mixed by an injector).

The manufacturer shall state the kinematic viscosity in cSt for types HFA (other than type HFA1), HFB, HFC and HFD fluids at the temperatures specified in table 1. When such fluids are tested at a temperature of 40°C, the average value of all measurements of viscosity shall lie within ± 10% of the stated value for that temperature.

The manufacturer's stated value for viscosity should, where practicable, lie within one of the viscosity ranges which are permitted by the viscosity grading system of ISO 3448 i.e. within ± 10% of the viscosity grade designations specified in section 2.2.2.2. Fluids may be exempted from this requirement by agreement between the user and the manufacturer. Exemption may be granted where it is impossible or impracticable to manufacture a fluid to a predetermined level of viscosity.

5.2 METHODS OF TEST FOR THE STABILITY OF EMULSIONS

The methods of test for the stability of emulsions described in sections 5.2.1 to 5.2.4 are intended for use with types HFA and HFB fluids only. The temperatures and durations of the tests are summarised in the following table :

Fluid	Tests specified
HFA	One test at either 70°C for 168 h or 50°C for 600 h
HFB	Two tests: (a) 20°C for 1000 h (b) 70°C for 48 h
HFB...LT	As for HFB then: One test at - 10°C for 336 h followed by 168 h at 20°C

Note that in the case of type HFA fluids, an alternative temperature and test time are included. This is to permit those countries which have, up to now, used exclusively the methods of test prescribed in Part III of the Fifth Report to continue to do so for the time being. In some countries tests at 70°C have been done for many years and this permits a shorter test period.

The appropriate acceptance criteria including those for the alternative temperatures and times are specified for each individual test method.

5.2.1 METHOD OF TEST FOR EMULSION STABILITY OF TYPE HFA FLUIDS (EXCLUDING TYPE HFAS FLUIDS)

5.2.1.1 Scope

This method of test shall be applied to type HFA fluids excluding type HFAS fluids to assess the stability of emulsions made up with waters having clearly defined concentrations of salts. The specifications for three standard waters used for this purpose are given in section 2.2.2.6.

5.2.1.2 Principle

The sample of emulsifying oil submitted for test is used to make up an emulsion with the particular test water with which it has been declared compatible by the manufacturer (see section 2.2.2.6). Observations of instability of the emulsion are made after it has been stored for 168 h at a temperature of 70°C. Alternatively the test may be carried out at a temperature of 50°C for a period of 600 h.

5.2.1.3. Apparatus

a) Flasks with graduated necks (Hirschsohn flasks) as shown in figure 1 and of nominal capacity 150 or 200 ml, or alternatively, flasks to DIN 51368;

b) Cork bungs to fit the above flasks;

c) An oven capable of maintaining a temperature of 70°C within ± 2°C or, when appropriate, 50°C ± 2°C.

5.2.1.4. Procedure

An emulsion shall be prepared for test using one of the standard test waters (normally the hardest of the test waters with which the manufacturer has declared the emulsifying oil to be compatible), see section 2.2.2.6. The proportion of emulsifying oil in the emulsion shall be that recommended by the manufacturer for normal applications or such other value as may be specified by the approving/accepting authority.

A clean dry flask shall be filled with the emulsion to approximately the middle of the graduated scale. Either capacity flask may be used but if there is reason to expect much separation it is advisable to use the larger capacity flask. A cork bung shall then be inserted to prevent evaporation. The stoppered flask shall be allowed to stand in the oven for 168 hours at a temperature of 50°C whichever is the chosen alternative.

As the emulsion heats to the test temperature, it is necessary to ensure that the cork bung is not displaced and that the emulsion level does not rise above the top graduation mark of the flask.

At the end of the test period and while the flask and its contents are still hot, any free oil floating on the surface of the emulsion shall be observed. Its volume shall be measured if it is sufficient to allow the graduations on the neck of the flask to be used for this purpose. Similarly the extent of any creaming of the emulsion in the neck of the flask shall be observed and, where possible, measured. Any separated water in the bottom of the flask and any flocculation in the emulsion shall be observed.

5.2.1.5. Results

The following shall be reported :

a) the identification of the emulsifying oil supplied ;

b) the designation of the test water used ;

c) the concentration of the oil in the emulsion ;

d) the presence or otherwise of free oil on the surface of the emulsion and, where measurable, its volume in ml per 100 ml of emulsion sample ;

e) the presence or otherwise of creaming in the upper part of the emulsion and, where measurable, its volume in ml per 100 ml of emulsion sample ;

f) the presence or otherwise of separated water at the bottom of the emulsion ;

g) the presence or otherwise of flocculation in the emulsion.

5.2.1.6. Acceptance criteria

HFA fluids made up from emulsifying oils and the selected test water shall satisfy the following requirements:

i) no free oil evident on surface of emulsion sample;

ii) creaming to be not greater than 0.1 ml per 100 ml of emulsion sample;

iii) no water separation at bottom of emulsion sample; and

iv) no flocculation evident in the emulsion sample.

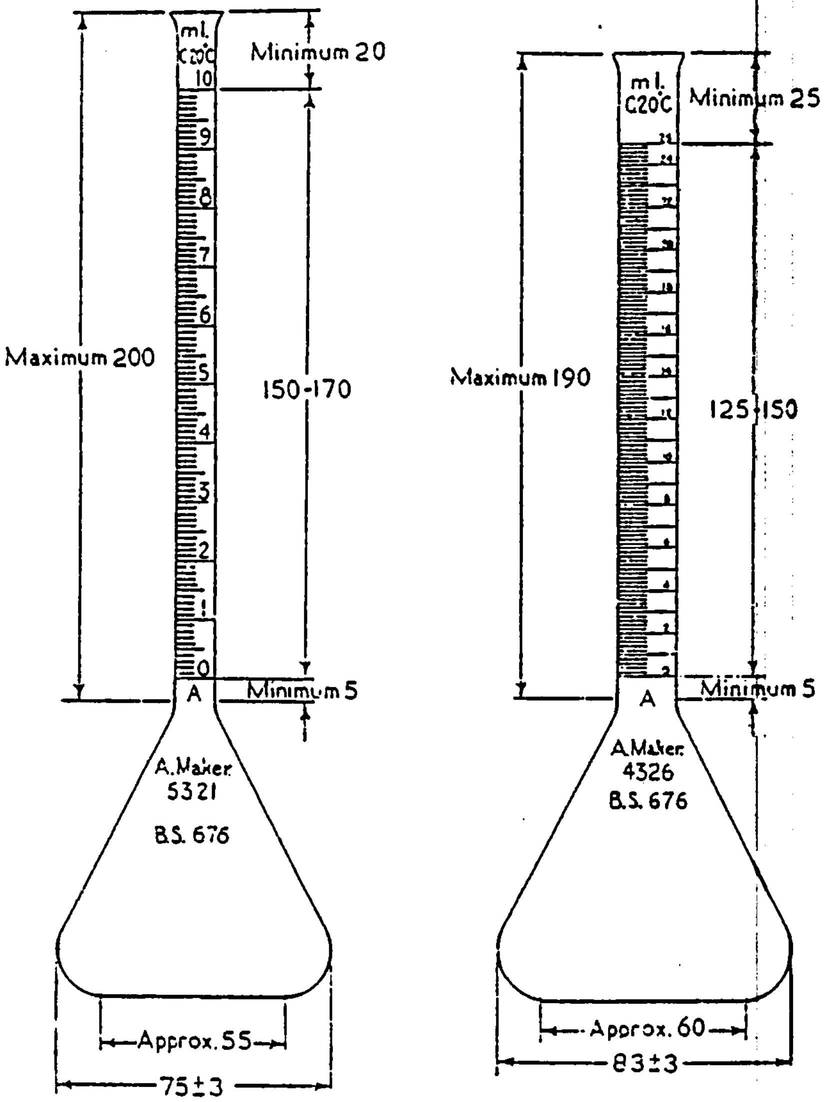


Figure 1 Hirschsohn flasks

Note: Where Hirschsohn flasks to the above dimensions are not available a suitable alternative would be flasks to DIN 51368.

5.2.2 METHOD OF TEST FOR EMULSION STABILITY OF TYPE HFB FLUIDS AT AMBIENT TEMPERATURE

Note : This method of test is technically equivalent to the Institute of Petroleum Test Method IP 290/73.

5.2.2.1 Scope

This test shall be applied to type HFB and type HFB...LT fluids only to assess their stability during storage at ambient temperature.

5.2.2.2 Principle

A graduated measuring cylinder is filled with 450 ml of emulsion above a 50 ml layer of water. After 1,000 hours storage 50 ml samples are pipetted from two defined levels in the upper and lower regions of the emulsion column. The water contents of these samples are compared with the measured initial water content. Measurements of the surface oil layer volume and the volume of accumulated free water are also taken.

5.2.2.3 Apparatus

a) Test vessels

500 ml capacity graduated measuring cylinders with a scale length from the 50 ml to the 500 ml graduations of 250 ± 25 mm and an overall height of approximately 390 mm.

b) Pipettes

50 ml capacity pipettes to B.S. 1583 grade A or B are suitable. Note that precautions should be taken to guard against danger from implosions whilst the pipettes are under vacuum.

c) Vacuum pump

A pump capable of maintaining a vacuum of 50 kPa (375 mm of mercury), fitted with a vacuum gauge and a means of adjusting the applied suction.

d) Sampling jig

A jig consisting of a retort stand and clamp as shown in figure 1.

e) Stop clock

f) Equipment and reagents for water content determination by the distillation method described in section 5.5.1.

g) Polyethylene film approximately 0.05 mm thick.

5.2.2.4 Sampling

A sample of not less than 1.5 litre of fluid shall be supplied for test. Care shall be taken to ensure that the sample is representative of the bulk.

5.2.2.5 Procedure

Two 500 ml measuring cylinders shall be cleaned, dried and prepared as follows : Each cylinder shall be filled to the level of the 50 ml graduation with distilled water from a pipette, taking care not to splash the internal surface of the vessel. The supply of test fluid shall be thoroughly mixed and from this about 50 ml of emulsion shall be added gently on top of the water layer in the cylinders, using a pipette. The filling of the cylinders to the 500 ml graduations shall be completed by pouring emulsion down a glass rod. The mouth of each test vessel shall then be sealed by a piece of polyethylene film bound in place.

Storage

The cylinders shall stand undisturbed for 1,000 hours at a temperature of 20 ± 2°C. They shall be protected from direct sunlight and draughts and stored at a location remote from sources of heat.

Oil layer and free water measurement

From each cylinder readings shall be taken of the surface oil layer volume and the change in water layer volume and the change in water layer volume, i.e. the volume of accumulated free water, after 1,000 hours standing time.

Sampling from the test vessels

Samples shall be taken from the cylinders after 1,000 hours standing time, using the following procedure :

The seal shall be removed from the mouth of the test vessel. A 50 ml pipette shall be mounted vertically in clamp A of the sampling jig shown in figure 1. The tip of the pipette shall be aligned externally with the 425 ml graduation of the measuring cylinder using a set square, and stop C shall be released and the pipette raised and positioned with its stem on the vertical axis of the measuring cylinder. The mouth of the pipette shall be sealed.

The pipette shall then be lowered carefully into the fluid until clamp B rests on stop C, when clamp B shall again be tightened.

The seal shall be removed from the mouth of the pipette and the pipette shall be filled at a steady rate by applying suction, until the fluid level is about 50 mm above the pipette graduation mark.

The vacuum used for this purpose shall be established in advance by trial sampling runs from a uniformly dispersed sample of the fluid under test, at a temperature of 20 ± 2°C. The vacuum shall be such that any pipette used for subsequent sampling from the stability columns is filled in a time of not less than 60 seconds.

The pipette shall be removed from the sampling jig and all surplus fluid shall be wiped from the exterior of the pipette stem. The fluid volume in the pipette shall be adjusted to 50 ml by dispensing excess fluid.

The 50 ml sample shall be allowed to drain from the pipette into a clean and dry 500 ml round bottomed flask, as used for water determination by the distillation method. The residual fluid shall be rinsed from the pipette into the flask using several charges of water-free petroleum spirit (90 to 160°C boiling range), from a graduated wash bottle. A total volume of approximately 100 ml petroleum spirit shall be used for this operation. The diluted sample of emulsion shall be used for subsequent water content measurement. The sampling procedure shall be repeated in order to obtain a 50 ml sample from the 125 ml level of the same measuring cylinder.

The sampling procedure shall be repeated in order to obtain samples from the 425 ml level and the 125 ml level of the second measuring cylinder.

Water content measurements

The percentage water content by volume x_s, of each pipetted sample shall be determined to the nearest 0.1 by the distillation method, given in section 5.5.1. (Note 1).

Two determinations of the initial water content, x_i shall be made on thoroughly mixed samples of emulsion retained for this purpose.

5.2.2.6. Results

The following calculations shall be made :

the mean $\bar{x}_i$ of the two values of initial water content;

the changes in water content x_d which shall be calculated from $x_s - \bar{x}_i$;

the means of the two determinations of x_d which correspond to each sampling level.

The following results shall be reported :

1. the mean change in percentage water content at the 425 ml level after 1,000 hours;
2. the mean change in percentage water content at the 125 ml level after 1,000 hours;
3. the mean surface oil layer volume (in ml) after 1,000 hours;
4. the mean volume of accumulated free water (in ml) after 1,000 hours.

Note 1 : The apparent water content shall be determined where appropriate.

5.2.2.7. Acceptance Criteria

Types HFB and HFB..LT fluids shall satisfy the following requirements when tested in accordance with the above method.

i) the mean change in percentage water content at the 425 ml level shall not exceed 5 ;

ii) the mean change in percentage water content at the 125 ml level shall not exceed 5 ;

iii) the mean surface oil layer volume shall not exceed 10 ml ;

iv) the mean volume of accumulated free water shall not exceed 2 ml.

PART V 5.2.2.

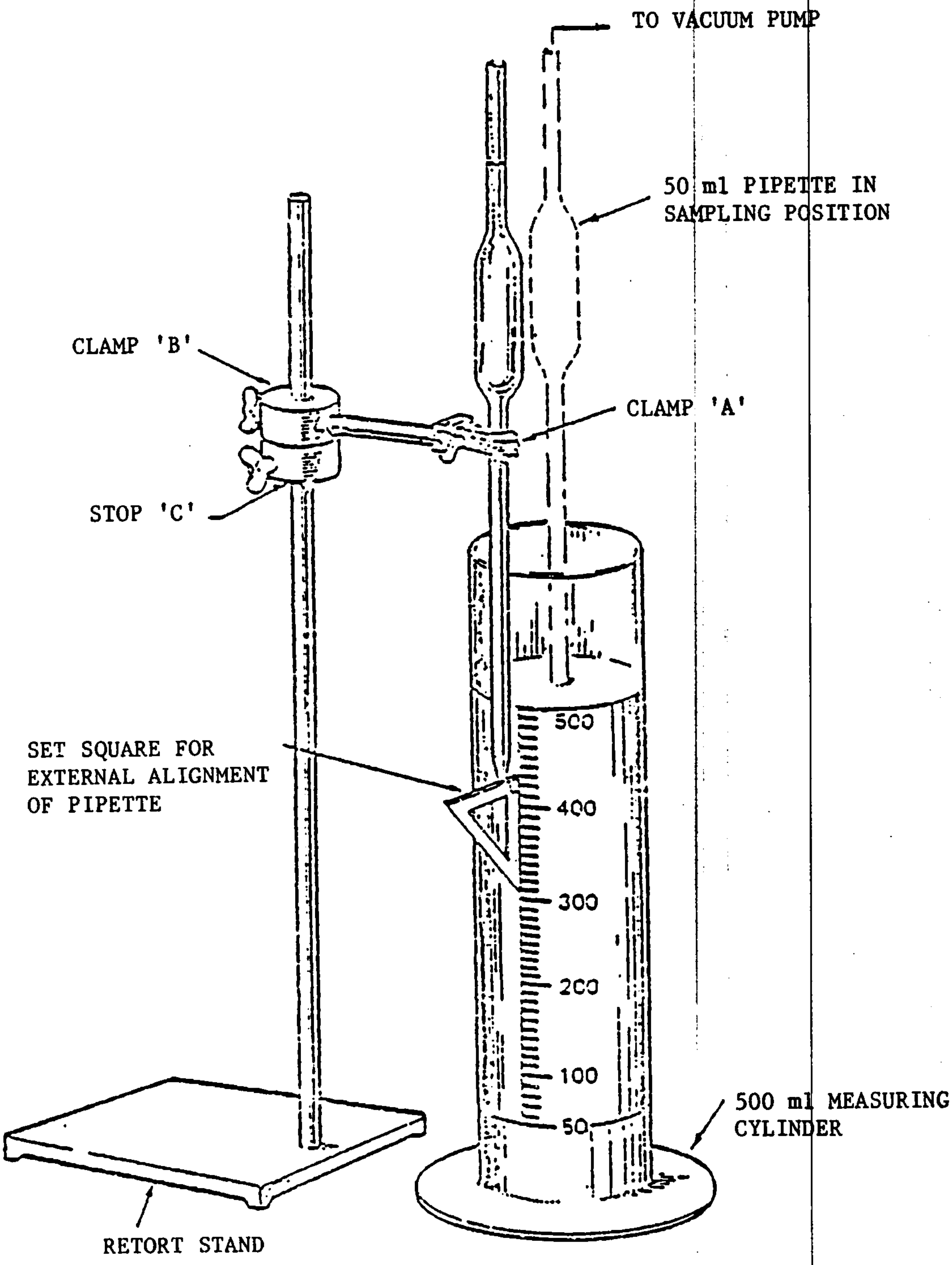


Figure 1 Sampling jig

5.2.3. METHOD OF TEST FOR EMULSION STABILITY OF TYPE HFB FLUIDS AT MEDIUM TEMPERATURE

5.2.3.1. Scope

This test shall be applied to type HFB and type HFB..LT fluids only, to assess emulsion stability at medium temperature.

5.2.3.2. Principle

The emulsion is held at a temperature of 70°C in a 100 ml Crow receiver and measurements are made of the volumes of water and oil which separate after a period of 48 h.

5.2.3.3. Apparatus

a) Test Vessels

100 ml Crow receivers conforming the dimensions given in Figure 1

b) Polyethylene film - approximately 0.05 mm thick.

c) Oven

An air oven capable of maintaining a temperature of 70°C within ± 1°C.

5.2.3.4. Sampling

A sample of not less than 500 ml of fluid shall be supplied for test. Care shall be taken to ensure that the sample is representative of the bulk.

5.2.3.5. Procedure

The sample shall be thoroughly mixed by shaking in a vessel having a capacity substantially greater than the volume of the sample.

Two clean dry 100 ml Crow receivers shall then each be filled with fluid to the 97 ml graduation (See Note 1). The mouth of each test vessel shall then be sealed by a piece of polyethylene film bound in place.

The test vessels shall then be placed, without delay, in an air oven which shall be maintained at a temperature of 70°C throughout the period of test. The test vessels shall stand upon a perforated shelf and shall be positioned centrally within the oven. The test vessels shall remain undistributed for a period of 48 h. At the end of this period readings shall be taken to the nearest 1 ml of the volume of the surface oil layer (see note 2) and the volume of accumulated free water in each test vessel. These four readings shall be taken without disturbing the test vessel and before any appreciable cooling occurs.

5.2.3.6. Results

The following results shall be reported:

a) the surface oil layer volume in ml in each test vessel;

b) the volume of accumulated free water in ml in each test vessel;

c) the average of the two measurements of surface oil layer volume;

d) the average of the two measurements of the volume of accumulated free water.

Note 1 : The fluid will expand to approximately 100 ml on heating to the test temperature.

Note 2 : It is unlikely that the upper surface of the fluid will coincide exactly with the 100 ml graduation of the test vessel. The surface oil layer volume shall be estimated as the difference in scale readings between the upper surface of the fluid and the lower surface of the oil layer.

5.2.3.7. Acceptance criteria

Types HFB and HFB..LT fluids shall satisfy the following requirements when tested in accordance with the above method :

i) the average value of the two measurements of surface oil layer volume shall not exceed 3 ml;

ii) the average value of the two measurements of accumulated free water shall not exceed 1 ml.

PART V 5.2.3.

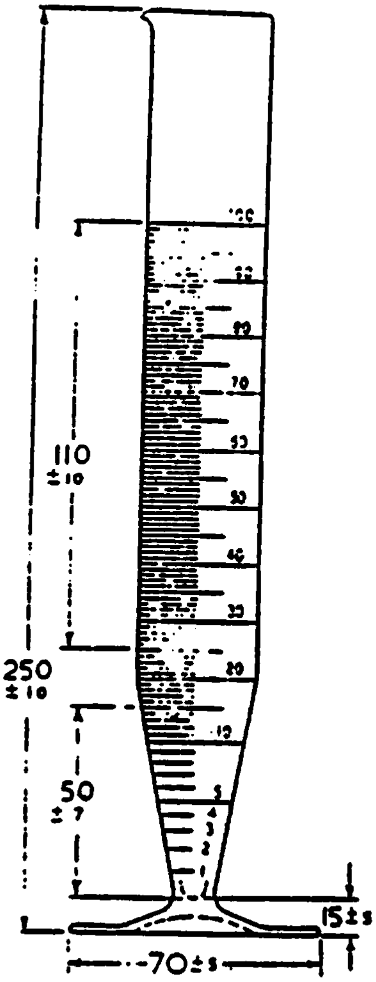


Figure 1 Test vessel, 100 ml Crow receiver (all dimensions in mm)

5.2.4. METHOD OF TEST FOR EMULSION STABILITY OF TYPE HFB...LT FLUIDS AT LOW TEMPERATURE

5.2.4.1. Scope

This test shall be applied to type HFB..LT fluids only, to assess the effect on emulsion stability of storage at low temperature.

5.2.4.2. Principle

The emulsions is held at a temperature of - 10°C for 336 h in a 100 ml Crow receiver. The temperature is then raised to + 20°C for a further 168 h and readings are taken of the volumes of water and oil which separate from the emulsion.

If these readings are satisfactory, a 10 ml sample is pipetted from the 5 ml level of the test vessel. The water content of this sample is determined and compared with a measurement of initial water content.

5.2.4.3. Apparatus

a) Test vessels - 100 ml Crow receivers conforming to the dimensions given in figure 1.

b) Refrigerated chamber - capable of maintaining a temperature of - 10°C within ± 1°C.

c) Polyethylene film - approximately 0.05 mm thick.

d) Pipettes - 10 ml capacity.

e) Stop clock or other suitable interval timing device.

f) Equipment and reagents for water determination by the distillation method given in section 5.5.1.

5.2.4.4. Sampling

A sample of not less than 1 litre of fluid shall be supplied for the test. Care shall be taken to ensure that the sample is representative of the bulk.

5.2.4.5. Procedure

The sample shall be thoroughly mixed by shaking in a vessel having a capacity substantially greater than the volume of the sample.

Six clean, dry 100 ml Crow receivers shall each be filled with the fluid to the 100 ml graduation. The mouth of each test vessel shall be sealed by a piece of polyethylene film bound in place.

The test vessels shall be placed, without delay, in a refrigerated chamber which shall be maintained at a temperature of - 10 ± 1°C throughout the period of the test.

The test vessels shall remain undisturbed for a total of 336 h. At the end of this period the test vessels shall be carefully transferred to an ambient temperature of 20 ± 2°C.

The test vessels shall remain undisturbed at this temperature for a further 168 h, after which readings shall be taken to the nearest 1 ml of the volume of the surface oil layer and the volume of accumulated free water in each test vessel.

If the readings of surface oil layer volume and accumulated free water volume satisfy the requirements (i) and (ii) of section 5.2.4.7. then the following additional procedure shall be carried out without delay. If these readings do not satisfy the requirements (i) and (ii) of section 5.2.4.7. then the following procedure shall be omitted.

A sample shall be taken from each test vessel as follows.

A 10 ml pipette shall be introduced carefully into the test vessel and held in place such that the tip of the pipette is positioned on the vertical axis of the test vessel at the depth of the 5 ml graduation.

The pipette shall be filled at a steady rate by applying suction until the fluid level is about 20 mm above the pipette graduation mark. The time of filling shall not be less than 60 s.

The pipette shall be removed from the test vessel and all surplus fluid shall be wiped from the exterior of the pipette stem. The fluid volume in the pipette shall be adjusted to 10 ml by dispensing excess fluid. The percentage water content by volume of this 10 ml sample shall be determined to the nearest 0.5 by the distillation method, given in section 5.5.1. (See Note 1). Two determinations of initial water content shall also be made on a thoroughly mixed portion of the original fluid sample retained for this purpose.

Note 1 : The Apparent Water Content shall be determined where appropriate.

5.2.4.6. Results

The change in percentage water content at the 5 ml level shall be calculated for each test vessel by subtracting the mean value of initial water content from the water content of the sample pipetted from the test vessel.

The following results shall be reported :

1. the mean surface oil layer volume in ml;
2. the mean volume of accumulated free water in ml;
3. the change in percentage water content at the 5 ml level for each test vessel;
4. the mean of the six changes in percentage water content at the 5 ml level.

5.2.4.7. Acceptance Criteria

Type HFB..LT fluids shall satisfy the following requirements when tested in accordance with the above method.

i) the average value of the six measurements of surface oil layer volume shall not exceed 2.0 ml ;

ii) the average value of the six measurements of the value of accumulated free water shall not exceed 1.0 ml ;

iii) the change in percentage water content at the 5 ml level shall not exceed 15 for each test vessel ;

iv) the mean change in the percentage water content at the 5 ml level for the six test vessels shall not exceed 10.

PART V 5.2.4.

Figure 1 Test vessel, 100 ml Crow receiver (all dimensions in mm)

5.3 AGEING AND THERMAL STABILITY

The test requirements for the ageing and thermal stability of fire-resistant fluids depend to a large extent on the type of application and the conditions under which the fluid is to be used and, in particular, on whether it is to be used in a hydrostatic or a hydrodynamic application.

The tests described in this report have been selected to be representative of both types of application. They are intended to be applied to types HFC and HFD fluids only. Having regard to their normal conditions of use, types HFA and HFB fluids are considered to be adequately tested by the methods of test for emlsion stability described in section 5.2.

Fluids intended for use in hydrostatic applications are to be tested for their ageing properties by the methods of test described in sections 5.3.1 and 5.3.2 for types HFC and HFD fluids respectively.

For those type HFD fluids which are intended for use in hydrodynamic applications, suitable tests for thermal stability, including tests in fluid couplings under operating conditions similar to those of normal service are also described. As the acceptance criteria for the latter tests in couplings cannot be precisely defined and are usually the subject of agreement between the user and the manufacturer, all tests relating to type HFD fluids for hydrodynamic applications are contained in Part VI, Discretionary Technological Tests (see section 6.7).

5.3.1. METHOD OF TEST FOR AGEING PROPERTIES OF TYPE HFC FLUIDS

Note : This method uses the apparatus and ageing conditions described in method ASTM D 943-54, DIN 51587. The apparatus, the determination of the ageing properties and the examination of the samples used are modified to suit the field of application of HFC fluids.

5.3.1.1. Scope

This method is used to determine the ageing properties of type HFC fluids.

5.3.1.2. Principle

The sample is exposed to a temperature of 95°C in the presence of oxygen and of iron and copper, which act as catalysts.

5.3.1.3. Apparatus and chemicals

a) An oxidation cell (figure 1) with a condenser 200 mm in length.

b) A thermostatically-controlled bath to maintain the liquid in the oxidation cell at a temperature of 95 ± 0.2°C and fitted with a stirrer to ensure that the temperature in the bath is uniform. The dimensions of the latter should be such that the necessary number of oxidation cells are surrounded by the liquid in the bath to a height of 350 mm.

c) A flow-meter with a minimum capacity of 3 l/h and an accuracy of ± 0.1 l/h.

d) A wire coiling mandrel (figure 2).

e) A thermometer, graduated in increments of 0.1°C allowing temperatures to be measured in the range 75 to 125°C.

f) A catalyst consisting of a Washburn and Moen No 16 low-alloy steel wire (low-alloy steel wire, type A material, specification ASTM A 129, part 1, standard specification ASTM 1955 for ordinary-quality open-hearth furnace iron plate) with a diameter of 1.59 mm with a diameter of 1.625 mm.

g) Hydrochloric acid. tech. conc. (Specific gravity = 1.18).

h) Hydrofluoric acid. tech. conc. (approx. 50%)

i) Standard grade petrol, boiling range 65 to 95°C.

j) Nitric acid. tech. conc. (Specific gravity = 1.42).

k) Acetone.

l) Sulpho-chromic acid.

m) Oxygen with a pressure regulator to ensure a constant flow of gas. It is recommended that a two-stage regulator is used on the oxygen cylinder.

5.3.1.4. Preparation of the Apparatus

a) Cleaning the catalyst

On the day of the test 3 m of steel wire and 3 m of copper wire are cleaned with an absorbent cotton-wool wad soaked in petrol. The surface of the wire is then polished with abrasive paper (emery) No 600 (00). A dry cotton-wool wad is then used to remove any metal or emery particles remaining on the wire. Any subsequent handling of the wire is done with cotton-wool or with cotton gloves in order to prevent contact with the skin.

b) Preparation of the wire coils

The two wires are fixed together at one end with about six twists. They are then wound in parallel fashion around a threaded mandrel (figure 2) and the free ends of the two wires are twisted together about six times. The reels are then removed from the mandrel and the length adjusted to 230 ± 5 mm.

c) Cleaning of the oxidation cell

The inlet pipes and the reaction flasks are cleaned by rinsing with acetone, tap water, sulpho-chromic acid and then tap water again until the latter contains no more acid. They are then rinsed again twice with small quantities of acetone and three times with distilled water. Finally, the reaction flask is filled with distilled water, the oxygen inlet pipe is placed in position together with the cooler and the apparatus is left thus for at least 24 hours before commencing the test. Shortly before the test is begun the reaction flasks are emptied and dried and in addition the outer surfaces of the inlet pipes and cooler are dried with cotton-wool.

d) Cleaning of the reaction flasks after use

After use the reaction flasks are washed in petrol and rubbed with a long handled brush. This operation is then repeated with acetone instead of petrol and the mines are filled with an oxidizing mixture composed of three parts HCl and one part HNO_2. The apparatus is then left for at least 24 hours at room temperature. It is then rinsed with tap water to remove all trace of acid and the organic products from the reaction are removed using acetone. If deposits remain inside it is rinsed again with a mixture comprising equal amounts of hydrofluoric acid and hydrochloric acid. This mixture of acids is left in the flask until the deposits are dispersed or dissolved, after which the acid mixture is washed out with running water. Subsequent cleaning is carried out as described in paragraph c).

5.3.1.5. Procedure

a) The bath is brought to a sufficiently high temperature so that the test liquid in the necessary number of reaction flasks can be maintained at the required temperature of 95 ± 0.2°C.

b) The catalyst coils are then slid on to the oxygen inlet pipe and the pipe with the coils are then centred in the flask. 390 ml of test liquid is poured over the coils into the reaction flask and the reaction flask is immersed in the bath with the liquid in the bath being at least 75 mm above the level of the test liquid. The condenser is then fitted to the inlet pipe and the cooling water is circulated (during the test the temperature of the cooling water must not exceed 35°C).

c) The oxygen inlet pipe is connected to the oxygen cylinder via flow-meter and the flow of gas is set at 3 ± 0.5 l/h. A note is kept of the time of commencement of the test. The flow of oxygen must be checked at least twice a day to ensure that the required test limits are maintained.

d) For at least three hours after the start of the test hourly checks must be made of the temperature of the mixture in the reaction flask until two successive readings give a constant temperature of 95 ± 0.2°C. After this daily checks must be carried out to ensure that the temperature in the bath remains constant during the period of the test.

e) The level of liquid in the oxidation call is kept constant by topping up with test liquid. Under certain conditions, caused by deposits or the formation of emulsions, the liquid cannot be inspected. It is for this reason that the level of the liquid is marked before commencing the test. If analysis samples are taken from the cell a mark is made of the total volume removed and the level of test liquid is maintained exactly according to this mark.

5.3.1.6. Analysis of the ageing process

a) During the ageing process samples of approx. 10 ml are taken every two days from the liquid in the reaction flask for analysis purposes. When this is being done care must be taken to ensure that the oxygen supply is cut off.

b) Each analysis sample of 10 ml is divided into two parts, one (approx. 5 g) being used to determine the pH value and the other for determining the nonsoluble substances which have been produced (sludge formation).

c) Determining the pH value

Measurement of the pH value is carried out in accordance with the method outlined in Section 5.4.

d) Determining the content of insoluble components

5 g of the liquid are diluted in a beaker containing 50 ml of distilled water. This solution is then filtered under partial vacuum by means of a membrane filter which has been weighed in advance (filter specifications: average pore diameter = 0.4 pm filter diameter = 40 mm) (1). One must be careful to ensure that the filter does not operate in a dry state since this causes the pores to become blocked. After this washing is carried out with distilled water until the filtrate is perfectly clear.

The membrane filter is dried in a drying oven at 105°C. It is then placed in a desiccator for one hour and finally weighed (2). It is advisable to carry out a blank filtration test with distilled water and to establish any possible variation in weight. The percentage of insoluble material is calculated from the formula P = (B-A) x 20.

(1) The weight is noted : A

(2) The new weight is noted : B

e) The test duration should amount to at least 200 hours. When the ageing test has been completed one can, if desired, proceed to the implementation of other tests.

5.3.1.7. Results

Information is required regarding the duration of the test and the following results :

- the pH value to ± 0.3 ;
- the increase in the content of insoluble components to an accuracy of 0.2% ; and
- the state of the metal coils : corrosion and deposits.

5.3.1.8. Acceptance criteria

Type HFC fluids should have a resistance to deterioration and oxidation close to that presented by petroleum products. Variations accepted during the test are as follows :

a) After the test period (200 hours) the pH value must be higher than 4.

b) The increase in the content of insoluble constituents must be less than 4%.

c) There should be no heavy deposits or corrosion on the metal coils as a result of any test.

DETERMINATION OF THE ACEING PROPERTIES OF TYPE HFC FLUIDS

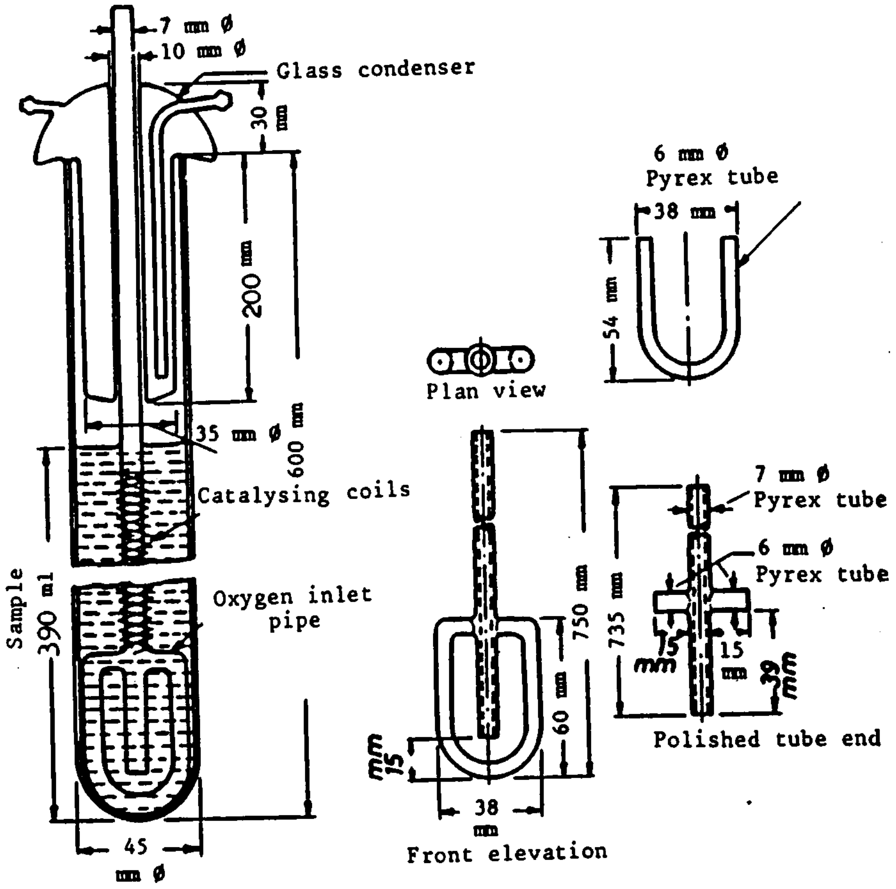


Fig. 1: OXIDIZING CELL

dimensions in mm

PART V 5.3.1.

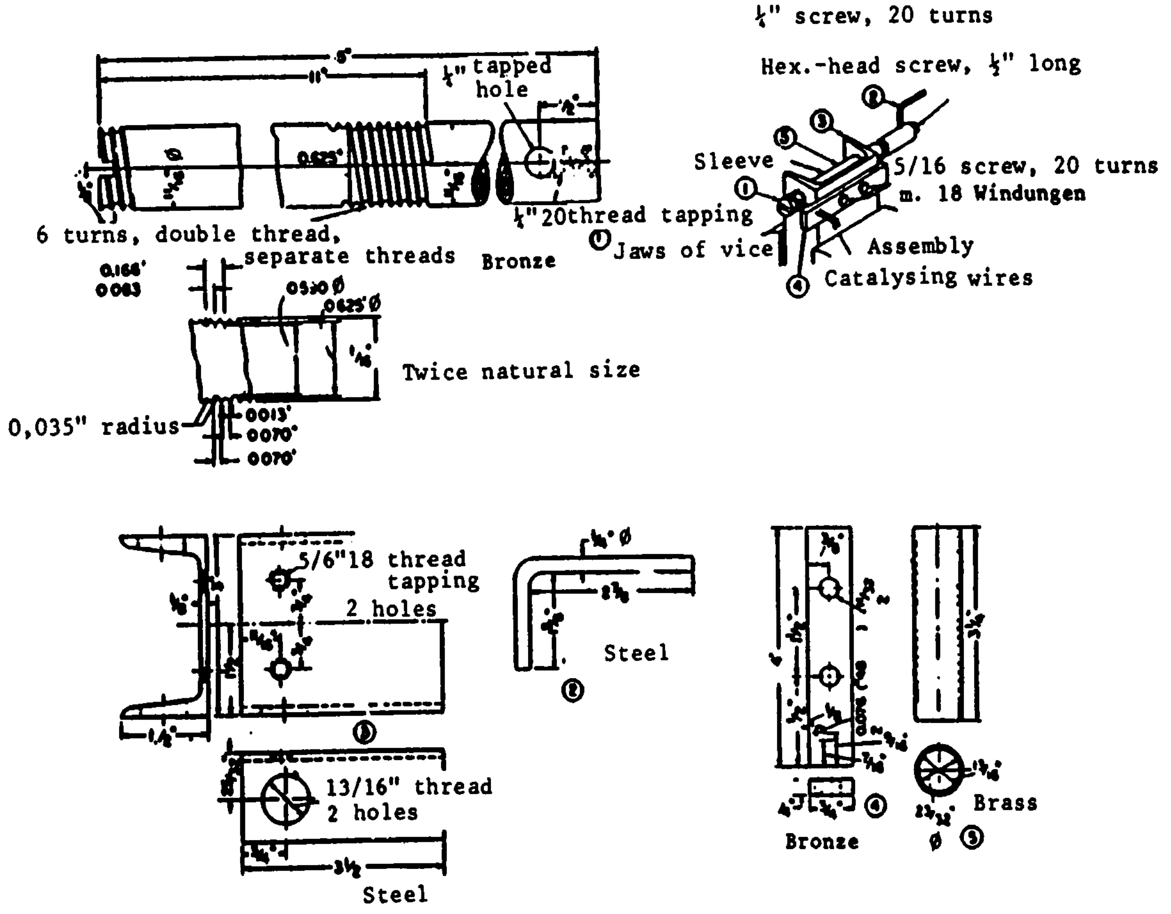


Figure 2 Mandrel for winding catalysing coils

Note: This drawing is dimensioned in inches : 1 inch = 25.4 mm

5.3.2. METHOD OF TEST FOR AGEING PROPERTIES OF TYPE HFD FLUIDS

Note : This method uses the apparatus and the ageing conditions described in standards ASTM D 94354 and DIN 51587. The method used to study the aged sample has been modified to cover the field of application of type HFD fluids.

5.3.2.1. Scope

This method serves to determine the ageing characteristics of type HFD fluids which are intended for use in hydrostatic applications.

5.3.2.2. Principle

The sample is subjected to a temperature of 95°C, in the presence of water and oxygen, with iron and copper as catalysts.

5.3.2.3. Apparatus and chemicals

a) Oxidizing call (figure 1)

b) A thermostatically-controlled heating bath which maintains the sample in the oxidizing cell at a temperature of 95 ± 0.2°C, with a suitable stirring device to ensure that the bath temperature is uniform. The dimensions of the bath must be such that the required number of oxydizing cells, 350 mm high, are completedly surrounded by the fluid in the bath.

c) Flowmeter with a minimum capacity of 3 l/h and an accuracy of ± 0.1 l/h.

d) A device for winding the catalyzing coils (figure 2)

e) Thermometer, range 75 to 125°C, graduated in 0.1°C

f) A catalyst consisting of No 16 low-alloy steel wire. Washburn & Moen (low-alloy steel wire, material type A, specification ASTM A 129, Part 1, 1955 ASTM Standards for open metal electric heating plates of ordinary grade) with a diameter of 1.59 mm, and a No 14 electrolytic copper wire, American wire, with a diameter of 1.625 mm.

g) Hydrochloric acid, concentrated industrial grade (Specific gravity = 1.18)

h) Hydrofluoric acid, concentrated industrial grade (approximately 50%)

i) Standard grade petrol, boiling range 65 to 95°C.

j) Nitric acid, concentrated industrial grade (Specific gravity = 1.42)

k) Acetone

l) Sulphochromic acid.

m) Oxygen in a container with a pressure regulator. It is convenient to use an oxygen bottle with a two-step regulator.

5.3.2.4. Preparation of the apparatus

a) Cleaning the catalyst

On the day fixed for the start of the test 3 m lengths of steel wire and copper wire respectively are cleaned with cotton wool soaked in petrol ; the surface of the wire is the polished with No 100 (00) emery paper. Any particles of metal or emery are then wiped off with dry cotton wool. In all subsequent operations, the wire should be handled with cotton cloth or cotton gloves to avoid contact with the skin.

b) Preparation of the coils of wire

The two wires are firmly joined at one end by making about six turns, and then wound uniformly next to one another on a threaded spindle (figure 2). The free ends of the steel wire and copper wire are then also joined by six turns ; the coils are removed from the spindle and then adjusted to a length of 230 ± 5 mm.

The determination of lengths makes it possible to take samples periodically from inside the oxidizing cell for analytical purposes, without materially changing the ratio of the volumes to be examined with respect to the active catalysis area (figure 1).

c) Cleaning the oxidizing cell

The inlet tubes and reaction vessels are cleaned by rinsing with acetone, tap water, sulphochromic acid and again tap water, until the latter contains no more acid. They are then rinsed twice with small quantities of acetone and three times with distilled water. Finally, the reaction vessel is filled with distilled water, the oxygen inlet pipe is fitted, together with the cooling jacket, and the whole assembly is left in this state for at least 24 hours before beginning the test. Shortly before the test begins, the reaction vessels are emptied and dried, and the outer walls of the inlet pipes and the cooling jacket dried with cotton wool.

d) Cleaning the vessels after use

After use, the reaction vessels should be washed with regular-grade motor spirit and wiped with a long-handled brush. The cleaning process is repeated with acetone replacing the petrol, after which the tubes are filled with an oxidizing mixture composed of three parts HCl and one part HNO_3, and left for at least 24 hours at room temperature. The apparatus is then rinsed with tap water to remove all traces of acid and the organic reaction products are removed by means of acetone. If a circular mark remains inside the vessel, it should be rinsed with a mixture of equal parts of hydrofluoric acid and hydrochloric acid. This mixture of acids should be left in the vessel until the mark is destroyed or dissolved, and the acids are then rinsed away with large quantities of tap water. The final cleaning is then carried out as in paragraph c).

5.3.2.5. Procedure

a) The bath is heated to a temperature sufficiently high to ensure that the test fluid, contained in the required number of reaction vessels, is maintained at the prescribed temperature of 95 ± 0.2°C, for 600 hours.

b) The catalyzing coils are then slid over the inlet end of the oxygen inlet pipe, and coil and pipe are centred. A quantity of 360 ml of the test fluid is poured on to the coil until it is thoroughly immersed. The reaction vessel is then immersed in the heating bath in such a way that the liquid in the bath stands at least 75 mm above the surface of the test fluid. The cooling jacket is then pushed on to the inlet pipe and connected to the cooling water supply (the temperature of the cooling water must not exceed 35°C during the test).

c) The oxygen inlet pipe is connected to the oxygen bottle via the flowmeter, the quantity of gas adjusted to 3 ± 0.5 l/h, and the flow of gas is allowed to continue for 30 minutes before pouring 60 ml of distilled water into the oxidizing cell. The time is then recorded. It is necessary to adjust the volume of oxygen at least twice a day to respect the prescribed tolerance.

d) For at least three hours after the beginning of the test the temperature of the mixture in the reaction vessel must be checked, every hour until two successive temperature readings are obtained at a constant 95 ± 0.2°C. Thereafter it is necessary to check once a day that the temperature remains constant throughout the test.

e) By constantly topping up with distilled water the level of fluid in the oxidizing cell is maintained constant. In certain conditions, because of deposits or the formation of emulsions, the fluid cannot easily be inspected. For this reason it is necessary to mark the fluid level before the test begins. If this level is maintained by periodic topping up, the volume of water in the cell remains constant. If test samples are removed from the cell, the total volume thus reduced is marked and the volume of water maintained exactly to this mark.

5.3.2.6. Analysis of the ageing process

a) During the ageing process, a sample of about 10 ml is taken roughly every 8 days from the centre of the fluid in the reaction vessel, for analysis, after stopping the oxygen supply.

b) The 10 ml sample is divided into two parts, one (approx. 5 g) being used to determine the neutralization number and the other to determine the presence of substances (particles of sludge) insoluble in benzene.

c) Determination of the neutralization number

This determination is carried out by the normal commercial method using alkali blue as a colour indicator.

d) Determination of the proportion of particles insoluble in benzene (1)

Some 5 g of the sample are dissolved in a flask in ten times this quantity of pure benzene (1). The solution is filtered under slight vacuum on a membrane filter which has previously been weighed (type : average pore diameter 0.4 µm filter diameter 40 mm weight "A". Care must be taken to ensure that the filter is not subjected to dry suction because this blocks the pores. The filter is then washed with pure benzene (1) until the filtrate is completely clear. After it has been allowed to stand for an hour, the benzene (1) is completely evaporated from the filter. The membrane filter is then placed to dry for half an hour in a dessicator and weighed weight "B". It is advisable to carry out a blank filtering test with pure benzene (1) since the filter itself may undergo a weight loss of 1 to 2 mg when treated with benzene. The percentage insoluble in benzene (1) is then calculated by the formula $\rho = (B-A) \times 20$.

e) The duration of the test must not exceed 600 hours of ageing. Once the ageing is finished, other tests of many different kinds can be carried out if desired.

Note (1) : The use of benzene is not permitted in the laboratories of certain countries. In such cases, the procedures should be carried out using toluene.

5.3.2.7. Results

Information is required regarding the duration of the test and the following:

- the neutralization number ;
- the increase in the content of insoluble components to an accuracy of 0.2%; and
- the state of corrosion of and deposits on the metal coils.

5.3.2.8. Acceptance criteria

The fluid should normally exhibit a resistance to ageing and oxidation close to that of petroleum based liquids. The permissible variations during the test are as follows :

After the test period (600 hours) :

a) the increase in the neutralisation number shall not be greater than 2 mg KOH/g;

b) the increase in the content of insoluble components shall not exceed 2%; and

c) there shall be no heavy deposits or corrosion on the metal coils.

PART V 5.3.2.

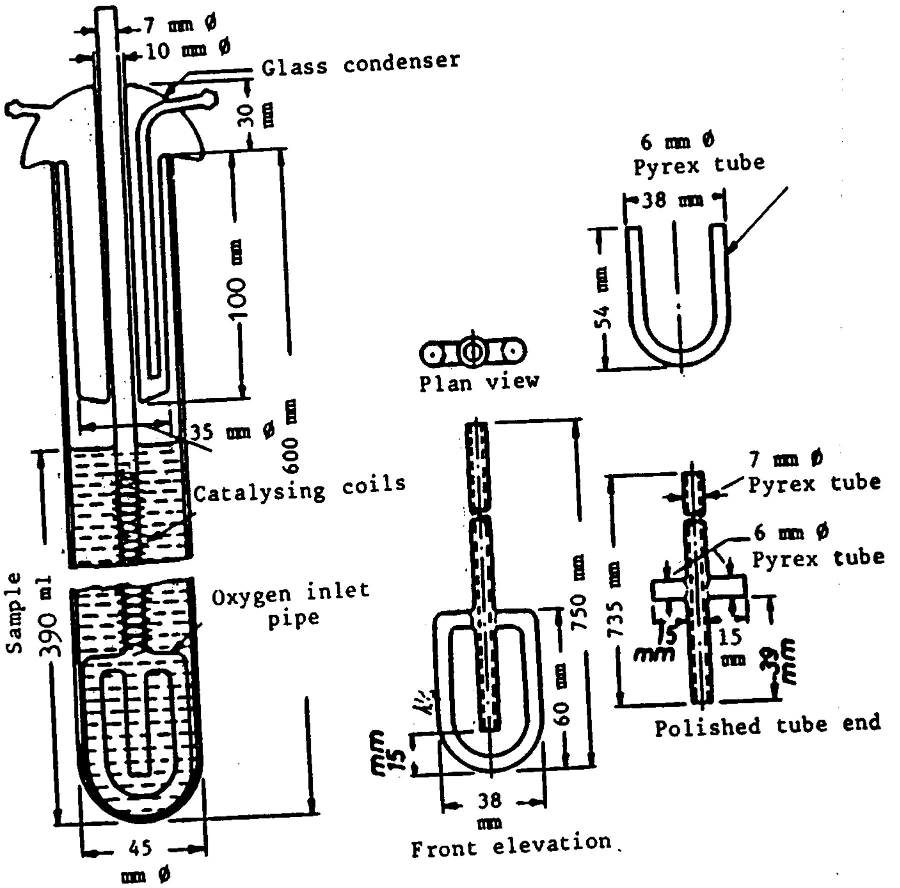


Fig. 1: OXIDIZING CELL

dimensions in mm

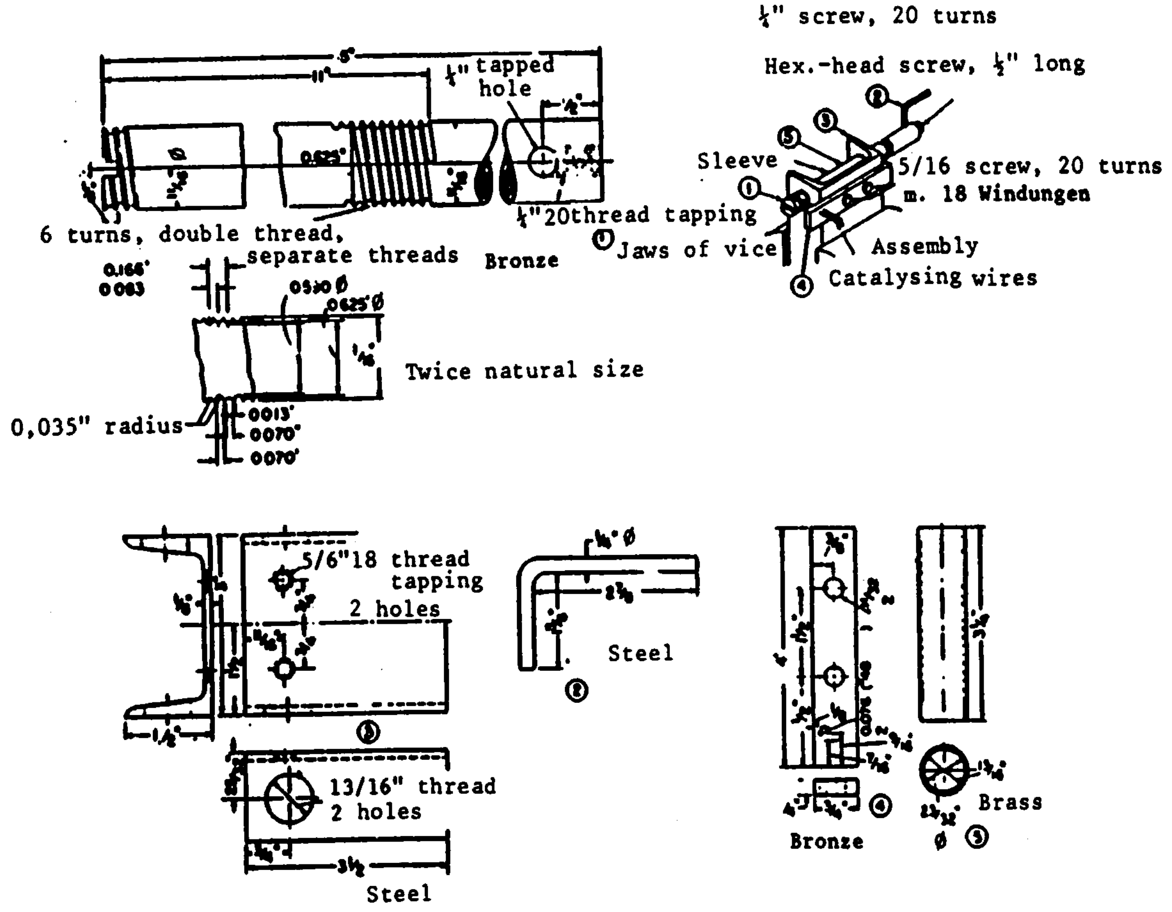


Figure 2 Spindle for winding catalysing coils

<u>Note</u>: This drawing is dimensioned in inches : 1 inch = 25.4 mm

5.4. METHOD OF TEST FOR pH VALUE OF TYPES HFA AND HFC FLUIDS

5.4.1. Scope

This method covers the determination of the pH value of HFA and HFC fluids. In the case of type HFA fluids the stability of the emulsion may be adversely affected by the inherent pH value of the raw make-up water. In such cases, therefore, the make-up water should also be tested for pH value by this method.

5.4.2. Principle

The pH value is established by using a pH meter to determine the potential difference between two electrodes immersed in the fluid being tested. One of the electrodes should be of glass and the other should be of calomel. Measurement should be carried out at a temperature of 20°C.

5.4.3. Apparatus and chemicals

a) a pH meter

b) a glass electrode (measuring electrode, alkali-resistant)

c) a saturated or non saturated calomel electrode (standard electrode)

d) buffer solutions

The buffer solutions used to calibrate the pH meter and the pair of electrodes must be prepared from salts intended for this purpose. Solutions with a pH value of less than 2.5 should be kept in chemical-proof bottles. The solution of alkaline phosphate (pH 11.72) should be kept in a tightly stoppered polypropylene bottle or a glass bottle with a parafin wax inner coating.

By way of examples, some of the buffer solutions which could be used are as follows:

- Buffer solution pH 7

A. Dissolve 1.1876 g of $Na_2HPO_4 . 2H_2O$ in 100 ml of distilled water.

B. Dissolve 2.1008 g of $C_6H_8O_7 . H_2O$ (citric acid) in 20 ml 1N-NaOH + 80 ml of distilled water.

Mix 32.94 ml of solution A with 7.06 ml of solution B.

- Buffer solution pH 9.18

Dissolve 3.81 g of borax ($Na_2B_4O_7 . 10H_2O$) in distilled water and make up to 1 litre. This solution must be shielded from atmospheric carbon dioxide.

- Buffer solution pH 11.72

Dissolve 1.42 g of anhydrous disodium hydrogen phosphate (Na_2HPO_4) in 100 ml of a 0.1 M carbonate-free solution of sodium hydroxide and dilute to 1 litre with water.

Buffer tablets or ready mixed buffer solutions may be used in place of the buffer solutions listed above, see Note (1).

5.4.4. Calibration of the pH meter

Immediately before use, soak the prepared electrodes in distilled water for at least 2 min. Touch the tips of the electrodes with a dry cloth or tissue to remove excess water.

The two electrodes are connected to the meter and then immersed, in two buffer solutions* which are maintained at a temperature of 20°C. The theoretical values are compared with those indicated on the meter. If the meter readings do not coincide with the theoretical values, the meter should be adjusted in accordance with the instructions for its use.

* The pH values of these solutions should lie on both sides of the expected pH value of the fluid being tested.

5.4.5. Procedure

The fluid which is to be tested is heated in a glass beaker to a temperature of 20 ± 1°C. The electrodes, which have been carefully cleansed in distilled water, are immersed in the fluid and the pH value is read off on the meter.

In the event that the electrodes become coated with an oil film as the result of testing two-phase products, it is necessary to remove such a film with a suitable solvent, such as a 1 - 1 mixture of toluene and ethyl acetate.

In the case of HFA fluids, the emulsion shall be prepared for test using one of the standard test waters (normally the hardest of the test waters with which the manufacturer has declared the emulsifying oil to be compatible), see section 2.2.2.6. The proportion of emulsifying oil in the emulsion shall be that recommended by the manufacturer for normal applications or such other value as may be specified by the approving/accepting authority.

5.4.6. Results

The results are indicated in pH units to the nearest 0.1. In the case of HFA fluids, the amount of emulsifying oil contained in the emulsion tested should be recorded.

5.4.7. Acceptance criteria

The pH value of HFA and HFC fluids must lie between the limiting values of 6.7 and 11 inclusive.

5.4.8. Determination of the pH Value of the water used for type HFA fluids

The stability of certain type HFA fluids, particularly when they are based on anionic emulsifying oils, may be adversely affected if the water used is acidic. With highly acidic water it may not be possible to form emulsions with this type of oil. It is recommended, therefore, that the pH value of the water used should be determined prior to the specification of a type HFA fluid for any given application.

The method described in the previous sections 5.4.1 to 5.4.7 should be used for this purpose with the addition of a buffer solution of pH value 4. This can normally be obtained in either tablet form or from pre-mixed proprietary solutions.

Note (1) Buffer solutions in the range pH 4 to 6 are available from the Merck Company, Darmstadt and an acetate buffer solution, pH 4.66, can be obtained from Riedel-DeHaen, D 3016 Seelze 1.

5.5. METHODS OF TEST FOR WATER CONTENT OF TYPES HFB AND HFC FLUIDS

5.5.1. METHOD OF TEST FOR WATER CONTENT OF TYPE HFB FLUIDS

Note : This method is based on test procedures which are included in the following Standards : ISO 3733 - 1976, BS 4385, AFNOR NFT 60-113, DIN 51582, IP 74/70 and ASTM D95-70.

5.5.1.1. Scope

The water content of type HFB and type HFB..LT fluids shall be determined by the following distillation method.

Certain fluids contain appreciable quantities of water soluble substances which will distil in addition to water during a test. Type HFB..LT fluids, for example, may contain ethylene glycol (Ethanediol). For such fluids the water content determined by distillation shall be termed the Apparent Water Content. A procedure is also given for estimating the True Water Content of such fluids by determining the concentration of water in the distillate.

5.5.1.2. Principle

A sample of fluid shall be heated under reflux with a water immiscible solvent which will co-distil with the water in the sample. The condensed water is collected in a graduated trap and the solvent returns to the still.

5.5.1.3. Apparatus

The apparatus shall comprise a glass still, a heater, a reflux condenser and a graduated glass trap. A typical assembly is shown in the figure 1. The following apparatus is recommended.

a) Still - a round bottom flask of 500 ml capacity.

b) Heater - an electric heating mantle. Means shall be provided for regulating the heating power.

c) Condenser - a straight water cooled condenser with a length of 400 mm.

d) Trap - a 10 ml trap graduated in increments of 0.1 ml and fitted with a stopcock.

e) Measuring cylinders - 10 ml capacity.

The still, trap and condenser should be connected with ground glass joints.

5.5.1.4. Reagents

Solvent-carrier liquid : - Water-free petroleum distillate of 90 - 160°C boiling range.

5.5.1.5. Sampling

A sample of not less than 250 ml of fluid shall be supplied for test. Care shall be taken to ensure that this sample is representative of the bulk.

5.5.1.6. Calibration of Apparatus

It is recommended that the apparatus should be calibrated using, for example, the calibration procedure given in ISO 3733. A mixture of 20 ml of water and 30 ml of clear hydrocarbon oil shall be tested and the permissible limits for recovered water at 20°C are 20 ± 0.4 ml.

5.5.1.7. Procedure

A 50 ml sample of fluid shall be measured to an accuracy of ± 1%, using a measuring cylinder or a pipette, and this sample shall be transferred to the still.

The residual sample adhering to the measuring cylinder or pipette shall be rinsed into the still, using one 50 ml portion and two 25 ml portions of the solvent carrier liquid. Glass beads or other boiling aids shall be added if necessary.

The apparatus shall be assembled as shown in figure 1. The condenser tube and trap shall be chemically clean in order to ensure free drainage of water to the bottom of the trap. A loose cotton-wool plug shall be inserted in the top of the condenser to prevent condensation of atmospheric moisture inside it.

Cold water shall be circulated through the jacket of the condenser. Heat shall be applied to the still and the rate of boiling adjusted so that condensed distillate discharges from the condenser at a rate of 2 to 5 drops per second.

When more than 10 ml of water has collected in the trap a portion of this water shall be drained into a 10 ml measuring cylinder by means of the stop cock. Further quantities of water shall be drained from the trap into 10 ml measuring cylinders as necessary.

The distillation shall continue until no water is visible in the still or in the side arm of the trap and the volume of water in the trap remains constant over a 5 minute period. The trap and its contents shall then be allowed to cool to room temperature. Any drops of water adhering to the sides of the trap shall be dislodged with a glass rod and transferred to the water layer. The total volume of water collected during the distillation shall be measured to the nearest 0.05 ml.

5.5.1.8. Calculation and Report

Calculate the percentage water content by volume as follows: Water Content, % Volume = $\frac{V}{50} \times 100$,

where V = total volume of water distilled in ml.

If any water soluble substance is distilled in addition to water, then this result shall be reported as the Apparent Water Content.

5.5.1.9. Estimation of True Water Content

Where a fluid contains an appreciable quantity of a water soluble substance, e.g. ethylene glycol. which is distilled during the test, the Apparent Water Content will be greater than the True Water Content. In such cases the fractional concentration by volume of the water in the distillate shall be measured by a suitable method. The True Water Content shall then be found by multiplying the Apparent Water Content by the fractional concentration of water in the distillate.

In the case of fluids containing ethylene glycol for example the concentration of water in the distillate may be found from measurements of refractive index. The total distillate obtained during the distillation, including the carrier liquid remaining in the trap, shall be transferred to a separating flask. The flask shall be agitated and then left undisturbed whilst the contents separate. A sample of the aqueous phase shall be drained from the separating flask and its refractive index measured. The concentration of water in the distillate is then found graphically from measurements of refractive index of solutions of ethylene glycol of known concentration. A method based on specific gravity of the distillate is also satisfactory. The ethylene glycol content may be estimated as the difference between the Apparent Water Content and the True Water Content.

5.5.1.10. Acceptance Criteria

The manufacturer shall state the water content of type HFB and type HFB..LT fluids as a percentage by volume when measured by the above method. When the fluid is tested in accordance with this method the mean of two determinations of water content shall be within ± 1.5 of the stated percentage value.

The manufacturer shall also state the True Water Content of the fluid, and the chemical composition and concentration of any substance in the fluid which will lead to a difference between the True Water Content and the Apparent Water Content as defined in section 5.5.1.8.. In such cases the manufacturer shall submit a method or methods for the measurements of water content.

PART V 5.5.1.

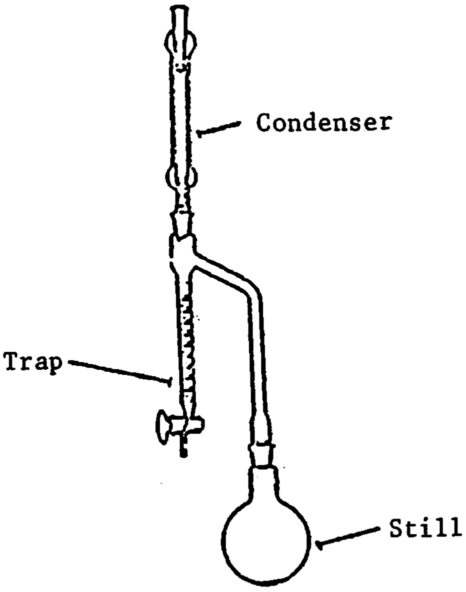


Figure 1 Distillation apparatus

5.5.2. METHOD OF TEST FOR WATER CONTENT OF TYPE HFC FLUIDS

Note : As an alternative to the method described below, the water content of type HFC fluids may be measured by any other Karl Fischer titration procedure of comparable accuracy, suitably modified to permit the direct addition of a fluid sample from a micrometer syringe. The methods which may be used for this purpose include ISO R766, AFNOR NFT 20-052, BS 2511, DIN 51777 and ASTM D1744-64. It is recommended that the test sample of 100 μl should be used in all cases.

5.5.2.1. Scope

The method shall be used for the measurement of water content of type HFC fluids only.

5.5.2.2. Principle

A small volume of the sample is titrated with standard Karl Fischer reagent to an electrometric end point.

5.5.2.3. Apparatus

a) The titration assembly shown in figure 1 comprising the following:

- i) Titration vessel - a three neck flask with a drain tap, approximate capacity 100 ml.
- ii) Burette - 25 ml capacity graduated in 0.1 ml sub-divisions and fitted with a two-way stopcock.
- iii) Reagent reservoir - a glass bottle of approximate capacity 500 ml.
- iv) Drying tubes - filled with a suitable dessicant. Anhydrous magnesium perchlorate is recommended.

b) The electrical circuit, also shown in figure 1 comprising the following:

- i) Microammeter - a dc microammeter with a range of 0 - 100 μA and an internal resistance of approximately 2000 ohms.
- ii) Battery - a 1.5 V dry cell.
- iii) Potentiometer - with a resistance of 10,000 ohms.
- iv) Electrodes - two platinum wires sealed into a glass tube. It is recommended that the diameter of the wires should be approximately 0.4 mm, the exposed length of each wire should be 5 mm, and that the wires should be spaced approximately 2 mm apart.

c) Compressed Nitrogen - a cylinder of compressed nitrogen fitted with a suitable regulator. The nitrogen is passed through a drying tube before entering the titration vessel.

d) Micrometer syringe - capable of dispensing 50 to 100 μl of liquid to an accuracy of ± 0.5 μl or better.

Note : Any other type of Karl Fischer apparatus may be used provided that it is substantially similar to the apparatus described.

5.5.2.4. Reagents

a) Karl Fischer reagent - with a water equivalent of approximately 5 mg water/ml.

b) Titration solvent - a mixture of equal volume of toluene and methanol.

5.5.2.5. Sampling

A sample of about 100 ml shall be supplied for test. Care shall be taken to ensure that the sample is representative of the bulk.

5.5.2.6. Procedure

The test shall be carried out in well ventilated conditions.

The potentiometer shall be adjusted so that a suitable meter reading of 10 to 50 μA is obtained with an excess of about 5 ml of Karl Fischer reagent present in 20 ml of titration solvent. The setting of the potentiometer shall then remain fixed for all subsequent tests.

The electrometric end point of a titration is reached when the addition of a single drop of reagent produces an increased deflection of the microammeter that is maintained for at least 30 s.

The burette readings shall be taken to the nearest 0.05 ml.

a) Standardisation of the Karl Fischer Reagent.

The Karl Fischer reagent shall be standardised daily, using the following procedure : 20 ml of titration solvent shall be introduced into the titration vessel from a pipette and the flow of nitrogen shall be adjusted to give steady agitation. The moisture present in the solvent shall be titrated with Karl Fischer reagent to an electrometric end point.

50 μl of distilled water shall then be introduced into the titration vessel from the micrometer syringe. The added water shall then be titrated with Karl Fischer reagent to an electrometric end point.

The water equivalent of the Karl Fischer reagent shall be calculated as:

$$F = \frac{50}{T_1} \text{ mg/ml}$$

where T_1 is the volume in ml of reagent used to titrate the added water.

The contents of the titration vessel shall then be drained off and the vessel shall be cleaned by rinsing through with titration solvent.

b) Determination of water content

20 ml of titration solvent shall be introduced into the titration vessel from a pipette and the flow of nitrogen shall be adjusted to give steady agitation. The moisture present in the solvent shall be titrated with Karl Fischer reagent to an electrometric end point.

100 μl of the type HFC fluid under test shall then be introduced into the titration vessel from the micrometer syringe. The fluid sample shall then be titrated with Karl Fischer reagent to an electrometric end point.

The quantity of water in the fluid sample shall be calculated as :

$$Q = T_2F \text{ mg}$$

where T_2 is the volume in ml of reagent used to titrate the fluid sample.

5.5.2.7. Results

The percentage water content by volume shall be the numerical value of Q and shall be reported to the nearest 0.1.

The volume of sample used for the determination and the test method reference shall also be reported with the test result.

5.5.2.8. Acceptance Criteria

The manufacturer shall state the water content of type HFC fluids as a percentage by volume, when measured in accordance with the above method. When the fluid is tested in accordance with this method the mean of two determinations of water content shall be within ± 2 mg of the stated value.

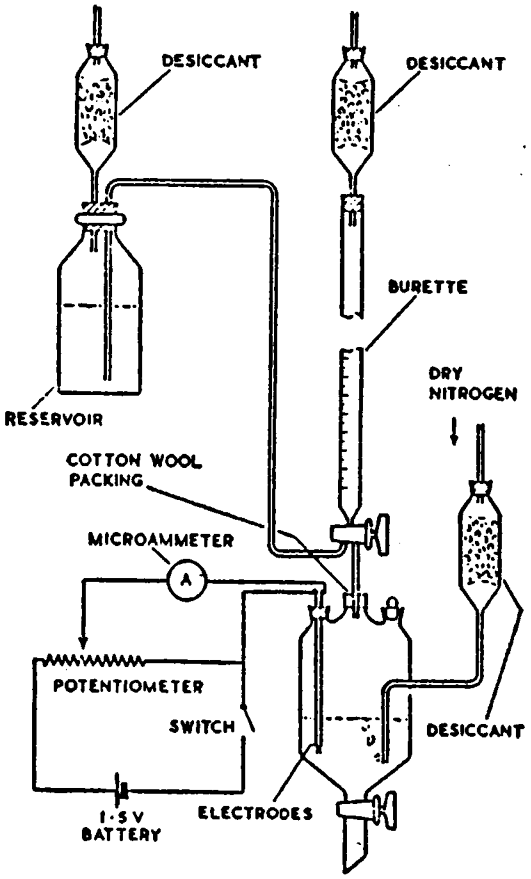


Figure 1 Apparatus for measurement of water content of type HFC fluids

5.6. METHOD OF TEST FOR AIR SEPARATION OR DEAERATION CAPACITY

Note : This method of test is based on the methods described in DIN 51381, AFNOR NFE 48-614, BS 5303, IP 313 and ASTM D3427.

5.6.1. Scope

The determination of the deaeration capacity of fire-resistant hydraulic fluids, i.e., the determination of the capacity of fluids to separate from the air contained therein. This method of test is applicable to all types of fluids except for those type HFA fluids with a kinematic viscosity of less than 10 cSt. This test is prescribed, however, for type HFC fluids only. For fluids of types HFA, HFB and HFD the test is discretionary.

5.6.2. Principle

Compressed air is introduced through a tube into the fluid which is contained in a test flask. This intensive mixing of the fluid with air causes it to become supersaturated with air. The time required for the bubbles to rise to the surface of the fluid is a measure of its deaeration capacity. In view of the fact that it is impossible to measure the surfacing speed of the bubbles by simple means, what is measured is the time which elapses before the air bubbles separate from the fluid sample. The time taken for the fluid to expel the dispersed air (in the form of air bubbles) to a level of 0.2% by volume is taken as the criterion of deaeration capacity.

5.6.3. Apparatus

a) Filter containing, for example, silica gel to eliminate the oil and other impurities contained in the compressed air introduced into the test flask.

b) Heating device for preheating the compressed air.

c) Thermometer correct to 0.1°C and graduated in 0.1°C intervals enabling readings to be taken from 0 to 100°C.

d) Pressure gauge to enable pressures to be measured in the range 0 to 0.35 bar (0 to 35 kPa).

e) Test flask with air inlet tube in accordance with figure 1.

f) Density balance to measure the density of the fluid correct to 0.001 g/cm^3 ; the sinker having a dispacement volume of 5 ml and a length of 80 ± 1.5 mm.

g) Thermostatically controlled circulating bath enabling the temperature to be regulated to 25 ± 0.1°C.

5.6.4. Reagents

a) Carbon tetrachloride, see Note (1);

b) Chrome sulphuric acid; and

c) Acetone.

5.6.5 Preparation

Type HFA fluids are prepared for test using one of the standard test waters (normally the hardest of the test waters with which the manufacturer has declared the emulsifying oil to be compatible), see section 2.2.2.6. The proportion of emulsifying oil in the emulsion shall be that recommended by the manufacturer for normal applications or such other value as may be specified by the approval/accepting authority.

The apparatus is assembled in accordance with figure 2. The source of the compressed air may be either a compressed air circuit or a cylinder of compressed air. Before entering the test flask, the air must be heated to the test temperature by a heating device.

The interior of the test flask and the air inlet tube must be cleaned thoroughly before each test. Traces of type HFD fluids should be removed by rinsing with carbon tetrachloride, see Note (1), while traces of types HFA, HFB and HFC fluids should be removed with water or a detergent solution.

Further cleaning is then required with chrome sulphuric acid, followed by successive rinses with distilled water, until no further traces of acid appear in the last rinse. The various parts of the apparatus are then rinsed with acetone and dried by means of compressed air which has been cleaned by the filter.

The test sample of fluid is placed in an air-bath at a temperature of approximately 35°C following which it is shaken vigorously. 180 ml of the heated sample are placed in the measuring flask and the thermostat is regulated so as to heat the fluid to 25 ± 1°C for types HFA, HFAS or HFB fluids and 50 ± 0.1°C for types HFC or HFD fluids. The temperature must be monitored by a thermometer.

When a temperature of 25°C or 50°C as appropriate, is reached the sinker of the density balance is introduced into the fluid, taking care that no air bubbles adhere to it and that its lower edge remains 10 ± 2 mm above the bottom of the test flask. When the sinker reaches the same temperature as the fluid, the density of the fluid is measured and the value is recorded as P_0 (initial value of the density of the fluid containing no air bubbles). The sinker is then withdrawn and placed in a bath maintained at the test temperature (e.g. in a glass receptacle placed in the circulating bath).

5.6.6. Procedure

The air inlet tube is fitted to the test flask and the tubes are connected as shown in figure 2. The air outlet tube should be attached to a discharge pipe. After a five minute wait, the air flow is started. The control valve is adjusted so as to give a pressure reading on the pressure gauge of 0.2 bar, and this pressure is maintained for seven minutes, further adjustments being made as required.

At the end of seven minutes, the air inlet is closed, the connectors removed, and the air inlet tube removed quickly from the test flask. The

Note (1) The use of carbon tetrachloride is not permitted in the laboratories of some countries. In such cases, the procedures should be carried out using methyl di-chloride.

sinker is then introduced immediately into the fluid, taking care to maintain a distance of 10 ± 2 mm between its lower edge and the bottom of the test flask. The first measurement of fluid density p_1 must be carried out no later than one minute after stopping the air flow.

Subsequent measurements p_n of the density should be made at one minute intervals but no further measurements are required after the density reaches the initial value p_o for the fluid containing no air. When the separation time reaches fifteen minutes, the time interval between measurements may be increased to five minutes.

5.6.7. Results

The amount of air L_n dispersed in the fluid after n minutes (n = 1, 2, 3, etc.) expressed as a percentage by volume may be calculated by the following formula:

$$L_n = \frac{100\ (Po - Pn)}{Po - Pa}$$

where: Po is the density of the fluid containing no air bubbles, in g/cm^3;*

Pn is the density of the fluid containing air after n minutes (n = 1, 2, 3...), in g/cm^3;

Pa is the density of air under the test conditions (temperature and pressure) in g/cm^3.

After being calculated in this way, the air content of the fluid, expressed as a percentage by volume, is plotted on a graph against the time which has elapsed since the inflow of air was stopped.

The deaeration capacity of the fluid is then established from the graph by determining the time-lapse between stopping the inflow of air and the point where the volume of air dispersed in the fluid falls to the level of 0.2% by volume.

The result of the test should be expressed in minutes and rounded to the nearest whole number.

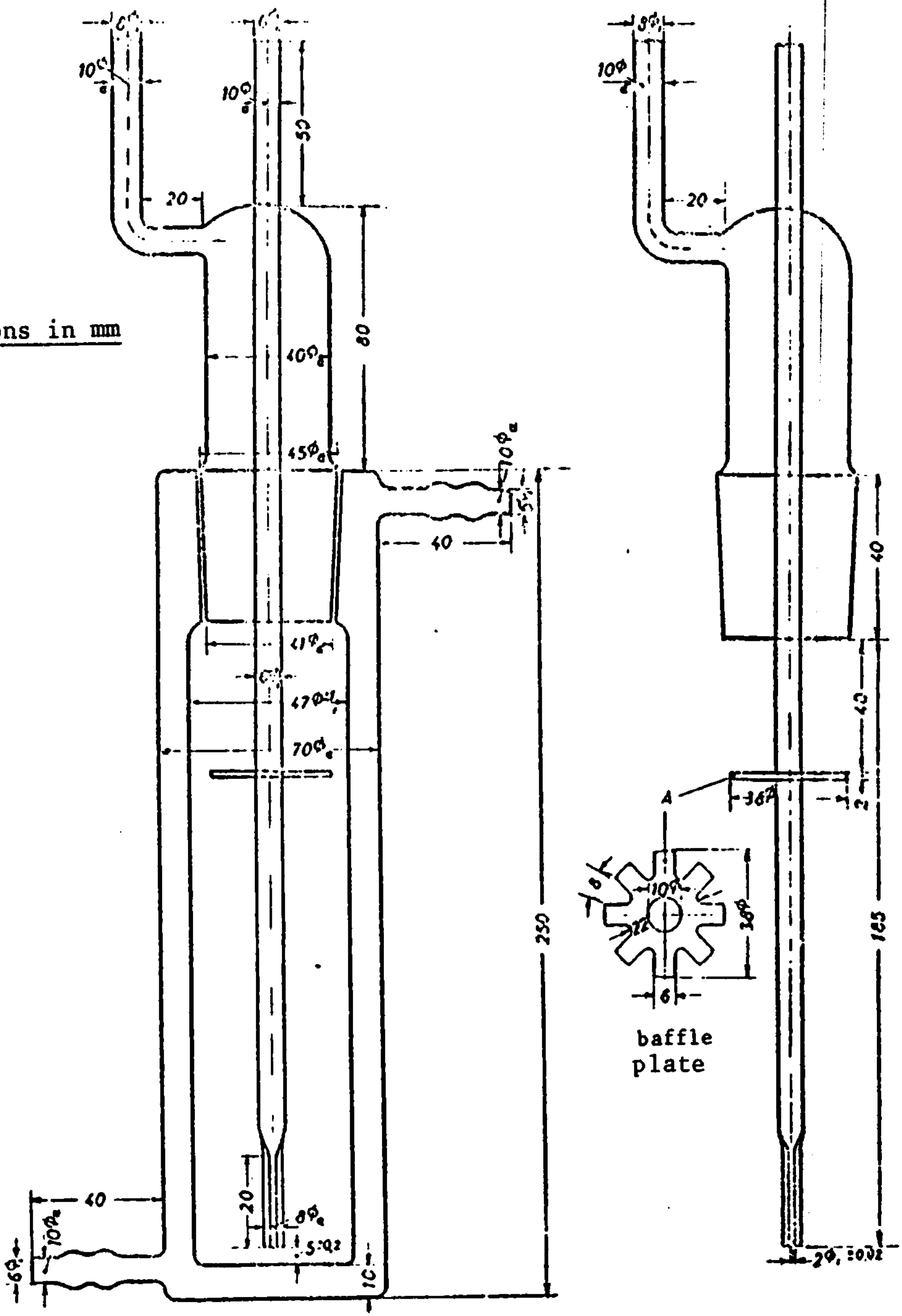
5.6.8. Acceptance criteria

This test is carried out for information purposes only on fluids of types HFA, HFB and HFD and no acceptance criteria are specified for these fluids.

For type HFC fluid the deaeration capacity, as determined by the above method of test, should not exceed 25 minutes.

* Note In the case of HFC fluids a loss of water from the test fluid may be excepted following the inflow of air. This result in a change of the air free fluid density before and after the test. Po should therefore always be taken as the before test density.

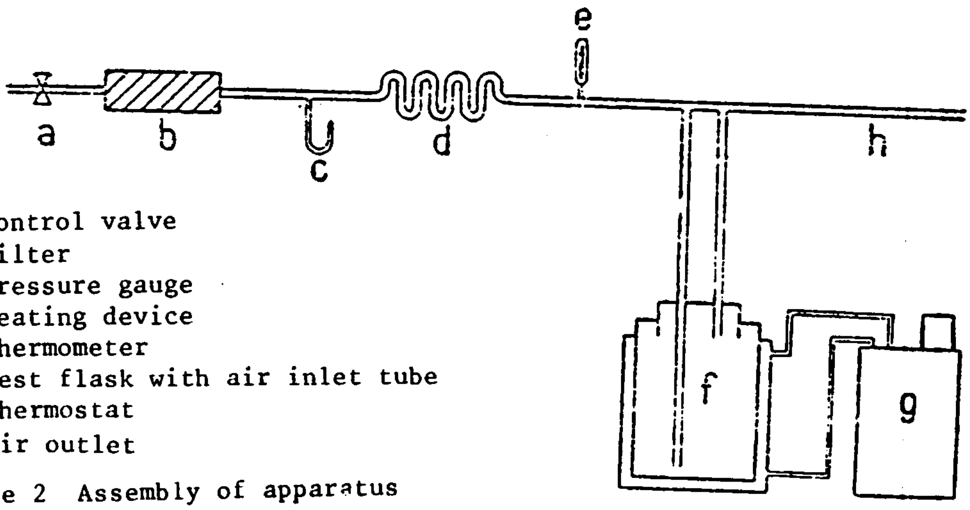
All dimensions in mm



Test flask Test vessel (impinger)

Air inlet pipe
Air inlet tube

Figure 1 Test flask and air inlet tube



a - control valve
b - filter
c - pressure gauge
d - heating device
e - thermometer
f - test flask with air inlet tube
g - thermostat
h - air outlet

Figure 2 Assembly of apparatus

5.7. METHOD OF TEST FOR FOAMING CHARACTERISTICS

Note : This method of test is based on the test procedure which is described in AFNOR NFT 60-129, ASTM 892-79, BS 5092 - 1974 and IP 146/80.

5.7.1. Scope

This method of test is applicable to fluids of types HFA, HFC and HFD. (Type HFA fluids are only tested if the kinematic viscosity exceeds 10 cSt).

5.7.2. Principle

The determination of the volume of foam produced when a well diffused air-current is passed through the test fluid at different temperatures.

5.7.3. Apparatus

a) Measuring cylinder of 1,000 ml capacity. The volume of 1,000 ml should correspond to a length of 365 ± 15 mm of the graduated scale on this cylinder.

b) Air inlet tube with an alumina diffusion sphere (Norton Company, Refractories Division, Worcester, 6, Mass., USA, reference No ME-46239, Grade P 2180 fine).

The air inlet tube should be made of brass and should measure at least 450 mm in length. A porous diffusion sphere, diameter 25 mm, made of compacted aluminium oxide is attached to the lower end of this inlet tube.

c) Thermostatically controlled bath consisting of a glass vessel of water, equipped with a heating system, an automatic temperature regulating device and a stirrer. The glass vessel must be sufficiently large to allow the measuring cylinder to be immersed in it as far as the 900 ml level on the graduated scale. It must be possible to regulate the temperature of the bath to 25 ± 0.5°C, 50 ± 0.5°C and 100 ± 0.5°C.

d) Flow meter for measuring the air flow : normal rotameters or manometer type flowmeters (consisting of a U tube partly filled with liquid which measures the pressure difference across a capillary tube placed in the air current) can be used. (There should be a narrow constriction in the lower part of the U tube to reduce oscillations of the liquid due to pressure fluctuations)..

e) Stop watch.

f) Thermometer such as type IP 64 C with scale graduated from 0 to 100°C in 0.2°C graduations.

5.7.4. Chemicals

a) Petroleum ether or heptane.

b) Chemically pure acetone.

c) Pure carbon tetrachloride, see Note (1).

5.7.5. Preparation for test

a) In order to remove all traces of fluid used in previous tests, which may invalidate the results, it is essential to clean the apparatus thoroughly.

This compulsory cleaning procedure refers particularly to :

i) The measuring cylinder ; this must be thoroughly cleansed with petroleum ether or heptane, then with the acetone and finally with distilled water. It should then be dried by means of a clean, oil-free air current.

ii) The diffusion sphere attached to the air inlet tube ; this sphere must be washed with petroleum ether or heptane once again. It must be immersed in approximately 300 ml of each of the above solvents, which are drawn in under vacuum and expelled under pressure (five times for each solvent). The diffusion sphere is then placed in carbon tetrachloride (1), rinsed with acetone and dried in an oil-free air current. The tube must then be wiped with a clean dry cloth.

b) The air inlet tube is then inserted through one of the two holes in a rubber stopper which is fitted to the measuring cylinder. The diffusion sphere must just touch the bottom of the measuring cylinder.

c) At this point, approximately 200 ml of the fluid sample are poured into a clean glass container and heated to a temperature of 50 ± 2°C and allowed to cool to a temperature of 25 ± 2°C. The water bath is then also heated to 25 ± 0.5°C. The prepared sample is then poured into the measuring cylinder up to the 190 ml level and is then placed inside the bath so that the water level reaches at least the 900 ml level.

d) Type HFA fluids are prepared for test using one of the standard test waters (normally the hardest of the test waters with which the manufacturer has declared the emulsifying oil.

5.7.6. Procedure

a) Once the fluid in the measuring cylinder reaches the temperature of the bath, the rubber stopper into which the air inlet tube has been inserted is fitted to the measuring cylinder (the diffusion sphere just touching the bottom of this cylinder). A period of 5 minutes is allowed to elapse whilst the diffusion sphere becomes saturated with the fluid. On completion of this operation, the tube is connected to the air inlet and an air flow of 94 ± 5 ml per minute is established. (The air must be dry and oil-free and if this is not the case, then the air must pass through filters consisting of calcium chloride, activated carbon and wadding). A period of 5 min ± 10 s is then timed, starting from the moment when the first air bubbles appeared on the surface of the diffusion sphere, after which the air flow is cut off. The volume of foam produced, in ml, is then immediately measured - i.e. from the upper level of the foam (average reading) to the surface of the fluid below.

Note (1) The use of carbon tetrachloride is not permitted in the laboratories of some countries. In such cases, the procedures should be carried out using methyl dichloride.

The air inlet tube is not removed and the measuring cylinder is left in the water bath for a further 10 min ± 10 s after which the volume of foam is measured once again.

b) A second test at a higher temperature is carried out on a fresh sample of fluid as follows:

For fluids of types HFA and HFC the temperature used for the test is 50°C and for type HFD fluids the temperature used is 100°C.

The preparations are identical to those previously described but the temperature of the bath is raised to 50 ± 0.5°C and 100 ± 0.5°C, respectively.

The air current is introduced as before by means of the air inlet tube equipped with a diffusion sphere which has been freshly cleaned.

The measurement is carried out as soon as the requisite blowing time or settling time, as appropriate has elapsed.

c) The foam remaining after the previous test (Sub-section (b) above) is then collapsed by gentle stirring. The fluid is then cooled to a temperature of below 40°C by exposing the measuring cylinder to room temperature and the air inlet tube, which has been previously cleaned for the purpose, and to which a diffusion sphere has been attached, is then inserted. The cylinder is then placed in a bath maintained at 25 ± 0.5°C and when the fluid has reached the temperature of the bath the procedure described in Sub-section (a) above is repeated. Once the requisite blowing and settling times have elapsed the amount of foam is measured.

5.7.7. Results

The results of the tests described in the previous Section are presented in tabular form as follows:

Temperature of fluid sample	Volume of foam measured immediately after the air flow is stopped	Volume of foam measured after a settling time of 10 minutes
25°C	.... ml	.... ml
50°C (or 100°C)	.... ml	.... ml
then at 25°C following the test at 50°C or 100°C	.... ml	.... ml

5.7.8. Acceptance criteria

The volume of foam measured by the above method of test should not exceed the following values :

Immediately after the air flow is stopped	350 ml
Immediately after 10 min settling period	15 ml

5.8. METHOD OF TEST FOR SHEAR STRENGTH OF FLUIDS

5.8.1. Scope

The method is used to determine the mechanical shear strength of fluids used in hydraulic applications. It can be used for all types of fluid, with the exception of those type HFA fluids which have a viscosity of less than 10 cSt at 20°C. The test is a discretionary test for type HFA fluids.

5.8.2. Principle

A defined volume of fluid is passed through an injector nozzle a specified number of times. The viscosity, flow temperature, and pH value (neutralisation number) for all types of fluid and the water content for type HFA, HFB and HFC fluids are determined before and after the test to establish the magnitude of any changes.

5.8.3. Apparatus and solvents

The apparatus is as shown in figure 1 and comprises :

a) An injector system consisting of a Bosch injector Type KD 43 SA/53/13 with a spray nozzle of Type DN 0 SD 211. The nozzle is to be adjusted to 100 bar;

b) Glass containers (3 and 3a) with loose fitting lids;

c) Three-way cock and hoses to connect the container (3a) to the injection pump;

d) Stop watch;

e) Beaker (400 ml);

f) Solvent to clean the glass components:

For HFB fluid a mineral based gasoline is suitable, for HFA and HFC fluids water should be used and for HFD fluids the solvent should be trichlorethylene or monochlorbenzene. Pure ethyl alcohol should be used for drying after cleaning with water.

5.8.4. Preparation for test

a) 250 ml of the test fluid is poured into the container (3) with the three-way cock in the indicated setting (6).

b) The control screw (13) is unscrewed to allow air to escape from the pump (14) ; the screw (13) is tightened once the fluid starts to flow steadily.

5.8.5. Procedure

a) Start the motor.

b) Start the stop watch immediately the fluid starts to flow through the control tube (2).

c) Using the manometer (11) check that the pressure is effectively between

100 and 110 bar, by using the cock (12). This must be closed after checking.

d) After operating 30 minutes, corresponding to at least 50 passages of the fluid, the motor is stopped. The cock is placed in indicated setting (5) and the fluid is collected in a clean Pyrex glass beaker (normally the temperature of the fluid collected in this way is more than 55°C). The motor is switched on again for a few moments to completely evacuate this circuit, until such time as no further flow occurs through tube (2).

N.B. Throughput in tube 8 should not be excessive. Appreciable runback of fluid through tube 8 when testing viscous fluids (120 cSt at 50°C) has been observed.

5.8.6. Cleaning the apparatus

a) Cleaning the fluid circuit. As the apparatus cannot be taken to pieces, 100 to 150 ml of the test fluid is allowed to circulate for about 10 min before each test for cleaning purposes. If fluids from the same group are to be tested in sequence, a single flush will be sufficient. If the fluid to be tested differs from that in the preceding group, then the apparatus must be flushed twice over. The fluid used for cleaning purposes must be drained completely, each time.

b) Cleaning the containers and hoses. This is done by means of solvents and products compatible with the test fluids (section 5.8.3).

5.8.7. Results

The following information is required after tests on samples of fresh fluid and on samples of fluid after is has been subjected to the shear test.

Viscosity :	for type HFA fluid at		20°C	and 50°C	
	for type HFB fluid at	0°C	20°C	and 50°C	
	for types HFC and HFD fluid at	0°C	20°C	50°C	and 100°C

Flow temperature for all types of fluid

pH value .... for types HFA and HFC fluids

Neutralisation number .... for types HFB and HFD fluids

Water content .... for types HFA, HFB and HFC fluids.

Note : The maximum tolerance on the measured viscosities is ± 3%.

5.8.8. Acceptance criteria

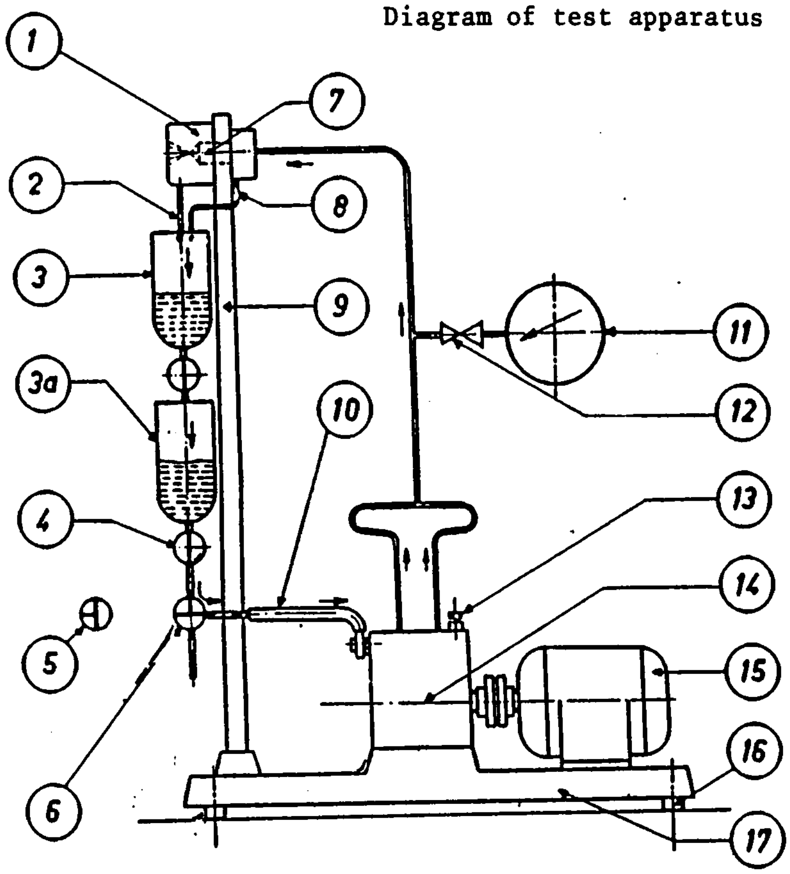
The differences between the values relating to tests on the samples of fluid subjected to the shear test and the corresponding values obtained from samples of fresh fluid shall not exceed the limits given in the following Table. The limits relating to the permissible differences in other properties are also given in the Table.

Characteristics measured	Differences in properties for fluid of type			
	HFA(1)	HFB	HFC	HFD
Kinematic viscosities at :				
0°C	-	± 15%	≤ + 25%	≤ ± 20%
+ 20°C	≤ ± 10%	≤ ± 15%	≤ ± 15%	≤ ± 10%
+ 50°C	≤ ± 10%	≤ ± 15%	≤ ± 15%	≤ ± 7%
+ 100°C	-	-	-	≤ ± 7%
Pour point - see Note (2)	-	-	-	-
pH	≤ ± 0.5	-	≤ ± 1	-
Neutralization number (mg KOH/g)	-	≤ ± 0.5	-	≤ ± 0.5
Reduction in water content, %	≤ 15	≤ 5	≤ 8	-

(1) Except type HFA fluids with a viscosity less than 10 cSt at 20°C.

(2) The test for pour point is a discretionary test, see section 6.4, for which no acceptance criteria are specified. The acceptable differences should be declared by the manufacturer or specified by the approving/accepting authority.

Diagram of test apparatus



Legend :

1	Atomizing chamber	10	Connection to pump intake
2	Outlet for atomized fluid	11	Pressure gauge, 0/250 bar
3	600 ml glass container with cover	12	Pressure gauge tap
3a	-ditto-	13	Valve to evacuate air from pump
4	Stop cock	14	Two-cylinder injection pump e.g. Bosch PE 2 B/100 or PE 2 A900 300/3 S 226
5	3-way cock at the end of test	15	Electric motor, 1.1 kW, 920 r.p.m.
6	3-way cock during test	16	Rubber buffers
7	Injector, set to 100 bar	17	Base-plate
8	Excess fluid return		Direction of flow of test fluid
9	Support		

Figure 1 Shear strength test

5.9. METHOD OF TEST FOR CORROSION INHIBITING PROPERTIES OF FLUIDS

Note :

It is important that fire-resistant hydraulic fluids do not cause corrosion of the equipment in which they are used. The tests described in this section are designed to determine the corrosion inhibiting properties of the fluids in contact with selected metals.

Four methods of test are described. The first (section 5.9.1) is based on the method of test specified in the Fifth Report and is characterised by a low test temperature (35°C) and a long duration of test (28 d). The second method (section 5.9.2) is very similar in its procedure but it employs a higher test temperature (60°C) for a shorter period (14 d). The third test is particularly intended for application to emulsifying oil used to make-up type HFA fluids and provides a very rapid method of assessment of the corrosion inhibiting properties of such oils (24 h).

The fourth method of test (section 5.9.4) is not yet intended as an alternative to the two established methods (5.9.1 and 5.9.2) but is proposed for assessment as a harmonised test for universal adoption at a later date. It has many features in common with the existing methods and it is recommended that it is used in parallel with the existing methods of test so that laboratories may obtain comparative data which might lead to its early acceptance (see section 2.4.1).

5.9.1. METHOD OF TEST FOR CORROSION INHIBITING PROPERTIES (ALL FLUIDS)

Note : This method of test is based on that described in section 3.8. of the Fifth Report.

5.9.1.1. Scope

This method of test serves to determine the corrosion inhibiting properties of hydraulic fluids in contact with metals. The method of test can be applied to all types of fire-resistant fluids. In the case of type HFA fluids, the test is to be conducted with the made-up emulsion.

5.9.1.2. Principle

Specimens of materials are partly immersed in the test fluid for a fixed period of time. The change in weight of the specimens and changes in the surface condition of the material and in the colour of the test fluid are noted.

5.9.1.3. Apparatus

a) Glass beakers, 400 ml capacity, without pouring lip and with a height of about 135 mm (tall type).

b) Watch glasses to cover the beakers, with an opening in the middle for a lifting hook.

c) Glass hooks from which the specimens can be freely suspended in the beakers.

d) Thermostatically controlled heating bath which can maintain the temperature of the test fluid in the beakers at 35 ± 1°C. The bath must be provided with a stirring device to ensure uniform temperature distribution throughout the bath. A drying cupboard with similar temperature regulation may be substituted for the heating bath.

e) An analytical balance with a sensitivity of 0.2 mg or better.

f) Forceps with rounded ends and smooth faced internal gripping surfaces.

5.9.1.4. Materials

a) Test specimens, each 100 mm long, 20 mm wide and about 1 mm thick, with a 4 mm diameter hole near the edge of one short side so that the specimen can be hung on a glass hook, and cut from the following materials:

i) steel with the following composition :

C	0.35 to 0.45%
Mn	0.5 to 0.8%
Si	0.35% max.
S	0.035% max.
P	0.035% max.

ii) Electrolytic copper.

iii) Pure zinc.

iv) Pure aluminium (at least 99.5%).

v) Pure cadmium (where required).

vi) Brass (63% Cu and 32% Zn).

vii) Other materials as may be considered necessary.

b) Emery papers of decreasing roughness (P.120; P.240; P.400; P.600).

c) Cotton wool.

d) Acetone and n-heptane.

e) Toluene.

f) 96% ethyl alcohol.

5.9.1.5. Preparation

The specimens should be prepared using emery papers of decreasing roughness to give the best possible finish. The specimens are then held with the forceps and cleaned with dry cotton wool followed by cotton wool soaked in n-heptane. The traces of cotton wool remaining on the specimens are then washed off with pure n-heptane. As soon as the last traces of solvent adhering to the specimens have evaporated, the specimens are weighed on the analytical balance. They must then be used immediately for the corrosion tests.

5.9.1.6. Procedure

At least 11 beakers are needed for a complete series of tests, each of which is filled with 250 ml of the test fluid. The beakers are placed in the thermostatically controlled heating bath or drying cupboard which is adjusted to maintain a temperature of 35 ± 1°C in the test fluid in the beakers.

Six of the beakers are provided with one each of the following specimens, prepared as described in section 5.9.1.5 and suspended from the glass hook in such a way that approximately 60 mm of the specimen is immersed in the test fluid (see figure 1):

steel;
copper;
zinc;
aluminium;
cadmium; (where required)
brass.

In addition, in order to test the behaviour of the test fluid in the presence of two metals, the following pairs of specimens are immersed in four of the remaining beakers:

steel and cadmium (where required);
copper and zinc;
aluminium and zinc;
steel and aluminium.

The two specimens forming each pair should hang in the beaker approximately 1 mm apart. It is the normal practice to connect the two specimens of each pair together electrically.

The eleventh beaker containing 250 ml of the test fluid in which no specimen is suspended serves to check any precipitation which may occur in the fluid itself during the period of the test.

Care must be taken throughout the test to maintain a constant temperature in the heating bath or drying cupboard and the test should be run for a minimum period of 28 days.

At the end of the test period, the changes in the surface condition of the specimens such as oxidation colours, corrosion and deposits, must be observed. Also, for each specimen, a record must be made of the colour and appearance of the test fluid and of any deposits in the fluid. After this visual examination, each test specimen must be gripped in the vicinity of the hole with the forceps and washed to eliminate traces of the test fluid. In the case of types HFA and HFB fluids the test pieces should be immersed in a beaker filled with n-heptane and then in a beaker of acetone, each immersion being for a period of 1 minute. The test pieces should be waved in air, always using the forceps, until the surface appears dry. If after this first washing operation traces of the test fluid still remain on the metal surfaces the operation should be repeated with fresh solvents until all traces of moisture have been removed. The specimens should then be weighed on the analytical balance.

For type HFC fluids the same washing and drying method is employed but using distilled water and acetone while for type HFD fluids the specimens are washed in toluene alone.

5.9.1.7. Results

The following should be reported:

a) the test fluid identification;

b) the increase or decrease in the weight in mg of each specimen;

c) the visual effects produced by the fluid on each of the specimens;

d) the visual changes in the fluid in which each of the specimens was immersed.

5.9.1.8. Acceptance criteria

There should be no perceptible corrosion of the test specimens.

The reduction in the weight of the specimens must not exceed:

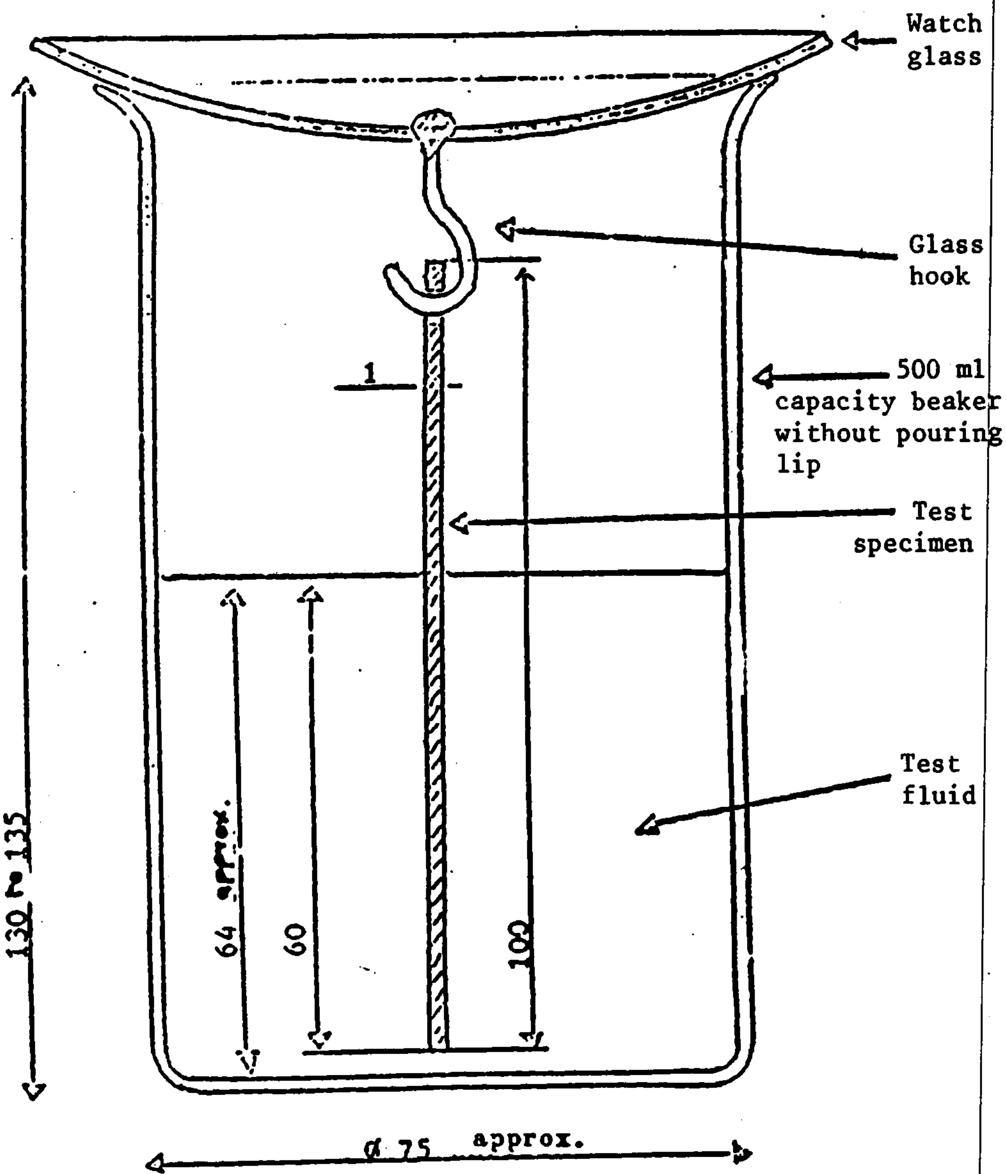
for cadmium and zinc	22 mg;
for steel, copper and brass	11 mg; and
for aluminium	5.5 mg.

Exceeding the above limit for cadmium and zinc, shall not automatically eliminate the fluid but the accepting/approving authority should be notified of the result.

Normally there should be no deposit observable, with the naked eye on the test specimens. However, provided this does not exceed 5 mg, the fluid may be accepted/approved.

The surfaces of the specimens which were immersed in the fluid must not show any appreciable change in colour nor should the surfaces of the specimens outside the fluid in the vapour phase. If there is a change of colour, the reasons must be investigated.

PART V 5.1.1.



Dimensions in mm

Figure 1 Position of test strip in the beaker

5.9.2. METHOD OF TEST FOR CORROSION INHIBITING PROPERTIES (ALL FLUIDS)

Note : This method of test is based on that described in the "Supplementary test methods for fire-resistant fluids" of the National Coal Board (United Kingdom). This test method is similar in many respects to that described in section 5.9.1 but for a higher test temperature and a lower duration of test.

5.9.2.1. Scope

This method of test serves to determine the corrosion inhibiting properties of hydraulic fluids in contact with metals. This method of test can be applied to all types of fire-resistant fluids.

5.9.2.2. Principle

Specimens, representing a range of metals, are partially immersed in the fluid under test in closed containers and are maintained at an elevated temperature. After a period of time the specimens are examined for evidence of attack in the liquid phase, the vapour phase and the interface.

5.9.2.3. Apparatus

a) Boiling tubes.

b) Tube closures.

c) Thermostatically controlled oven, suitable for maintaining specimens at a temperature of 60 ± 1°C, and fitted with a glass door.

d) Mercury-in-glass total immersion thermometer, with a range of 45 to 105°C and an accuracy of 0.2°C.

e) Cork bung to fit boiling tube and with a central hole to accomodate the thermometer.

f) Analytical balance for weighing to within 0.1 mg.

g) Desiccator.

h) A stiff nylon or bristle brush.

5.9.2.4. Materials

a) Specimens, 100 mm long and 25 mm wide and of convenient thickness, cut from the following materials:

  i) mild steel sheet having the following chemical composition (BS 1449 En 2A/1):

  C 0.10% max.
  Mn 0.50% max.
  S 0.04% max.
  P 0.05% max.

  ii) zinc-electroplated mild steel;

  iii) cadmium-electroplated mild steel (not passivated);

  iv) copper sheet, commercial grade;
  v) brass sheet, commercial grade;
  vi) aluminium sheet, commercial grade;
  vii) other materials as required.

b) Emery paper, Grade 0

c) Magnesium oxide powder

d) Petroleum spirit

e) Glycerol

f) Cotton wool

g) Test fluid

h) A solvent appropriate to the test fluid.

5.9.2.5. Preparation

Resurface the steel, copper, brass and aluminium specimens (two of each material) by hand polishing with emery paper. Resurface the zinc plated and cadmium plated specimens (two of each material) by lightly abrading with magnesium oxide powder applied on a moist cloth. Thoroughly clean all specimens by swabbing with petroleum spirit soaked cotton wool.

Weigh each specimen, taking care to avoid fingering the surfaces, and store the specimens in a desiccator for 24 h before test.

Mount the thermometer and cork bung in one of the boiling tubes into which glycerol has been introduced. The thermometer bulb should not touch the bottom of the tube and the level of the glycerol should be well above the 60°C scale marking on the thermometer stem.

5.9.2.6. Procedure

Place each specimen in a boiling tube in which a quantity of test fluid has been introduced. The quantity should be such that approximately four-fifths of the specimen is immersed when it rests on the bottom of the tube. Close each tube.

Place the boiling tubes, including the one containing the thermometer, close together in the oven. Position the tubes so that the temperature can be read from the thermometer. Maintain the specimens at a temperature of 60 ± 1°C for a period of 14 days.

At the end of the test period, remove the specimens and wash in a solvent appropriate to the type of fluid under test. Allow the specimens to dry. Visually examine each specimen and the test fluid in which it was immersed.

Clean any specimens to which an obvious corrosion product adheres, by means of a stiff nylon or bristle brush and to appropriate solvent, and allow the specimens to dry. Do not clean specimens that are tarnished only. Weigh each specimen.

5.9.2.7. Results

The following should be reported:

a) the test fluid identification;

b) the increase or decrease in weight (mg) of each specimen;

c) the visual effects produced in each specimen by the test fluid; and

d) the visual changes in the fluid in which each of the specimens was immersed.

5.9.2.8. Acceptance criteria

Precise limits cannot be defined for the acceptability of a fluid-metal combination, but judgement can be made on the basis of the result. Unsuitable combinations of fluid and metal will be indicated by obvious corrosion product, localised attack, appreciable change in the fluid appearance, or large increases or decreases in weight (more than about 20 mg). Tarnish or uniform attack giving small weight changes are acceptable.

5.9.3. METHOD OF TEST FOR CORROSION INHIBITING PROPERTIES OF OILS USED FOR THE PREPARATION OF HFA FLUIDS

Note : This method of test is based on IP 135/64 "Rust-preventing characteristics of steam turbine oils in the presence of water" but in this test 330 ml of an emulsion of the oil at a concentration of 2 per cent by volume made up in a 0.05M (2.9 g/l) solution of sodium chloride in distilled water is used in place of the mixture of lubricating oil and water. It is also technically equivalent to the methods of test specified in ASTM D665-60, BS 4387, DIN 51 585 and ANS Z11 85.

5.9.3.1. Scope

This method of test is intended to indicate the ability of emulsifying oils to form emulsions that do not cause rusting on immersed ferrous surfaces. Its application is particularly directed to those emulsifying oils used to prepare type HFA fluids for use in powered roof supports.

5.9.3.2. Principle

The method involves stirring an emulsion of the oil under test in a solution of sodium chloride at a temperature of 60°C for 24 h with a cylindrical steel specimen completely immersed therein.

5.9.3.3. Apparatus

The apparatus shall consist of the following:

a) Oil Bath.

A thermostatically controlled liquid bath capable of maintaining a temperature in the oil sample of 60 ± 1°C. An oil of ISO viscosity grade 32 is suitable for the bath. The bath shall have a cover with holes to accomodate the test beakers.

b) Beaker.

A 400-ml, Berzelius type, tall-form heat resistant glass beaker without pourout, as shown in figure 1, approximately 127 mm in height measured from the inside bottom centre and approximately 70 mm in inside diameter measured at the middle.

c) Beaker Cover.

A flat beaker cover of glass or methyl methacrylate resin (Note 1), kept in position, by suitable means such as a rim or groove. Two holes shall be provided on any diameter of the cover : one for a stirrer 11.70 to 12.17 mm in diameter with its centre 6.1 to 6.6 mm from the centre of the cover ; and the other on the opposite side of the centre of the cover, for the test specimen assembly (section 5.3.9.4), 17.6 to 18.1 mm in diameter with its centre 15.62 to 16.12 mm from the centre of the cover. In addition, a third hole 11.70 to 12.17 mm in diameter shall be provided for a thermometer, with its centre 21.9 to 22.5 mm form the centre of the cover and on a diameter of the cover at right angles to the diameter through the other two holes.

Note 1 : An inverted Petri dish makes a suitable cover, as the sides of the dish aid in keeping it in position. Figure 2 shows a methyl methacrylate resin cover for the beaker which has been found to be suitable. An optional feature is shown, consisting of a slot, 1.6 mm by 27 mm, which is centred on a diameter of the stirrer hole at right angles to the cover diameter through the specimen hole and stirrer hole. This feature allows withdrawal of the stirrer while the beaker cover is in place.

d) Stirrer.

A stirrer constructed entirely from stainless steel (Note 2) in the form of an inverted T. A flat blade 25 x 6 x 0.6 mm shall be attached to a 6 mm rod in such way that the blade is symmetrical with the rod and has its flat surface in the vertical plane.

Note 2 : If stainless steel is not obtainable, stirrers made of heat-resistant glass and having approximately the same dimensions as the stainless steel stirrers specified may be used.

e) Stirring Apparatus.

Any convenient form of stirring apparatus capable of maintaining a speed of 1,000 ± 50 rev/min.

f) Grinding and Polishing Equipment.

A 150 and 240 grit metal-working aluminium oxide cloth closed coat on a jeans backing; suitable chuck (see figure 4) for holding the specimen ; and a means of rotating the specimen at a speed of 1700 - 1800 rev/min.

5.9.3.4. Test specimen

a) The specimen assembly shall consist of a round steel specimen fitted to a plastic holder. The plastic holder shall be made of methyl methacrylate resin in accordance with the dimensions shown in figure 3. (Two types of holders are illustrated). The round steel specimen when new shall be 12.7 mm in diameter and approximately 68 mm in length exclusive of the threaded portion which screws into the plastic holder and shall be tapered at one end as shown in figure 3. It shall be made of steel having the following composition*:

C	0.25 max.
Mn	1.00 max.
Si	0.35 max.
S	0.060 max.
P	0.060 max.

The steel specimen, either new or from a previous test (Note 3), shall be prepared as described in paragraphs (b) and (c).

Note 3 : When making a check test, the steel specimen that showed rust should not be re-used. Specimens that repeatedly show rust in tests of various oils may be imperfect. Such specimens should be used with oil known to pass the test. If rusting occurs in repeat tests, these specimens should be discarded.

* Test specimens can be obtained from Stanhope-Sets Ltd., Station Road, Chertsey, Surrey, UK.

b) Preliminary Grinding - If the specimen has been used previously and is free from rust or other irregularities, the preliminary grinding may be omitted and it may be subjected only to final polishing as prescribed in Paragraph (c). If the specimen is new or if any part of its surface shows rust irregularities clean it with iso-octane or 60/80 petroleum spirit and grind with medium 150 grit metal-working aluminium oxide cloth to remove all irregularities, pits, and scratches, as determined by visual inspection (Note 4). Perform the grinding by mounting the specimen in the chuck of the grinding and polishing apparatus, and turning it at a speed of 1700 - 1800 rev/min while the 150 grit metal-working aluminium oxide cloth is applied. Old grit metal-working aluminium oxide cloth may be used to remove rust or major irregularities, but the grinding shall be completed with new cloth. Proceed at once with the final polishing with 240 grit metal-working aluminium oxide cloth, or remove the specimen from the chuck and store in iso-octane until needed. Discard re-used specimens when the diameter is reduced to 9.5 mm.

Note 4 : The specimen shall not be touched with the hands at any stage after cleaning with the petroleum spirit or iso-octane (which precedes either preliminary grinding or final polishing) until the test is completed. Forceps or a clean, lintless cloth may be used.

c) Final Polishing - Just before the test is to be made, subject the specimen to final polishing with 240 grit metal-working aluminium oxide cloth. If the preliminary grinding has just been completed, stop the motor which rotates the specimen. Otherwise, remove the specimen from the iso-octane (previously used unrusted specimens shall be stored, in this reagent) dry with a clean cloth and place in the chuck. Rub a new piece of 240 grit metal-working aluminium oxide cloth longitudinally over the static specimen until the rounded end, and the entire surface show visible scratches. Rotate the specimen at a speed of 1700 to 1800 rev/min and polish with a strip of 240 grit metal-working aluminium oxide cloth by wrapping it halfway around the specimen, and applying a firm but gentle downward pull to the loose ends of the cloth for about 1 to 2 minutes, so as to produce a uniform finely scratched surface free from longitudinal scratches. Carry out the final stage of the polishing with new cloth (Note 5). Remove the specimen from the chuck without touching with the fingers, wipe lightly with a clean, dry, lintless cloth or tissue (or brush the specimen lightly with a camel hair brush), attach to the plastic holder, and immediately immerse in the emulsion to be tested. This may be either the hot emulsion sample (section 5.9.3.6) or a clean test tube containing a portion of the sample. The specimen may be removed later from this tube and allowed to drain briefly before being placed in the hot emulsion.

Note 5 : To ensure that the flat shoulder (that portion of the specimen perpendicular to the threaded stem) is free of rust, this area should be polished. This may be done by holding a strip of 240 grit metal-working aluminium oxide cloth between the chuck and the shoulder while rotating the specimen for a brief period.

5.9.3.5. Test emulsion

Prepare a 0.05M (2.9 g/l) solution of sodium chloride in distilled water. Prepare an emulsion of 2% by volume of the emulsifying oil to be tested in the sodium chloride solution. 330 ml of this emulsion shall be used in the test. If the recommended concentration of the oil in service has a value other than 5%, then the test emulsion shall be made up at a concentration of 2/5 of this value.

5.9.3.6. Procedure

a) Clean the beaker with chromic acid cleaning solution, wash with distilled water to remove the acid completely, and dry in the oven. (Warning: chromic acid can cause burns). Clean a glass beaker cover and a glass stirrer by the same procedure. To clean a stainless steel stirrer and a methyl methacrylate resin cover, use iso-octane or 60/80 petroleum spirit wash thoroughly with hot water and finally with distilled water, and dry in an oven at a temperature not over 65°C for the cover. Pour 330 ml of the test emulsion into the beaker and place the beaker in the oil bath held at a temperature that will maintain 60 ± 1°C in the emulsion sample. The beaker shall be inserted into a hole of the bath cover and suspended in the hole with the beaker rim resting on the bath cover. The oil level in the bath shall not be below the emulsion level in the test beaker. Cover the beaker with the beaker cover with the stirrer in position in the proper opening. Adjust the stirrer so that the shaft is 6 mm off centre in the beaker containing the emulsion sample and the blade is within 2 mm of the bottom of the beaker. Then suspend a thermometer through the hole in the cover intended for that purpose so that it is immersed to a depth of 57 mm. Start the stirrer and when the thermometer reading reaches 60 ± 1°C insert the steel specimen prepared in accordance with section 5.9.3.4.

b) Insert the test specimen assembly through the specimen hole in the beaker cover and suspend so that its lower end is 13 to 15 mm from the bottom of the beaker. Either type plastic specimen holder (figure 3) may be used. The hole through which the specimen is suspended shall be unobstructed. Figure 1 shows the arrangement of the apparatus.

c) Continue stirring at a speed of 1,000 ± 50 rev/min for 24 h maintaining the temperature of the emulsion at 60 ± 1°C. Stop stirring at the end of the 24 h period and remove the specimen. Rinse the specimen with distilled water immediately, so that the water from the emulsion film does not evaporate to leave an oil residue. Dry the specimen by rinsing in acetone. If desired the specimen may be preserved by lacquering.

5.9.3.7. Acceptance criteria

a) All inspections at the end of the test to determine the condition of specimens shall be performed without magnification under normal light. For the purpose of this test, normal light is considered to be illumination of about 645.8 lux. Within the meaning of this method, a rusted specimen is one on which any rust spot or streak is visible by the above inspection procedure.

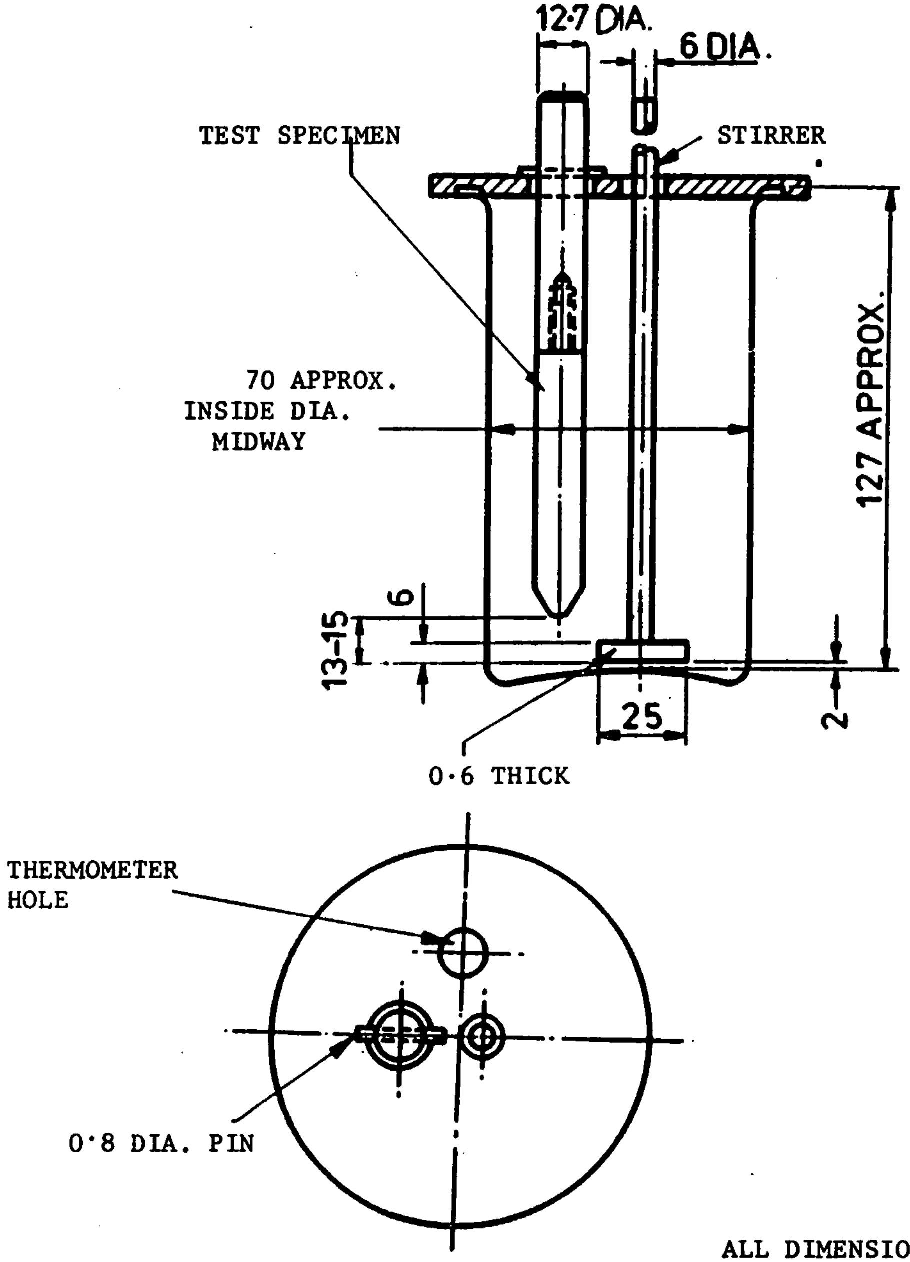
b) In order to report an oil as passing or failing, the test must be conducted in duplicate. An oil shall be reported as passing the test if both specimens are rust-free at the end of the test period. An oil shall be reported as failing the test if both specimens are rusted at the end of the test period (Note 6). If one specimen is rusted while the other is free of rust, tests on two additional specimens shall be made (Note 3). If either of these latter specimens shows rusting, the oil shall be reported as not passing the test. If neither of these latter specimens shows rusting, the oil shall be reported as passing the test.

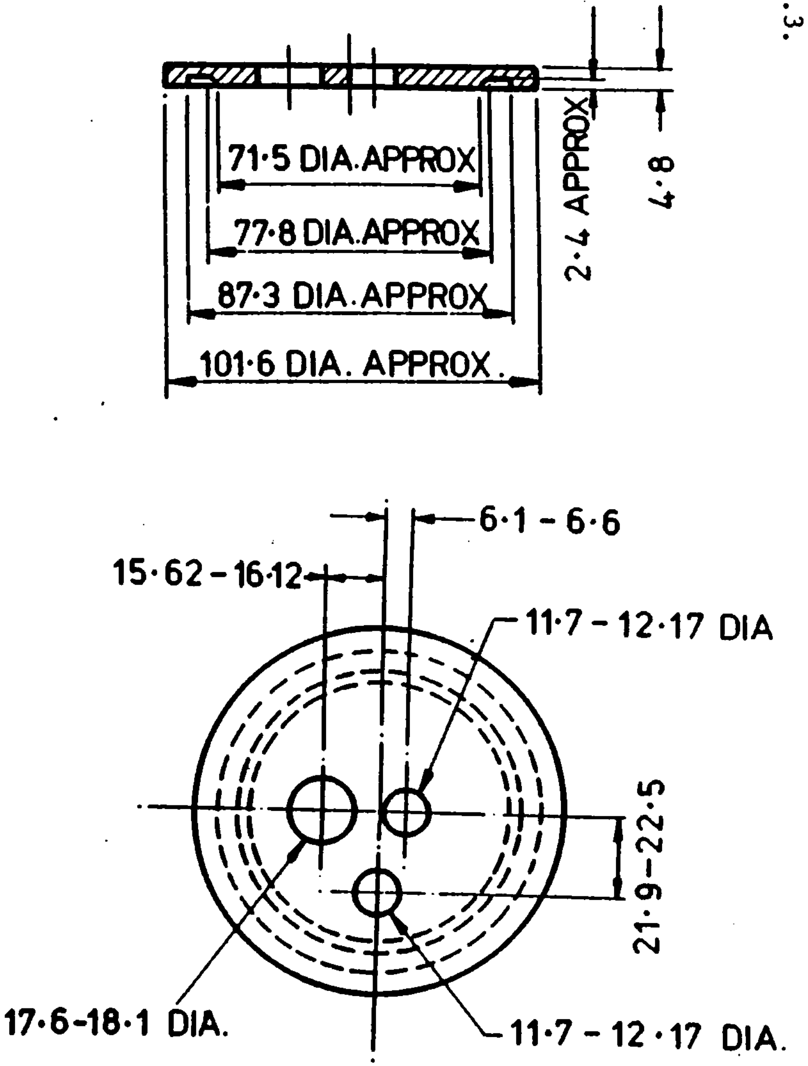
Note 6 : An indication of the degree of rusting occurring in this test may be desired. For uniformity in such cases, use of the following classifications of rusting severity is recommended :

Light rusting - Rusting confined to not more than six spots, each of which is 1 mm or less in diameter.

Moderate rusting - Rusting in excess of the above but confined to less than 5% of the surface of the specimen.

Severe rusting - Rusting covering more than 5% of the surface of the specimen.





ALL DIMENSION IN MILLIMETRES

Figure 1 Apparatus for rusting test

Figure 2 Beaker cover

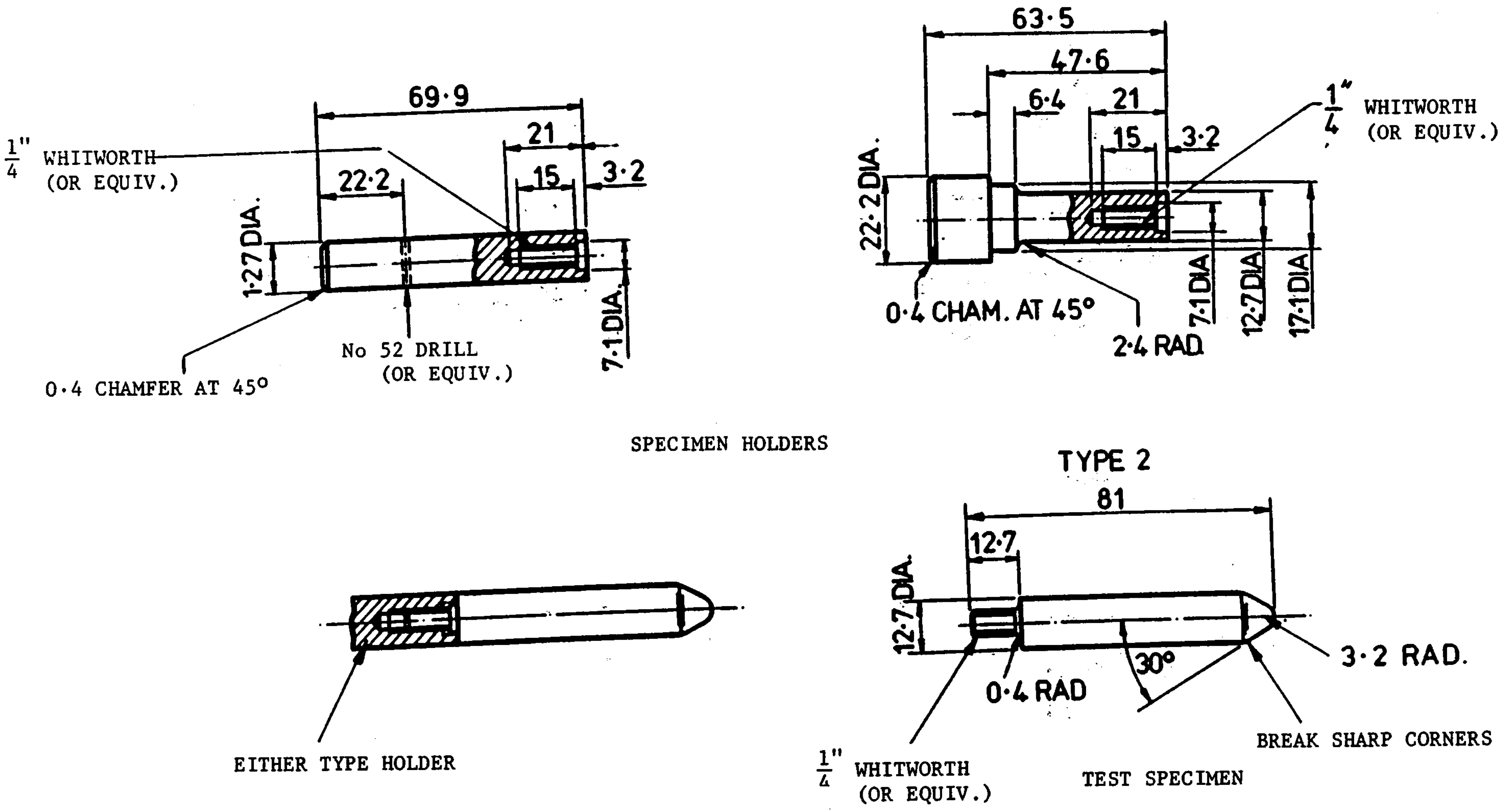
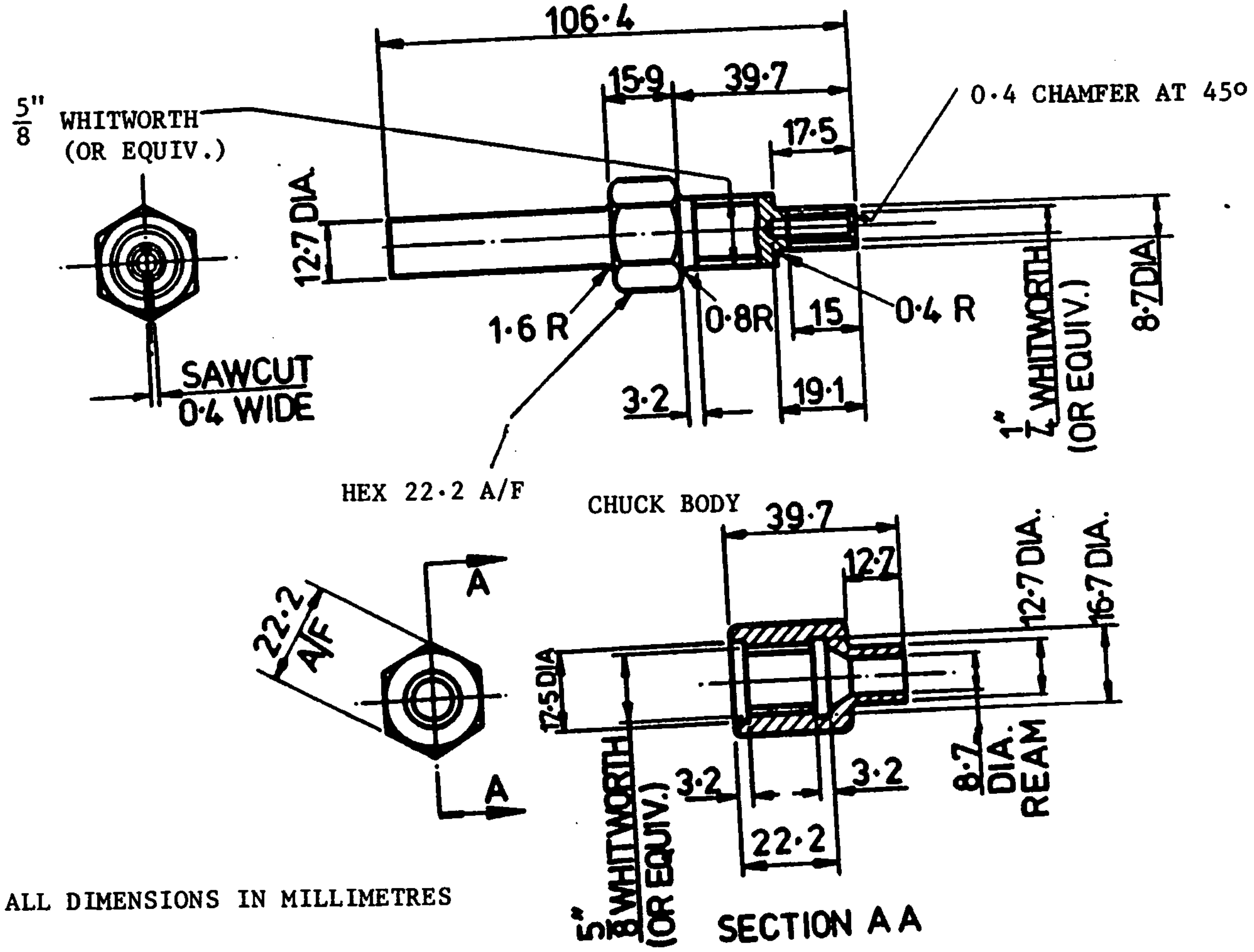


Figure 3 Test specimen assembly



CHUCK LOCK NUT

Figure 4 Chuck for polishing test specimens

5.9.4 METHOD OF TEST FOR CORROSION INHIBITING PROPERTIES (ALL FLUIDS)

Note : This method of test is under development and evaluation which will eventually replace the methods described in sections 5.9.1 and 5.9.2.)

5.9.4.1 Scope

This method of test determines the corrosion inhibiting properties of hydraulic fluids in contact with selected metals. The method of test can be applied to all types of fire-resistant fluids. In the case of type HFA fluids, the test is to be conducted with made-up emulsions.

5.9.4.2 Principle

Test pieces of selected materials are partly immersed in the test fluid for a specified time. The change in weight of the test pieces and changes in the surfaces of the materials, as well as changes in the colour of the fluid, are noted.

5.9.4.3 Apparatus and chemicals

a) Glass bottles with stoppers and seals as shown in figure 1.

b) Thermostatically controlled heating bath which can maintain the temperature of the test fluid in the bottles at 60 ± 1°C. The heating bath must be provided with a stirring device to ensure uniform temperature distribution throughout the bath. A drying cupboard with similar temperature regulation may be substituted for the heating bath.

c) Abrasive papers of increasing fineness, e.g. P 120, P 240, P 400, P 600.

d) Cotton wool.

e) Acetone and n-heptane.

f) Toluene.

g) Distilled water.

h) Test pieces each 100 mm long, 20 mm wide and about 1 mm thich, with a 5 mm hole near the upper edge of one short side, so that the test pieces can be hung on a glass hook.

The following materials are used for the test :

- steel with the following composition:

C	0.35 to 0.45%
Mn	0.5 to 0.8%
Si	0.10% max.
S and P	0.35% max.

- Electrolytic copper.
- Pure zinc.
- Pure aluminium (at least 99.5%)

- Pure cadmium.
- Brass (70% Cu and 30% Zn).

Note : The test may also be applied to other metals and alloys used in the manufacture of mining equipment.

i) Analytical balance with a sensitivity of 0.0002 g.

j) Forceps with rounded ends not ribbed on the inner faces.

5.9.4.4 Preparation

When testing type HFA fluids, an emulsion shall be prepared for the test using one of the standard test waters (normally the hardest of the test waters with which the manufacturer has declared the emulsifying oil to be compatible), see section 2.2.2.6. The proportion of emulsifying oil in the emulsion shall be that recommended by the manufacturer for normall applications or such other value as may be specified by the approving/accepting authority.

The edges of the test pieces are first smoothed with abrasive paper P 240, P 400 and P 600. The surfaces of the test pieces are then prepared with suitable abrasive papers, ending with the paper P 600 to give the best possible surface finish. The test pieces are then held with forceps and cleaned with dry cotton wool, followed by cotton wool soaked in heptane. The traces of cotton wool are washed off with n-heptane. As soon as the last traces of solvent adhering to the test pieces have evaporated, they are weighed on the analytical balance. The test pieces must then be used immediately for the corrosion tests.

5.9.4.5 Procedure

At least 11 bottles are filled for the entire test series, each with 300 ml of the test fluid.

Six of the bottles receive one each of the following test pieces, prepared in advance as described in section 5.9.4.4:

steel, copper, zinc, aluminium, cadmium and brass.

Each test piece is suspended from the hook in such a way that approximately 60 mm of the test piece is immersed in the fluid.

To test the behaviour of the fluid in the presence of two metals, the following pairs of test pieces are suspended in four bottles:

steel and pur cadmium;

copper and zinc;

aluminium and zinc; and

steel and aluminium.

The two test pieces of each pair should be kept about 1 mm apart, either by means of a plastic spacer measuring 1 x 10 x 15 mm, and pierced in its centre by a hole 5 mm diameter, which is placed between the two test pieces or by means of two notches on the suspension hook.

The eleventh bottle contains 300 ml of the test fluid, in which no test piece has been immersed, and serves to check the precipitation of the fluid itself during the test period.

The bottles are placed in the thermostatically controlled heating bath or the drying oven, whose temperature is set to give a constant temperature of 60 ± 1°C in the test fluid. This temperature is maintained for a minimum period of 14 days.

At the end of the test, the changes is the surface of the test pieces must be described including oxidation colours, corrosion and deposits. In addition, for each analysis of the metal surfaces, a record must be made of the colour and appearance of the test fluid and of deposits. After visual examination, the test pieces must be grasped by means of forceps in the immediate vicinity of the suspension hole.

In the case of types HFA and HFB fluids, the test pieces must be immersed in a bottle filled with n-heptane and then in a bottle of acetone to remove the fluid. The forceps are then used to agitate and the test pieces in air until the acetone has fully evaporated and the surfaces appear dry. The test pieces are then weighed on the precision balance. If wet patches remain on the surface after the first rinsing and drying the process must be repeated with new solvents as often as is necessary to ensure that no wet patches remain after evaporation of the solvent.

For type HFC fluids, the same method of cleaning is employed with distilled water and then acetone.

For type HFD fluids, the same method is again employed but with toluene only.

5.9.4.6 Results

The difference in mass is indicated in mg per test piece, both gains (+) and losses (-) in weight being recorded.

5.9.4.7 Acceptance criteria

These criteria will be fixed as soon as sufficient experience with the use of this test has been accumulated.

Note

This method of test is proposed as an eventual harmonised test. Its use alongside the methods described in sections 5.9.1 and 5.9.2 will enable laboratories to compile comparative data and effect an early evaluation and assessment of the method.

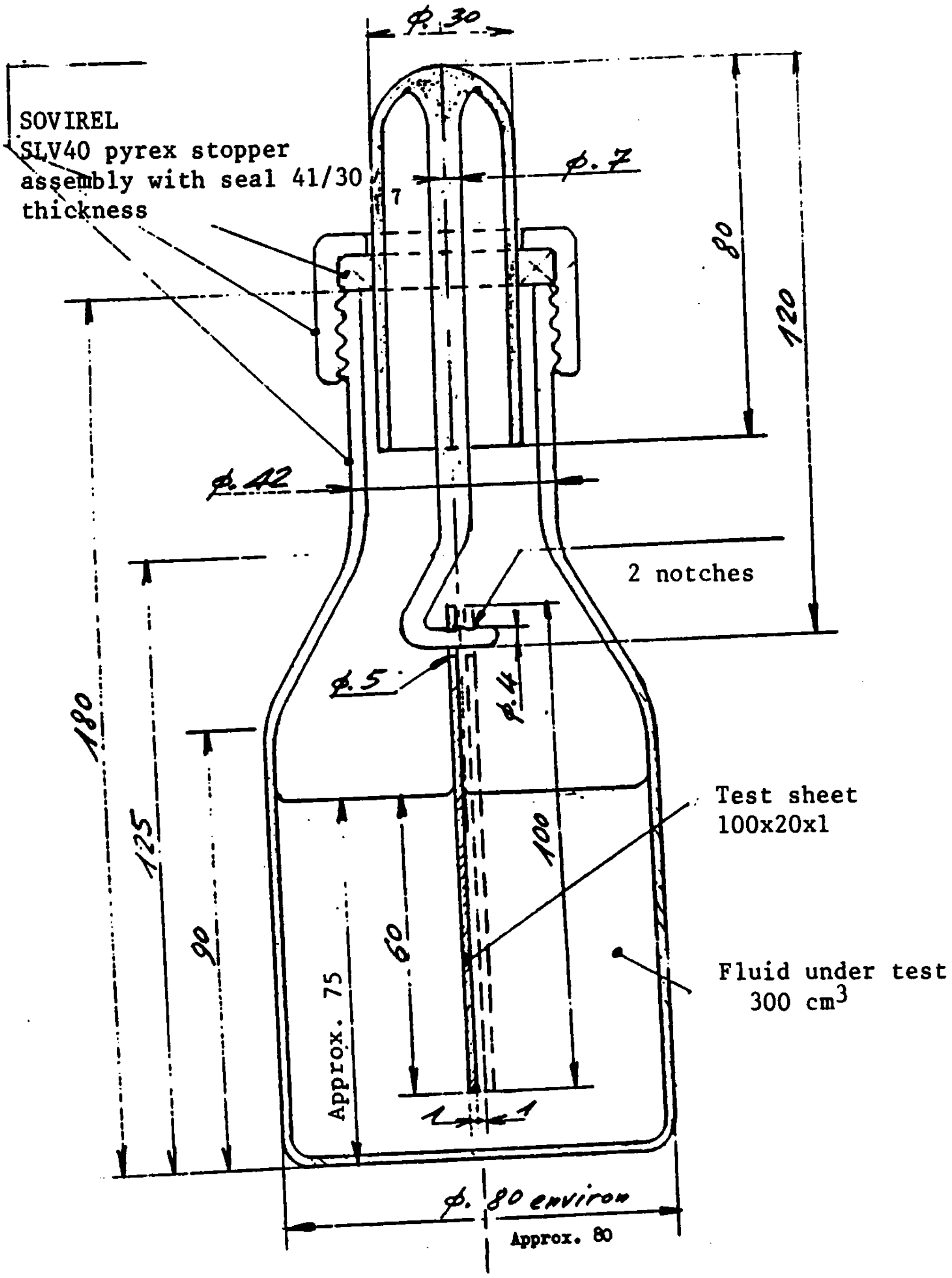


Figure 1 Test piece in bottle

5.10 METHODS OF TEST FOR COMPATIBILITY OF SEALING MATERIALS AND FLUIDS OF TYPES HFA, HFB, HFC AND HFD

Note :

These tests are intended to be used to determine the degree of compatibility between fire-resistant fluids and the materials used for seals and packings by measuring the changes in the volume and hardness of the material after a period of immersion in the fluid. In this context, the term sealing material covers all rubbers and other materials used to seal and prevent fluid leakage from tanks, enclosures and hydraulic machinery and includes both the materials used for static applications such as flange joints and those moulded or shaped seals for rotary and reciprocating motions.

Two methods of test are described, the first of which (section 5.10.1) is similar to the method of test described in the Fifth Report and the method of test specified in BS 903 and which have been used in the 'Community of Six' and the United Kingdom respectively. It is essentially a method of test to be applied to samples of actual sealing materials supplied by a manufacturer or user of seals.

The second method of test (section 5.10.1) is based on the relevant parts of CETOP RP 81 H and determines the effect of the fluid on selected standard elastomers. These elastomers are supplied specifically for test purposes after preparation under closely controlled conditions and to finer quality tolerances than is normal with commercially produced materials. This method of test will enable a fluid manufacturer to assess his product against clearly defined standard materials. This second method of test has not yet been fully evaluated and thus no acceptance criteria can be specified.

5.10.1 METHOD OF TEST FOR COMPATIBILITY OF SEALING MATERIALS AND FLUIDS OF TYPES HFA, HFB, HFC AND HFD

Note : This method of test is similar in principle to those described in section 3.11 of the Fifth Report and used in the 'Community of Six' and in BS 903 as used in the United Kingdom. The temperature and duration of test differ in those two specifications and are included in this test description as alternatives for fluids of types HFA, HFB and HFC.

5.10.1.1 Scope

This method of test is to determine the degree of swelling and the change in hardness of sealing materials which are intended for use with fire-resistant fluids of types HFA, HFB, HFC and HFD.

5.10.1.2 Principle

Test pieces of the particular type of sealing material are immersed in a sample of the fluid with which they are proposed to be used in service. The time of immersion and the temperature of the fluid are prescribed. The volume and hardness in International Rubber Hardness Degrees (IRHD) of each test piece are measured before and after immersion.

5.10.1.3 Apparatus

1) A wide mouthed glass bottle, stoppered or sealable with a screwed cap, of at least 500 ml capacity is required for each test piece.

2) A thermostatically controlled bath or oven capable of maintaining the bottles of test fluid at the prescribed temperature (60, 70 or 100°C whichever is relevant) with an accuracy of ± 1°C.

3) A IRHD Standard Tester with scale divisions of 1 IRH degree, conforming to the requirements of ISO Standard R 48.

4) A laboratory balance reading to 0.1 mg.

5.10.1.4 Preparation and sampling

1) The test pieces of the sealing material must be cut from flat sheets, 6 ± 1 mm thick. Sample sheets of approximately 100 x 100 mm and of this thickness are to be supplied by the seal manufacturer.

2) Three test pieces, each 50 mm long and 25 mm wide shall be cut from widely separated places from the sample sheet of sealing material supplied.

3) The test pieces shall be conditioned in air for not less than 3 h at a temperature of 23 ± 2°C immediately before the test.

4) A quantity of at least 1 litre of the fluid with which the sealing material is to be used.

5.10.1.5 Procedure

Weigh each of the test pieces to the nearest mg :

a) in air at 23 ± 2°C (W_1) ; and

b) in distilled water at 23 ± 2°C (W_2) (See note 1)

Blot the test pieces dry with filter paper. Determine the hardness of each test piece before its immersion in the test fluid by the following method:

Lightly dust the upper and lower surfaces of the test pieces with talc. Support each test piece, in turn, on a horizontal rigid surface and lower the foot of the IRHD Standard Tester to rest on the surface of a test piece. Apply the minor load on the indenting ball, vertically on the test piece, for 5 s. Turn the bezel of the gauge so that the pointer indicates 100, exercising care to avoid exerting any vertical pressure on the gauge.

Apply the major load to the plunger and maintain the total load on the ball for 30 s, gently vibrating the apparatus to overcome any friction. The reading on the gauge is the hardness in IRH degrees. Make one measurement at each of three points distributed over the test piece, at distances from the edges greater than 8 mm.

Suspended each test piece individually in a separate wide mouthed glass bottle and add to each bottle 300 ml of the test fluid.

Stopper or cap the bottles and place them in the thermostatically controlled bath or oven which, for types HFA, HFB and HFC fluids, is to be maintained at a temperature of 60 ± 1°C and for type HFD fluid 100 ± 1°C for the test period of 21 days.

Alternatively, for types HFA, HFB and HFC fluids, the bath or oven may be maintained at a temperature of 70 ± 1°C in which case the duration of the test is reduced to 7 days (168 h).

After the test period of 21 days (or 7 days as the case may be) restore the test pieces to a temperature of 23 ± 2°C, preferably by quickly transferring them to a fresh portion of the test fluid maintained at that temperature and holding them immersed for a period of not less than 30 min and not more than 60 min.

Note 1 : Care should be taken to ensure that all air bubbles are removed when weighing in water. To minimise the adherence of air bubbles to the test piece, it is permissible to add a trace (say 1 part in 10,000) of surface active material such as a detergent to the distilled water.

Remove surplus fluid from the surfaces of each test piece by wiping with filter paper. Place immediately in a tared and stoppered weighing bottle and weigh in air (W_3) to the nearest mg. Remove from the bottle and immediately weigh in distilled water (W_4) at 23 ± 2°C.

Determine the hardness of the test pieces after their immersion in the fluid by the method described above.

5.10.1.6 Results

Calculate the change in volume as follows:

The percentage change in volume is given by the expression:

$$\frac{(W_3 - W_4) - (W_1 - W_2)}{(W_1 - W_2)} \times 100$$

where W_1 = initial weight of test piece in air

W_2 = initial weight of test piece in distilled water

W_3 = weight of test piece in air after immersion in the test fluid

W_4 = weight of test piece in distilled water after immersion in the test fluid

The median result for the three test pieces shall be reported as the percentage change in volume.

Calculate the change in hardness as follows:

Change in hardness = $H_2 - H_1$

where H_1 = initial hardness of test piece, IRHD

H_2 = hardness of test piece after immersion in the test fluid, IRHD.

The median result for the three test pieces shall be reported as the change in hardness in IRH degrees.

5.10.1.7 Acceptance criteria

The increase in volume of the sealing material exposed to fluids of types HFA, HFB, HFC and HFD should not exceed 7%. Contraction of the test samples should not occur.

The change in hardness should not be more than 2 IRH degrees above or more than 7 IRH degrees below the initial hardness of the test pieces for all types of fluids.

5.10.2 METHOD OF TEST FOR COMPATIBILITY OF FLUIDS WITH STANDARD TEST ELASTOMERS

Note : This method of test is based on these parts of CETOP Method RP 81 H which are relevant to the types of fire-resistant fluids covered by this report. It is still under assessment and evaluation.

5.10.2.1 Scope

The object of this method of test is to assess the performance of fluids with materials which are representative of those used for the manufacture of seals and packings for hydraulic equipment.

Three standard test elastomers are specified for the purpose of making this evaluation and the standard elastomers to be tested with the different types of fluid are also specified.

5.10.2.2 Principle

The changes in volume and hardness suffered by test pieces of the selected elastomer when immersed in the test fluid under specified conditions are measured and reported.

5.10.2.3 Standard test elastomers

The three standard test elastomers are designated as follows:

Nitrile rubber	NBR 1
Fluoroelastomer	FPM 1
Ethylene propylene rubber	EPDM 1

Details of the formulations of these materials are given in Appendix 1 to this test method.

In these formulations it was the aim to provide materials which would be sensitive to fluid variations and therefore of comparatively high swelling characteristics. Stable cure systems were used to give adequate storage life.

It is to be noted that these formulations are not designed to provide elastomeric materials for actual service.

5.10.2.4 Apparatus

a) Analytical balance having a sensitivity of 0.1 mg with nylon filament and a beaker containing distilled water to be placed on a bridge as shown in figure 1.

b) Stoppered glass jars of dimensions such that the rubber test pieces remain completely immersed in the fluid under test, and are free to swell without restraint or distortion. The diameter of the mouth should be such as allow free entry and exit of the test pieces.

c) Fan assisted air circulating oven capable of maintaining temperature to within ± 2°C.

d) Apparatus for measuring hardness as described in ISO Recommendation R 48 - micro test.

5.10.2.5 Test pieces

a) Selection of standard elastomers and test conditions

The standard test elastomers to be used and the main conditions of test are given in the table 1:

Table 1. Standard elastomers and test conditions

Type of fluid	Suitable test elastomers	Temperature of test °C (± 2°)	Duration of test h (± 2h)
HFA	NBR 1 EPDM 1 FPM 1	60	168 *
HFB	NBR 1 FPM 1	60	168 *
HFC	NBR 1 EPDM 1 FPM 1	60	168 *
HFD Halogenated hydrocarbon	FPM 1	100	168
HFD Phosphate ester	EPDM 1 FPM 1 **	100	168

* 168 h is the test duration, but equilibrium may not be achieved in these fluids.

** FPM 1 is not suitable for use with alkyl phosphate esters.

A fluid should be tested against each of the test elastomers which are representative of the types of sealing or packing materials in equipment in which the particular fluid is to be used.

b) Test pieces

Test pieces should be cut from sheets of uniform thickness of 2 ± 0.2 mm and may be rectangular (50 x 25 ± 1 mm) or circular (diameter 36 ± 1 mm). Both shapes give practically the same results.

Note : Prior to carying out the percentage volume swell test, the initial hardness of the test pieces shall be determined by the method described in section.

5.10.2.6 Procedure

a) Determination of percentage volume change

Use three marked test pieces for each of the standard elastomers to be tested. Weigh each test piece in air to the nearest mg (m 1) and then reweigh each test piece in distilled water (m 2) at the standard laboratory temperature of 23 ± 2°C, care being taken to ensure that all air bubbles are removed. Formation of bubbles may be avoided by dipping the test pieces momentarily into a suitable liquid such as methyl alcohol. Blot the test pieces dry with filter paper or a piece of textile which does not deposit lint.

Completely immerse each of the test pieces in a separate glass jar containing fluid in such a way that they do not rest flat on the bottoms of the jars. In the case of type HFA fluids each jar shall contain at least 300 ml of the test fluid. With other fluids the volume shall be at least 15 times the volume of the test piece. Stopper the jars and put them in the test oven. The test time and temperature, which depend on the nature of the fluid and the test elastomer, are to be as given in table 1. Note that where there are relatively few test pieces to be tested, they may be placed in a single larger glass jar provided they can be effectively separated from each other and the volume of fluid in relation to the total volume of test pieces in the jar meets the above requirement.

At the end of the immersion period, bring the jars and their contents to the standard laboratory temperature. Remove any surplus fluid from the surfaces of the test pieces. For removing non-aqueous fluids, dip each test piece momentarily in a suitable volatile liquid such as petroleum ether and again quickly wipe with filter paper or a piece of textile which does not deposit lint. Weigh each test piece in air (m 3) to the nearest mg. Immediately afterwards weigh the test pieces in distilled water at the standard laboratory temperature.

Remove the test piece from the bottle and immediately weigh in distilled water (m 4) at the standard laboratory temperature.

Results

The percentage volume change is given by the formula:

$$\frac{(m\,3 - m\,4) - (m\,1 - m\,2)}{(m\,1 - m\,2)} \cdot 100$$

where:

m 1 is the initial mass of the elastomer in air;
m 2 is the initial apparent mass of the elastomer in water;
m 3 is the mass in air of the elastomer after immersion;
m 4 is the apparent mass in water of the elastomer after immersion.

The median of the three measurements (three test pieces) shall be reported.

b) Determination of change in hardness

For each elastomer to be tested, the same three test pieces used for the determination of percentage volume change shall be used. (They shall be reused after completion of the volume change test).

Measure the hardness of the three test pieces at the standard laboratory temperature of 23 ± 2°C (The procedure is described in ISO Recommendation R 48 - micro test). Three hardness readings at different points on the surface of the test piece, each of which is at least 2 mm away from the edge of the test piece, shall be made before and after immersion in the fluid. The average value of the hardness of each test piece before and after immersion shall be determined.

Results

The change in hardness is calculated as:

$$\Delta H = H_2 - H_1$$

where:

H_1 is the hardness (IRHD) measured before immersion;

H_2 is the hardness (IRHD) measured after immersion;

ΔH is the change in hardness.

The median value of the three determined values for change in hardness shall be reported.

5.10.2.7 Acceptance criteria

Evaluation of this method of test is not yet complete and thus acceptance criteria cannot be defined. It is anticipated that laboratories of the Member States will collaborate in the assessment of the results of these tests, the alignment of those results with the results of other types of test and in the determination of suitable acceptance criteria.

Tests so far carried out indicate that; with the exception of those material/fluid combinations marked with an asterisk (*) in the table below where high degrees of swelling (up to 25%) have been reported, most results show a swelling of not more than 8%.

Elastomer				Fluid		
	HFA	HFAS	HFB	HFC	HFD	
					PCB	Phos. Ester
NBR 1	√ *	√	√ *	√		
FPM 1	√		√	√	√	√ *
EDPM	√			√		√ *

APPENDIX 1. TEST ELASTOMERS, MIXING PROCEDURES AND PREPARATION OF STANDARD VULCANIZED SHEETS

A 1 Introduction

This appendix provides formulations, mixing and vulcanization procedures for three types pf elastomeric compositions:

Nitrile rubber	NBR 1
Fluoroelastomer	FPM 1
Ethylene propylene rubber	EPDM 1

In these formulations it was the aim to provide materials which would be sensitive to fluid variations and therefore of comparatively high swelling characteristics and which would give a positive swell in all hydraulic fluids. Stable cure systems were used to give adequate storage life.

The mixing and vulcanization procedures of these test elastomers should follow the general guidelines of ISO 2393.

It is desirable that there should be one source of test elastomers and an independent check on the quality of each batch produced.

It is to be noted that these formulations are not designed to provide materials for actual service seals or packings.

A 1.1 Standard nitrile rubber NBR 1

a) Composition by weight

1) NBR (Nitrile-butadiene rubber) (a)	100.0
2) Zinc oxide (rubber grade)	5.0
3) Polymerized 2.2.4 - trimethyl 1.2 - dihydroquinoline (m.p. 75 to 100°C)	0.5
4) FEF carbon black (ASTM designation : N 550)	70.0
5) "Dicumyl peroxide (grade with 40% peroxide content on inert filler"	3.0

Note (a) Acrylonitrile content 28 ± 1%, cold polymerized, Mooney viscosity (ML-4/100°C) - 45 ± 5.

b) Mixing procedure

Proceed as follows maintaining the roll temperature at 50 ± 5°C:

1) Band crude rubber with mill opening at 1.4 mm and break down.
2) Add zinc oxide and then polymerized 2.2.4 - trimethyl - 1.2 dihydroquinoline evenly across the rolls at constant rate. Make three 3/4 cuts from each side.
3) Add about half the carbon black evenly across the rolls at a constant rate and open the mill at intervals to maintain an approximately constant bank.
4) Make three 3/4 cuts from each side.
5) Add the rest of the black and make certain to add all the pigment that drops through to the pan.
6) Add dicumyl peroxide evenly across the rolls. Make six 3/4 cuts from each side.

7) Cut out the compound from the mill, set the opening at 0.2 mm and pass the rolled stock endwise through the mill six times.

8) Sheet off at 0.2 mm, cool on a flat metal surface and prepare samples for cure.

c) Preparation of standard vulcanized sheets

Standard vulcanized sheets of 2 ± 0.2 mm thickness should be cured 20 min at 170°C.

d) Control tests

All tests should be made on sheets of 2 ± 0.2 mm thickness.

1) Hardness in IRHD - micro test, ISO Recommendation R 48	78 - 83
2) Tensile strength, dumb-bell type 2, ISO Recommendation R 37, in dN/mm^2	210 - 250
3) Elongation at break, dumb-bell type 2, ISO Recommendation R 37, in percent.	200 - 240
4) Compression set 22 h/100°C, small type of test piece obtained by plying 3 discs (ISO 815)	14 - 18
5) Density (g/cm^3), ISO/DIS 2781	1.23 ± 0.0
6) Percentage change in weight after 22 h immersion in ISO liquid B (70% (V/V) pur iso-octane + 30% (V/V) pure toluene) at 23 ± 2°C, ISO Recommendation R 1817.	24.5 - 27.5

A 1.2 Standard fluoroelastomer FPM 1

a) Composition by weight

1) Vinylidene fluoride hexafluoropropylene copolymer:

1) VITON A EI DU PONT DE NEMOURS or equivalent	100
2) Magnesium oxide : MAGLITE Y (MERCK & Co.) or equivalent	20
3) MT carbon black (ASTM designation N 990)	20
4) N.N dicinnamylidene - 1.6 hexane diamine	3

b) Mixing procedure

Proceed as follows maintaining the roll temperature at 50 ± 5°C:

1) Band the crude rubber with mill opening of 1.4 mm.

2) Blend carbon black, magnesium oxide and biscinnamylidene-hexanediamine together and add evenly across the rolls at a constant rate.

3) Open the mill at intervals to maintain an approximately constant bank.

4) When all the pigment has been added, make a 3/4 cut from each side. Be certain to add all the pigment that drops through to the pan.

5) Make three 3/4 cuts from each side.

6) Cut the batch from the mill. Set the opening at 1.4 mm and pass the rolled stack endwise through the mill six times.

7) Sheet off at 2.2 mm and allow to cool on a flat metal surface, hold 24 h.

8) Refine mixed stock after 24 h ageing by passing six times through a tightly set mill (0.8 mm) with roll temperature of 40 ± 5°C.
9) Sheet off at 2.2 mm and allow to cool on a flat metal surface. Prepare samples for cure.

c) Preparation of standard vulcanized sheets

Standard vulcanized sheets of 2 ± 0.2 mm thickness should be mould cured 20 min at 170°C and postcured 24 h at 200°C in an air circulating oven.

d) Control tests

All tests should be made on sheets of 2 ± 0.2 mm thickness.

1) Hardness in IRHD micro test, ISO Recommendation R 48	73 - 78
2) Tensile strength, dumb-bell type 2, ISO Recommendation R 37, in dN/mm^2	140 - 160
3) Elongation at break, dumb-bell type, ISO Recommendation R 37, in percent	270 - 310
4) Compression set 22 h/150°C, small type of test piece obtained by plying 3 discs (ISO 815)	24 - 30
5) Density (g/cm^3), ISO/DIS 2781	1.91 ± 0.02
6) Percentage change in weight after 22 h, immersion in pure toluene at 23 ± 2°C	2.5 - 3.5

A 1.3 Standard ethylene propylene rubber EPDM 1

a) Composition by weight

1) EPDM (Ethylene propylene diene monomer rubber): VISTALON 2504 (EXXON CHEMICAL Co.) or equivalent	100.0
2) FEF carbon black (ASTM designation N 550)	50.0
3) Zinc oxide (rubber grade)	5.0
4) Polymerized 2.2.4 - trimethyl 1.2 - dihydroquinoline (m.p. 75 - 100°C)	0.5
5) Dicumyl peroxide (grade with 40% peroxide content on inert filler)	5.0

b) Mixing procedure

Proceed as follows maintaining the roll temperature at 50 ± 5°C:

1) Band the crude rubber with mill opening of 1.4 mm
2) Blend carbon black and zinc oxide together and add evenly across the rolls at constant rate.
3) Open the mill at intervals to maintain approximately constant bank
4) When all the pigment has been added, make one 3/4 cut from each side. Be certain to add all the pigment that drops through to the pan.
5) Add dicumyl peroxide evenly across the rolls. When it is incorporated, make three 3/4 cuts from each side.
6) Cut out the compound from the mill, set the opening at 0.8 mm and pass

the rolled stack endwise through the mill six times.

7) Sheet off at 2.2 mm, cool on a flat metal surface and prepare samples for cure.

c) Preparation of standard vulcanized sheets

Standard vulcanized sheets of 2 ± 0.2 mm thickness should be cured 20 min at 170°C.

d) Control tests

All tests should be made on sheets of 2 ± 0.2 mm thickness.

1) Hardness in IRHD micro test, ISO Recommendation R 48	67 - 72
2) Tensile strength, dumb-bell type 2, ISO Recommendation R 37, in dN/mm^2	160 - 200
3) Elongation at break, dumb-bell type 2, ISO Recommendation R 37, in percent	240 - 280
4) Compression set 22 h/150°C, small type of test piece obtained by plying 3 discs (ISO 815)	20 - 25
5) Density (g/cm^3), ISO/DIS 2781	1.08 ± 0.01
6) Percentage change in weight after 22 h immersion in methyl ethyl ketone at 23 ± 2°C, ISO Recommendation R 1817	8.5 - 9.5

PART VI

DISCRETIONARY TECHNOLOGICAL TESTS

6 DISCRETIONARY TECHNOLOGICAL TESTS

The tests described in this part of the report, and designated as 'discretionary technological tests, are those which are, firstly, supplementary to certain of the tests in Part V, such as the measurement of fluid density and the determination of the neutralisation number of a fluid and, secondly, required by a purchaser or accepting/approving authority to verify the suitability of a fluid for particular applications and conditions of use.

In the case of these discretionary tests, precise acceptance criteria are not specified and they will normally be the subject of agreement between the purchaser and the manufacturer. In certain cases, however, acceptance criteria are included in the test description but this is for information only and as guidance on the interpretation of the test results.

6.1. DETERMINATION OF FLUID DENSITY

Note : The density of a fluid, although varying with temperature and to a small extent with pressure, is a fundamental physical property. Provided, therefore, that the temperature of measurement is fixed, different methods should give results which, subject to the limitations of experimental accuracy, are equivalent.

6.1.1. Procedure

Density in g/ml at a temperature of 15°C should be determined by any standard method capable of an accuracy within the range ± 0.001 g/ml. The laboratory making the determination should be allowed to choose a suitable test method. For this purpose the following methods are considered satisfactory :

ISO/DIS 3675
BS 4714
IP 59
IP 160
IP 190
DIN 51757
AFNOR NF T 60-101
NBN 52.011
NBN 52.015
NBN 907
ASTM D 97-66

Other methods may be permitted subject to the above requirements for temperature of determination and accuracy.

6.1.2. Results

The method used for the determination of density should be stated and the density recorded in g/ml at 15°C.

6.1.3. Acceptance criteria

The value of the density of a fluid is required for information purposes only and no specific requirements are laid down. In some applications such as fluid couplings and hydraulic pumps, the density has an effect on the operating characteristics and thus its value should be known. For all practical purposes the above accuracy of ± 0.001 g/ml will be adequate.

6.2. METHOD OF TEST FOR THE NEUTRALISATION NUMBER OF TYPES HFB AND HFD FLUIDS

Note : This method of test by potentiometric titration is based on the methods described in ASTM D664, BS 4457 and IP 177.

6.2.1. Scope

This method describes procedures for the determination of the total acidity or basicity of fluids of types HFB and HFD by potentiometric titration.

The method may be used to indicate relative changes that occur in a fluid during use under oxidizing conditions regardless of the colour or other properties of the resulting fluid. Although the titration is made under definite equilibrium conditions, the method is not intended to measure an absolute acidic or basic property that can be used to predict performance of a fluid under service conditions. No general relationship between bearing corrosion and acid or base number is known.

The neutralization numbers obtained by this method may not be numerically the same as those obtained by methods utilising colour indicators.

6.2.2. Definitions

Total Acid Number - The quantity of base, expressed in milligrams of potassium hydroxide, that is required to neutralize all acidic constituents present in 1 g of sample.

Total Base Number - The quantity of acid, expressed in terms of the equivalent number of milligrams of potassium hydroxide that is required to neutralize all basic constituents present in 1 g of sample.

6.2.3. Principle

The sample is dissolved in a mixture of toluene and isopropyl alcohol containing a small amount of water and titrated potentiometrically with alcoholic potassium hydroxide or hydrochloric acid solution, using a glass indicating electrode and a calomel reference electrode. The meter readings are plotted against the respective volumes of titrating solution and the end points are taken at the inflexions in the resulting curve. When no definite inflexions are obtained, end points are taken at meter readings corresponding to those found for standard non-aqueous acidic and basic buffer solutions.

6.2.4. Apparatus

The apparatus shall consist of the following:

1) Meter. A voltmeter or potentiometer that will operate with an accuracy of plus or minus 0.005 V and a sensitivity of plus or minus 0.002 V, over a range of at least plus or minus 0.5 V, when the meter is used with the electrodes specified in Paragraphs (2) and (3), and when the resistance between the electrodes falls within the range of 0.2 to 20 megohms. The meter shall be protected from stray electrostatic fields so that no permanent change in the meter readings over the entire operating range is produced by touching with a grounded lead, the titration stand, or the meter. A desirable apparatus may consist of a continuous-reading electronic voltmeter with specified range, accuracy, and sensitivity, that is designed to operate on an imput of less than 5 x 10^{-12} amp when an electrode system, having 1000 megohms resistance is connected across the meter

terminals, that is provided with a metal shield connected to the ground, and that is provided with a satisfactory terminal to connect the shielded connexion wire from the glass electrode to the meter without interference from the presence of external electrostatic fields.

2) Glass electrode for pH 1 to 13. A pencil-type glass electrode (C. Figure 1) 125 to 180 mm in length and 8 to 14 mm in diameter. The body of the electrode shall be made of a chemically resistant glass tube with a wall thickness of 1 to 3 mm. The entire electrical connexion from the sealed contact cell to the meter terminal shall be surrounded by an electrical shield that will prevent electrostatic interference when the shield is grounded. The shield shall be insulated from the electrical connexion by insulating material of the highest quality, such as rubber and glass, so that the resistance between the shield and the entire length of the electrical connexion is greater than 50,000 megohms.

3) Calomel Electrode. A pencil-type calomel electrode (B. Figure 1) 125 to 180 mm in length and 8 to 14 mm in diameter. This electrode shall be made of glass and shall be provided with an external, removable glass sleeve on the sealed end that is dipped into the titration solution. The glass sleeve shall be 8 to 25 mm in length, shall be slightly tapered, and shall be ground to fit the electrode so that the sealed end of the electrode protrudes 2 to 20 mm beyond the sleeve. The ground surface shall be continuous and free of smooth spots. At a point midway between the extremities of the ground surface, the electrode tube shall be pierced by a hole or holes 1 mm in diameter. The electrode shall contain the necessary mercury, calomel, and electrical connexion to the mercury, all arranged in a permanent manner. The electrode shall be filled almost to capacity with saturated KCI electrolyte and shall be equipped with a stoppered port through which the electrolyte may be replenished. When suspended in the air and with the sleeve in place, the electrode shall not leak electrolyte at a rate greater than one drop in 10 min.

4) Stirrer. A variable-speed mechanical stirrer of any suitable type, equipped with a glass, propeller-type stirring paddle. A propeller with blades 6 mm in radius and set at a pitch of 30 to 45° is satisfactory. If electrical stirring apparatus is used, it must be electrically correct and grounded so that connecting or disconnecting the power to the motor will not produce a permanent change in meter reading during the course of the titration.

5) Burette. A 10 ml burette (E. figure 1) graduated in 0.05 ml division, and calibrated with an accuracy of ± 0.02 ml. The burette shall have a glass stopcock and shall have a tip that extends 100-130 mm beyond the stopcock.

6) Titration Beaker. A 250 to 300 ml beaker made of borosilicate glass.

7) Titration Stand. A suitable stand to support the electrodes, stirrer and burette in the position shown in figure 1. An arrangement that allows the removal of the beaker without disturbing the electrodes, burette, and stirrer is desirable.

6.2.5. Reagents

Reagent grade chemicals shall be used in the tests. Other grades may be used provided it is known that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

Unless otherwise indicated, references to water shall be understood to mean distilled water.

1) 2,4,6 Trimethylpyridine (y-Collidine) (mol wt 121.18) conforming to the following requirements :

Boiling range	168 to 170°C
Refractive index nb°	1.4982 ± 0.0005
Colour	colourless

Store the reagent over activated alumina and keep in a brown glass bottle.

2) m-Nitrophenol (mol wt 139.11) conforming to the following requirements:

Melting point	96 to 97°C
Colour	pale yellow

Store the reagent in a brown glass bottle.

3) Hydrochloric Acid Solution, Standard Alcoholic (0.1 N). Mix 9 ml of hydrochloric acid (HCl, sp gr 1.19) with 1 litre of anhydrous isopropyl alcohol. Standardize frequently enough to detect normality changes of 0.0005 by potentiometric titration of approximately 8 ml (accurately measured) of the 0.1 N alcoholic KOH solution diluted with 125 ml of CO_2-free water.

4) Hydrochloric Acid Solution, Standard Alcoholic (0.2 N). Prepare and standardize as directed in Paragraph (3), but use 18 ml of HCl (sp gr 1.19).

5) Potassium Hydroxide Solution, Standard Alcoholic (0.1 N). Add 6 g of potassium hydroxide (KOH) to approximately 1 litre of anhydrous isopropyl alcohol. Boil gently for 10 min to effect solution. Allow the solution to stand for two days and then filter the supernatant liquid through a fine sintered-glass funnel. Store the solution in a chemically resistant bottle. Dispense in a manner such that the solution is protected from atmospheric carbon dioxide (CO_2) by means of a guard tube containing soda lime or soda asbestos (Ascarite, Carbosorb or Indecarb), and such that it does not come into contact with cork, rubber or saponifiable stopcock grease. Standardize frequently enough to detect normality changes of 0.0005 by potentiometric titration of weighed quantities of potassium acid phtalate dissolved in CO_2-free water.

6) Potassium Hydroxide Solution, Standard Alcoholic (0.2 N). Prepare, store, and standardize as directed in Paragraph (5), but use 12 to 13 of KOH to approximately 1 litre of isopropyl alcohol.

7) Buffer Stock Solution A. Accurately weigh 24.2 ± 0.1 g of 2,4,6-trimethyl pyridine (y-collidine) and transfer to a 1 litre volumetric flask containing 100 ml of isopropyl alcohol. Using a 1 litre graduated cylinder add to the flask, while continuously stirring its contents, $150/N_1 \pm 5$ ml of 0.2 N alcoholic HCl solution (N_1 being the exact normality of the HCl solution found by standardization). Dilute to the 1000 ml mark with isopropyl alcohol, and mix thoroughly.

8) Buffer Stock Solution B. Accurately weigh 27.8 ± 0.1 g of m-nitrophenol and transfer to a 1 litre volumetric flask containing 100 ml of isopropyl alcohol. Using a 250 ml graduated cylinder, add to the flask while con-

tinuously stirring its contents, $50/N_2$ ± 1 ml of 0.2 N alcoholic KOH solution (N_2 being the exact normality of the KOH solution found by standardization). Dilute to the 1000 ml mark with iso-propyl alcohol, and mix thoroughly.

9) Buffer, Non-aqueous Acidic. Add 10 ml of buffer stock solution A to 100 ml of titration solvent. Use within 1 h.

10) Buffer, Non-aqueous Basic. Add 10 ml of buffer stock solution B to 100 ml of titration solvent. Use within 1 h.

11) Potassium Chloride Electrolyte. Prepare a saturated solution potassium chloride (KCl) in water.

12) Titration Solvent. Add 500 ml of toluene and 5 ml of water to 495 ml of anhydrous isopropyl alcohol. The titration solvent should be made up in large quantities, and its blank value determined daily by titration prior to use.

6.2.6. Preparation of electrodes

1. Maintenance of Electrodes. Clean the glass electrode at frequent intervals (not less than once every week during continual use) by immersing in cold chromic acid cleaning solution. Drain the calomel electrode at least once each week and refill with fresh KCl electrolyte. Maintain the electrolyte level in the calomel electrode above that of the liquid in the titration beaker at all times. When not in use, immerse the lower halves of the electrodes in water. Do not allow them to remain immersed in titration solvent for any appreciable period of time between titrations. While the electrodes are not extremely fragile, handle them carefully at all times.

2. Preparation of Electrodes. Before and after using, wipe the glass electrode thoroughly with a clean cloth, or a soft absorbent tissue, and rinse with water. Wipe the calomel reference electrode with a cloth or tissue, carefully remove the ground glass sleeve, and thoroughly wipe both ground surfaces. Replace the sleeve loosely and allow a few drops of electrolyte to drain through to flush the ground-glass joint. Wet the ground surfaces thoroughly with electrolyte, set the sleeve firmly in place, and rinse the electrode with water. Prior to each titration, soak the prepared electrodes in water for at least 5 min immediately before use, and touch the tips of the electrodes with a dry cloth or tissue to remove the excess water.

3. Testing of Electrodes. Test the meter-electrode combination when first put into use, or when new electrodes are installed, and retest at intervals thereafter by dipping the electrodes into a well-stirred mixture of 100 ml of the titration solvent and 1.0 to 1.5 ml of 0.1 N alcoholic KOH solution For the meter-electrode combination to be suitable for use, the potential between the electrodes should change by more than 0.480 V from the potential between the same electrodes when dipped in the non-aqueous acidic buffer solution.

   Considerably more sensitive electrodes are now available that will show a potential change of at least 0.590 V under these conditions, and their use is recommended.

6.2.7. Standardization of apparatus

Determination of Meter Readings for the Non-aqueous Buffer Solutions Corresponding to Acid and Base End Points - To ensure comparable selection of end points when definite inflection points are not obtained in the titration curve, determine daily, for each electrode pair, the meter readings obtained with the non-aqueous acidic and basic buffer solutions.

Prepare the electrodes as described in Section 6.2.6. Immerse them in the non-aqueous buffer solution, and stir for 5 min, maintaining the temperature of the buffer solution at a temperature within 2°C of that at which the titrations are to be made. Read the cell voltage. The readings so obtained are taken as the end points in titration curves having no inflexion points.

6.2.8. Preparation of sample of used fluid

Strict observance of the sampling procedure is necessary, since the sediment itself is acidic or basic or has absorbed acidic or basic material from the sample. Failure to obtain a representative sample causes serious errors.

As used fluid may change appreciably in storage, samples should be tested as soon as possible after removal from the lubricating system ; and the dates of sampling and testing should be noted in the original container until all of the sediment is homogeneously suspended in the fluid. If the original container is a can, or if it is glass and more than three fourths full, transfer the entire sample to a clear-glass bottle having a capacity at least one third greater than the volume of the sample. Transfer all traces of sediment from the original container to the bottle by violent agitation of portions of the sample in the original container.

After complete suspension of all sediment, strain the sample or a convenient aliquot through a 100 mesh screen for the removal of large contaminating particles.

Table 1. Size of sample

Acid number or base number	Size of sample, g	Sensitivity of weighing, g
0.05 to 1.0	20.0 ± 2.0	0.10
Over 1.0 to 5.0	5.0 ± 0.5	0.02
Over 5 to 20	1.0 ± 0.1	0.005
Over 20 to 100	0.25 ± 0.02	0.001
Over 100 to 250	0.1 ± 0.01	0.0005

6.2.9. Procedure for total acid number

Into the titration beaker, introduce a weighed quantity of sample as prescribed in Table 1 and add 125 ml of titration solvent. Prepare the electrodes as directed in Section 6.2.6. Place the beaker on the titration stand and adjust its position so that the electrodes are about half immersed. Start the stirrer, and stir throughout the determination at a rate sufficient to produce vigorous agitation without spattering and without stirring air into the solution.

Fill the burette with the 0.1 N alcoholic KOH solution and place the burette in position in the titration assembly, taking care that the tip is immersed about 25 mm in the liquid in the beaker. Record the initial burette and meter (cell potential) readings. Add suitable small portions of 0.1 N alcoholic KOH solution and, after waiting until a constant potential has been established, record the burette and meter readings. At the start of the titration and in any subsequent regions (inflexions) where 0.1 ml of 0.1 N KOH solution consistently produces a total change of more than 0.03 V (corresponding to 0.5 pH scale units) in the cell potential, add 0.05 ml portions. In the intermediate regions (plateaus) where 0.1 ml changes the cell potential less than 0.03 V, add larger portions sufficient to produce a total potential change approximately equal to, but not greater than 0.03 V. Titrate in this manner until the potential changes less than 0.005 V (corresponding to 0.1 pH scale units) per 0.1 ml and the cell potential indicates that the solution is more basic than the non-aqueous basic buffer. Remove the titrated solution, rinse the electrodes with isopropyl alcohol, and immerse the electrodes in distilled water.

Consider the cell potential constant when it changes less than 0.005 V (corresponding to 0.1 pH scale units) per min. This may require approximately 1 to 2 min per 0.1 V change in potential (corresponding to 1.7 pH scale units) when adding 0.05 ml increments ; 1 ml increments may require 3 to 5 min.

Blank - For each set of samples, make a blank titration of 125 ml of titratio solvent, adding 0.1 N alcoholic KOH solution in 0.05 ml increments and recording meter and burette readings when the former becomes constant after each increment.

6.2.10 Procedure for total base number

Proceed as directed above, but titrate with 0.1 N alcoholic HCl solution ; add the HCl at the same rate as specified for the KOH solution.

Blank - For each set of samples, make a blank titration of 125 ml of titration solvent, adding 0.1 N alcoholic HCl solution in 0.05 ml increments in a manner comparable to that specified in the previous section.

6.2.11. Results

Plot the volumes of the acid or base titrating solution, or both, added against the corresponding meter readings (see figure 2). Mark as an end point any inflexion point on the curve that occurs near the cell voltages representing the non-aqueous acidic and basic buffers. If no inflexion appears, mark the end points at those meter readings corresponding to the two non-aqueous buffers.

Calculate the total acid number as follows :

$$\text{Total acid number, mg KOH per g} = \frac{(A-B) \times N \times 56.1}{W}$$

where :

A = millilitres of alcoholic KOH solution used to titrate the sample to an end point that occurs at the meter reading corresponding to the basic non-aqueous buffer.

B = volume corresponding to A for the blank titration ;

N = normality of the alcoholic KOH solution ;

W = grams of sample.

Calculate the total base number as follows :

$$\text{Total base number, mg KOH per g} = \frac{(E-F) \times N \times 56.1}{W}$$

Calculate the total base number as follows :

$$\text{Total base number, mg KOH per g} = \frac{(E-F) \times N \times 56.1}{W}$$

where :

E = millilitres of alcoholic HCl solution used to titrate the sample to an end point which occurs at a meter reading corresponding to the acidic non-aqueous buffer :

F = volume corresponding to E for blank titration ;

N = normality of the HCl solution ;

W = grams of sample.

6.2.12. Report

Report the results as Total Acid Number or where applicable Total Base Number.

6.2.13. Acceptance criteria

The neutralisation number of HFB and HFD fluids are determined for information purposes and no acceptance criteria are specified.

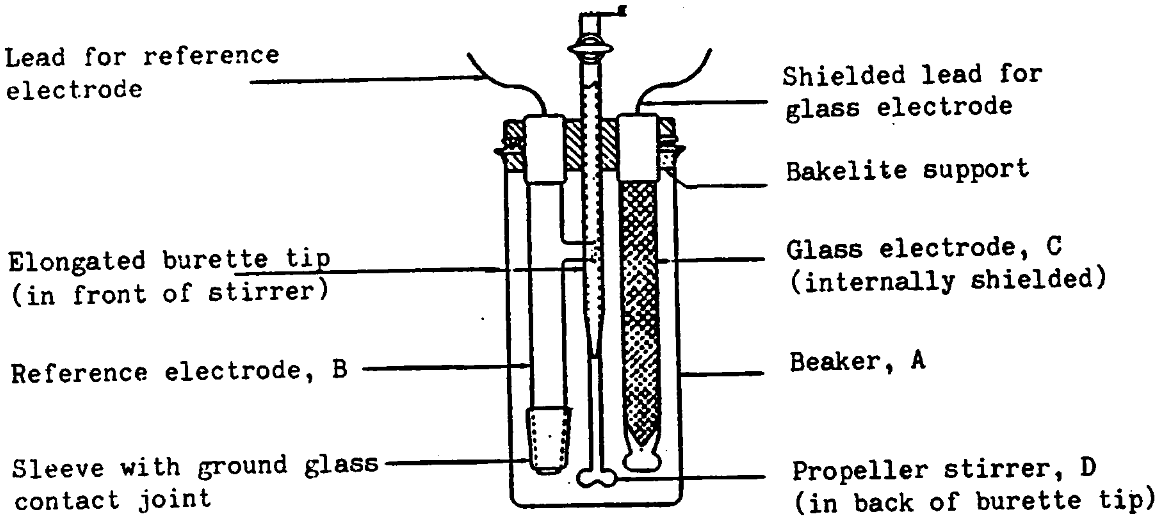
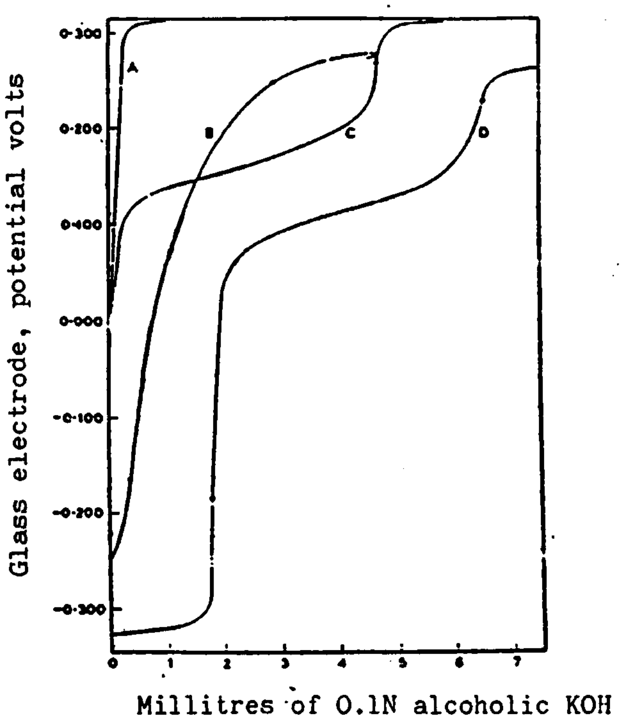


Figure 1 Cell for potentiometric titration



Titration blank

Curve without inflexion point. The end point is chosen at a meter reading which was obtained for the non-aqueous basis buffer.

Curve with a single inflexion point. The end point is chosen at the most vertical portion of the inflexion.

Curve with two inflexion points. The end point is chosen at the most vertical portion of the inflexion which is nearest to the meter reading which was obtained for the non-aqueous basic buffer

Note : On some meters the voltage sign is reversed.

Figure 2 Examples of titration curves for Total Acid Number

6.3. METHOD OF TEST FOR VISCOSITY INDEX OF TYPE HFD FLUIDS

6.3.1. Scope

This test shall be applied to type HFD fluids only, to provide an arbitrary measure of the change in kinematic viscosity with changes in temperature.

6.3.2. Procedure

Viscosity index shall be calculated from measurements of kinematic viscosity at 40°C and 100°C by ISO method 2909-1975, or by a technically equivalent method such as BS 4459, IP 226/77 or ASTM D 2270.75. If the calculation results in a negative value for viscosity index, then this value shall still be reported.

The required kinematic viscosities shall have been determined in accordance with one of the methods given in Section 5.1.

6.3.3. Acceptance criteria

Viscosity index shall be determined for information purposes only and no acceptance criteria are specified. The manufacturer shall state the viscosity index when determined by the above method.

6.4. METHOD OF TEST FOR POUR POINT

Note : The method given in sections 6.4.1 to 6.4.5 is technically equivalent to ISO 3016, AFNOR NFT 60-105, DIN 51 597, BS 4452, IP 15/67 and ASTM D 97-66. The procedure given in Section 6.4.6. is technically equivalent to the rapid pour point method given in Appendix A1 of IP 15/67.

6.4.1. Scope

The pour point shall be determined for all types of fire-resistant fluid, except type HFA 1 , using either the procedure given in section 6.4.5 or the procedure given in section 6.4.6. It is recommended that the procedure for Rapid Pour Point given in section 6.4.6 should be employed wherever possible, i.e. when the pour point is below about -18°C.

6.4.2. Summary of method

The sample is cooled at a specified rate and examined at intervals of 3°C for flow characteristics. The lowest temperature at which movement of the fluid is observed is recorded as the pour point.

6.4.3. Apparatus

The following apparatus shown in figure 1 shall be used :

a) Test Jar. A cylindrical test jar of clear glass, flat bottom, approximately 30 to 33.5 mm in inside diameter and 115 to 125 mm in height. To indicate sample height the jar should be marked with a line 54 ± 3 mm above the inside bottom.

b) Thermometers. Thermometers having ranges as shown below and conforming to the requirements as prescribed in ASTM specifications E 1, (Note 1) or in the specification for IP standard thermometers :

Thermometer	Temperature range	Thermometer No ASTM	IP
High cloud and pour	-38 to +50°C	5 C	1 C
Low cloud and pour	-80 to +20°C	6 C	2 C

c) Cork. A cork to fit the test jar, bored centrally to take the test thermometer.

d) Jacket. A water tight cylindrical jacket of glass or metal, flat bottom, about 115 mm in depth, with inside diameter 9.5 to 12.5 mm greater than the outside diameter of the test jar.

e) Disk. A disk of cork of felt 6 mm in thickness and of the same diameter as the inside of the jacket.

f) Gasket. A ring gasket about 5 mm in thickness, to fit snugly around the outside of the test jar and loosely inside the jacket. This gasket may be made of cork, felt, or other suitable material, elastic enough to

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Note 1 : Annual book of ASTM Standard, Parts 25 and 44.

cling to the test jar and hard enough to hold its shape. The purpose of the ring gasket is to prevent the test jar from touching the jacket.

g) Bath. A cooling bath of a type suitable for obtaining the required temperatures. The size and shape of the bath are optional but a support, suitable for holding the jacket firmly in a vertical position, is essential. For determination of pour points below 10°C two or more baths are needed. The required bath temperatures may be maintained by refrigeration if available, otherwise by suitable freezing mixtures, (Note 2).

Note 2 : The freezing mixtures commonly used are as follows :

	For temperatures down to
Ice and water	10°C
Crushed ice and sodium chloride crystals	-12°C
Crushed ice and calcium chloride crystals	-26°C
Solid carbon dioxide and acetone or petroleum naphtha *	-57°C

*This mixture may be made as follows :

In a covered metal beaker chill a suitable amount of acetone or petroleum naphta to -12°C, or lower, by means of an ice-salt mixture. Then add to the chilled acetone or petroleum naphta enough solid carbon dioxide to give the desired temperature.

Solid carbon dioxide is commercially available in many areas. If necessary, it may be prepared as follows : Invert a cylinder of liquid carbon dioxide and draw off carefully into a chamois skin bag the desired amount of carbon dioxide, which through rapid evaporation will quickly become solid.

6.4.4. Sampling

A sample of not less than 200 ml shall be supplied for test. Care shall be taken that the sample supplied for test is representative of the bulk. The sample shall be kept for 24 h at room temperature prior to testing.

6.4.5. Procedure

a) Pour the fluid into the test jar to the level mark. Close the test jar tightly by the cork carrying the high pour thermometer.

Adjust the position of the cork and the thermometer so the cork fits tightly, the thermometer and the jar are coaxial, and the thermometer bulb is immersed so the beginning of the capillary is 3 mm below the surface of the fluid (Note 3).

b) Subject the fluid in the test jar to the following preliminary treatment : Fluids having pour points between 32°C and -34°C. Heat the fluid, without stirring to 46°C in a bath maintained at 48°C. Cool the fluid to 35°C in air or in a water bath at approximately 25°C. Proceed as directed in paragraph (c) below.

Fluids having pour points below -34°C. Heat the fluid as directed above and cool to 16°C in a water bath maintained at 7°C. Remove the high cloud and pour thermometer and place the low cloud and pour thermometer in position. Proceed as directed in paragraph (c) below.

c) Place the disk in the bottom of the jacket. Place the ring gasket around the test jar, 25 mm from the bottom. The disk, gasket, and inside of the jacket shall be clean and dry. Insert the test jar in the jacket.

Note 3 : Since separation of the mercury, or toluene thread of cloud and pour thermometers occasionally occurs, and since such separation may otherwise escape immediate detection, it is suggested that the ice points of the thermometers be checked immediately prior to the test. Any thermometer that shows an ice point differing from 0°C by more than 1°C should be further examined and/or recalibrated before use.

d) Maintain the temperature of the cooling bath at -1° to +2°C. Support the jacket, containing the test jar, firmly in a vertical position in the cooling bath so that not more than 25 mm of the jacket projects out of the cooling medium.

e) During cooling take great care not to disturb the mass of the fluid nor permit the thermometer to shift in the fluid ; any disturbance may lead to low and fictitious results.

f) Beginning at a temperature 11°C above the expected pour point, at each test thermometer reading that is a multiple of 3°C remove the test jar from the jacket carefully and tilt it just enough to ascertain whether there is a movement of the fluid in the test jar. The complete operation of removal and replacement shall require not more than 3 sec. If the fluid has not ceased to flow when its temperature has reached 10°C, transfer the test jar to another jacket in a second bath maintained at a temperature of -18 to -15°C (Note 4). If the fluid has not ceased to flow when its temperature reached -7°C, transfer the test jar to another jacket in a third bath maintained at a temperature of -34 to -32°C. For the determination of very low pour points, additional baths are required, each bath to be maintained at 17°C below the temperature of the preceding bath. In each case, transfer the test jar when the temperature of the fluid reaches a point 28°C above the temperature of the new bath. Never place the cold test jar directly in the cooling medium. As soon as the fluid in the test jar does not flow when the jar is tilted, hold the test jar in a horizontal position for exactly 5 s, as noted by a stop watch or other accurate timing device, and observe carefully. If the fluid shows any movement under these conditions, replace the test jar immediately in the jacket and repeat a test for flow at the next temperature 3°C lower.

g) Continue the test in this manner until a point is reached at which the fluid in the test jar shows no movement when the test jar is held in a horizontal position for exactly 5 s. Record the observed reading of the test temperature.

6.4.6. Procedure for Rapid Pour Point

The following procedure may be applied to fluids which have a pour point of -18°C or lower.

a) Pour the sample, which should be at approximately laboratory temperature,

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Note 4 : The jacket may be left in the bath or transferred with the test jar.

into the test jar to a height 51 to 57 mm. Close the jar with the cork carrying a low cloud and pour thermometer so that the thermometer bulb is immersed vertically in the sample with the beginning of the capillary 3 mm below the surface.

b) Immerse the jar directly in a freezing mixture maintained at approximately -23°C in a vertical position so that the surface of the fluid is about 25 mm below the level of the freezing mixture. When the fluid has attained a temperature of approximately -7°C remove the jar from the freezing mixture, carefully wipe dry, fit the gasket on to the jar 25 mm from the bottom, and insert the jar into the jacket. The disk, the gasket and the jacket must be kept clean and dry.

c) Support the jacket and the jar in a vertical position in a bath maintained at a temperature of -57°C so that not more than 25 mm projects from the cooling medium. Beginning at a temperature about 12°C above the expected pour point of the fluid, at each fluid temperature that is a multiple of 3°C, carefully remove the jar from the jacket and tilt it just enough to see whether the fluid moves, and replace it ; this complete operation must not take more than 3 s.

d) If the sample remains fluid at a temperature of -39°C transfer to a further bath maintained at -68°C. Continue inspection as in (a) above.

e) As soon as the sample does not flow when the jar is tilted, hold the jar in the horizontal position for exactly 5 s. If the fluid shows any movement replace the jar in the jacket and cool the fluid a further 3°C and test again. If the fluid shows no movement during the 5 s, record the reading of the thermometer.

6.4.7. Results

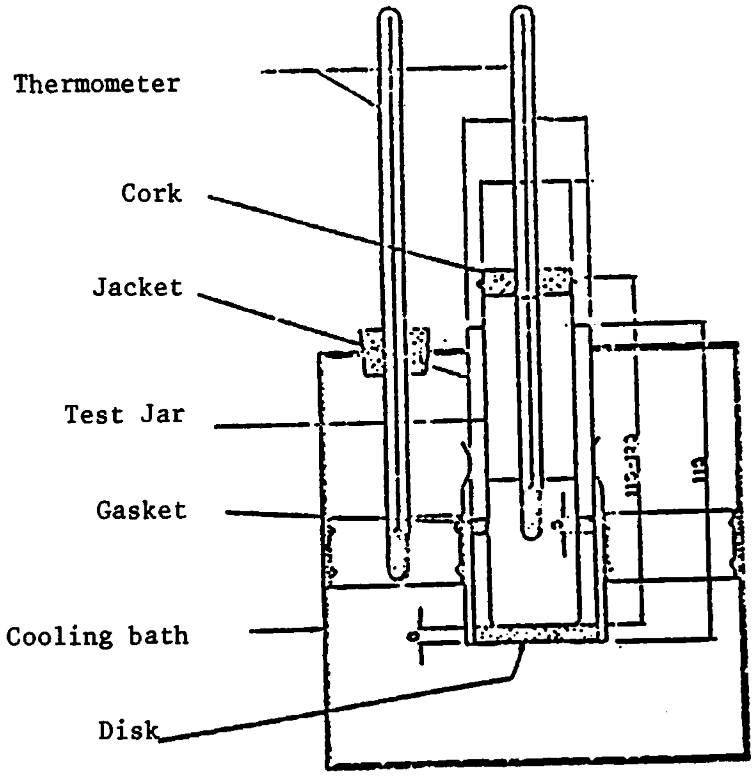
For the procedure given in section 6.4.5. above add 3°C to the temperature recorded in paragraph (g) of section 6.4.5. and report the result as the Pour Point.

For the procedure given in section 6.4.6. above add 3°C to the temperature recorded in paragraph (e) of section 6.4.6. and report the result as the Rapid Pour Point.

6.4.8. Acceptance criteria

Pour Point should be determined for information purposes only and no acceptance criteria are specified. The manufacturer shall state the Pour Point or the Rapid Pour Point when measured by the above method.

PART VI 6.4.



All dimensions in millimeters

Figure 1 Apparatus for pour point test

6.5. METHOD OF TEST FOR OIL CONTENT OF TYPE HFA FLUIDS

Note : The method given in this Section is technically equivalent to BS 5091 and IP 137.

6.5.1. Scope

The method described below shall be used where an accurate determination of the oil content of oil-in-water emulsions of type HFA is required, for example when adjusting automatic proportioning equipment. The oil content as determined by this method will generally be somewhat lower than the actual concentration of emulsifying oil present in the fluid. A procedure for estimating emulsifying oil concentration from measurements of oil content is given in section 6.5.8..

An approximate measurement of oil content can conveniently be obtained, in some cases, by using a pocket refractometer. A note on the use of pocket refractometers for this purpose is given in section 6.5.9..

6.5.2. Principle

A known volume of the fluid is treated with hydrochloric acid in a graduated flask and the volume of liberated oil is measured.

The oil content is defined as the amount of material which separates under the conditions of this test as an oily layer insoluble in the aqueous phase. In addition to the mineral oil originally present in the fluid the separated material may also include portions of those emulsifiers which are decomposed by the acid treatment.

6.5.3. Apparatus

Hirschsohn Flask - of 150 ml capacity and with a 10 ml graduated neck as shown in figure 1. or, alternatively, flasks to DIN 51368.

6.5.4. Reagents

Hydrochloric Acid, approximate molarity 2.4 M - prepared by diluting one volume of concentrated hydrochloric acid (specific gravity 1.18) with four volumes of distilled water.

6.5.5. Sampling

A sample of not less than 250 ml should be supplied for test. Care shall be taken to ensure that the sample is representative of the bulk.

6.5.6. Procedure

Measure 100 ml of the sample from a 100 ml measuring cylinder into the graduated flask and add 40 ml of the hydrochloric acid. When the fluid is known to have an oil content of more than 10%, or when a volume of oil of more than 10 ml is found to separate as a result of the procedures described in sections 6.5.6 (b) and (c), measure a smaller quantity of the fluid such as to give a convenient volume of separated oil ; add sufficient hydrochloric acid to give a total volume of approximately 140 ml.

Swirl the flask until its contents are thoroughly mixed and heat it in a boiling water-bath for 1 h. Remove the flask from the bath and fill it to the top graduation with the hydrochloric acid. Allow the flask to stand for 1 h, swirling it from time to time to detach any oil adhering to the sides.

Adjust the oil level if necessary by further addition of the hydrochloric acid. Determine the difference in the scale readings corresponding with the top edge of the meniscus at the upper and lower levels of the separated oil and record the volume to the nearest 0.05 ml.

6.5.7. Calculation and reporting

Calculate the result as a percentage by volume and report it to the nearest 0.1% as the Oil Content.

6.5.8. Estimation of emulsifying oil concentration

The oil content, as determined by the above method, will generally be somewhat lower than the actual concentration of emulsifying oil present in the fluid. The emulsifying oil concentration of a particular fluid can be estimated from an oil content measurement by means of a calibration graph. This should be constructed from measurements of oil content carried out on fluid samples of known emulsifying oil concentration prepared with the same emulsifying oil.

The calibration will be specific to a particular emulsifying oil and different calibration graphs will generally be required for use with different fluids.

6.5.9. Oil content determination by pocket refractometer

In many cases the emulsifying oil concentration can be readily assessed with sufficient accuracy for the purposes of routine checking of hydraulic systems by using a pocket refractometer. Refractometers which incorporate a scale calibrated in percentage sugar can give a direct reading of percentage emulsifying oil concentration by volume, which would be accurate to within ± 1 for a large number of fluids. However, before applying this instrument to a particular fluid, the validity of its use should be checked over the range of oil content that would be encountered in service, by testing emulsions of known concentration for that particular fluid. It should also be noted that the presence of significant amounts of materials other than the emulsifying oil and water, such as for example antifreeze solutions, may give rise to erroneous results. (Antifreeze solutions, which are used to protect components from freezing during storage on the surface, may be present in powered roof support systems).

6.5.10. Acceptance criteria

Most type HFA fluids are mixed by the user to suit a particular application and, in consequence, no acceptance criteria for oil content can be specified.

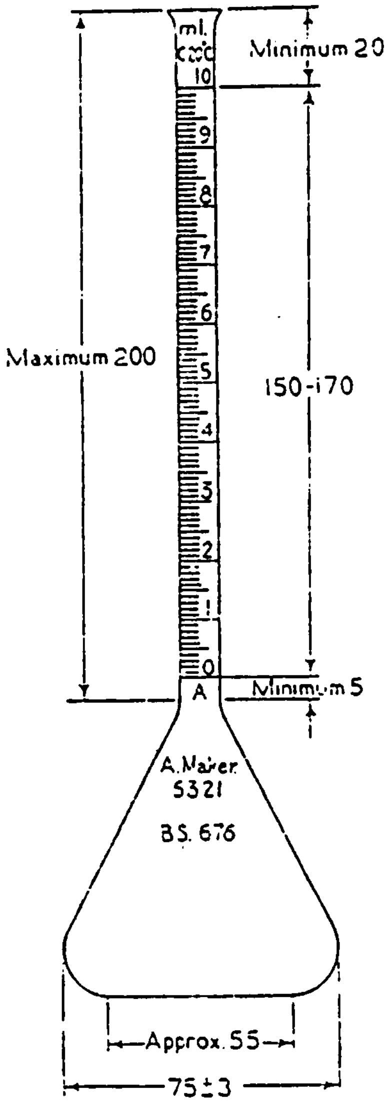


Fig. 2 150 ml flask with 10 ml scale

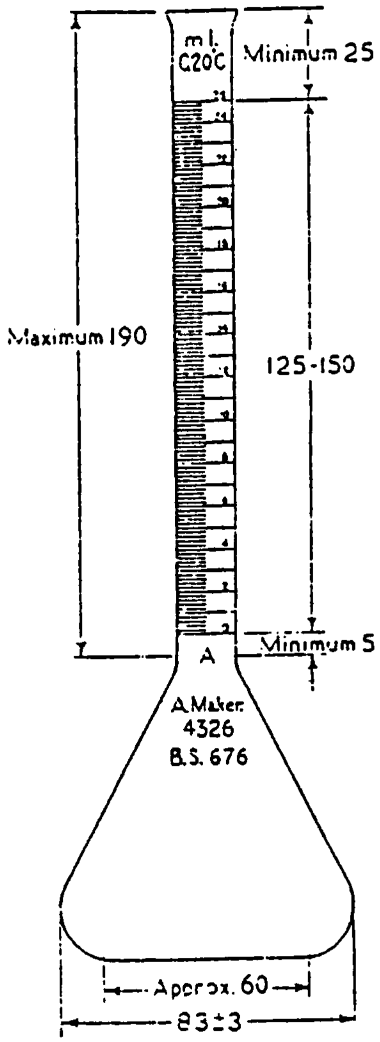


Fig. 3 200 ml flask with 25 ml scale

All dimensions are in millimetres

Figure 1 Hirschsohn flasks

Note: Where Hirschsohn flasks to the above dimensions are not available a suitable alternative would be flasks to DIN 51368.

6.6. METHOD OF TEST FOR THE FILTERABILITY OF FLUIDS

6.6.1. Scope

This method may be used to assess the filterability of mineral oils and fire-resistant fluids.

6.6.2. Principle

The method consists of finding the volume of fluid which passes through an ultra-fine filter of a given type. From this, the index of filterability is calculated.

6.6.3. Apparatus

Figure 1 shows the apparatus used ; it includes a graduated conical flask with a capacity of 1 litre which can withstand a vacuum of 71 cm of mercury, to which it is connected.

The filter membrane composed of a mixture of cellulose esters ; 47 mm in diameter ; surface for filtration 9.2 cm^2 ; pore diameter 1.2 um.

Mixing apparatus which includes a graduated beaker of capacity 1,000 ml ; and a stirrer of stainless steel having the following dimensions

length	165 mm
width	35 mm
thickness	2 mm.

This stirrer is brazed onto a 10 mm rod and is rotated at 1,500 rev/min. The stirrer is rotated about the longitudrival axis of the measuring cylinder, with the bottom of the stirrer 1 cm from the bottom of the fluid.

Vacuum drier.
Stop watch (to 0.1 s).

6.6.4. Preparation of the sample

a) 1 litre of fluid is used as it is received from the manufacturer.

b) In addition a sample is prepared using the fluid added to distilled water in the ratio 1 to 99 parts. Use the stirrer described above to mix this for 5 min. Evacuate the occluded air by applying the vacuum.

c) Prepare a 2 to 98 mixture in the same way.

d) Clean the whole of the apparatus.

6.6.5. Procedure

Having inserted the 1.2 µm filter in the test apparatus, the litre of undiluted fluid is put into the filter funnel. The test is completed at ambient temperature of 19 to 26°C, and the vacuum is applied, checking that this is 71 cm of mercury.

The time T is measured from the instant that the vacuum is applied until the flow rate through the filter falls to 1 drop every 2 s. The volume of fluid which is collected in the flask is measured at the end of this time.

Then the apparatus is emptied and cleaned and the 1% mixture is tested in the same way. After re-cleaning the apparatus, the 2% mixture is tested.

Note : For certain fluids, cellulose ester filters may not be satisfactory (i.e. the fluid attacks the filter) and in these cases, Teflon type filters should be used.

6.6.6. Results

For each fluid tested, the volume of fluid passing through the filter and the time is recorded for :

a) the non-diluted fluid;

b) the 1% mixture;

c) the 2% mixture;

The volume V in ml is measured, the time T in hours and from this, the factor of filterability is calculated

$$(FF) = \frac{V\text{ml}}{S}$$

where S = surface area of filter, cm^2

The filterability index is calculated as

$$FI = \frac{FF\ (\text{ml}/\text{cm}^2)}{T.\ (\text{hours})}$$

The curve of filterability index against water content of the fluid is drawn.

PART VI 6.6

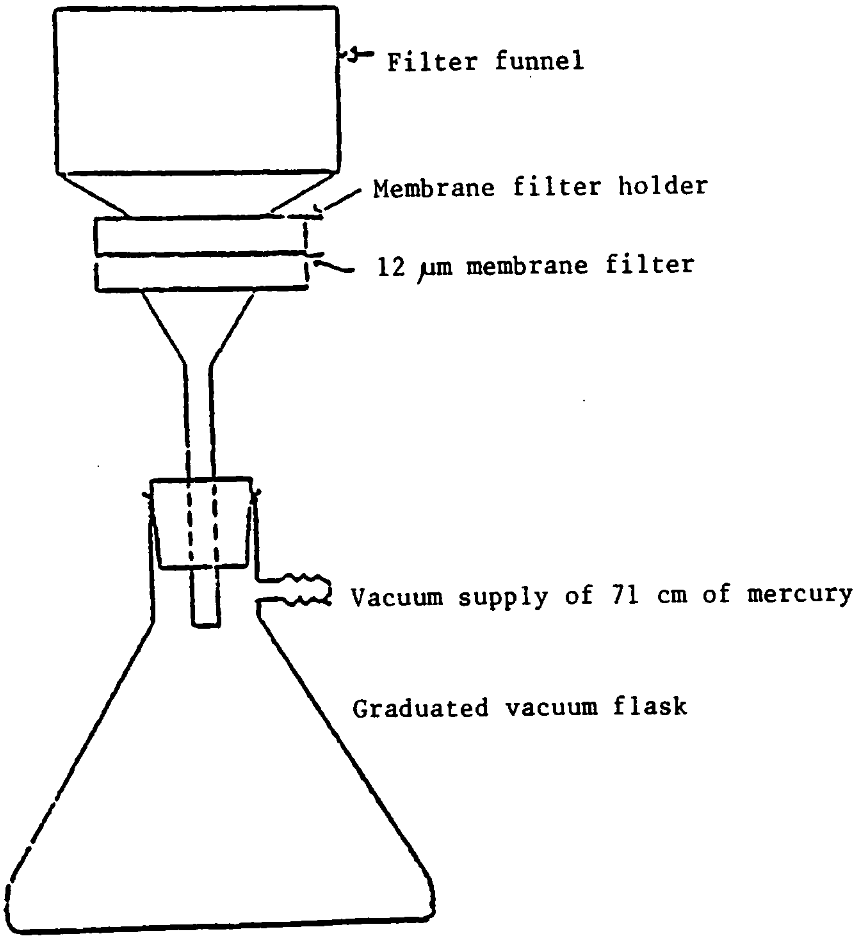


Figure 1 Filterability test rig

6.7 METHODS OF TEST FOR THE THERMAL STABILITY OF TYPE HFD FLUIDS

Methods of test for the ageing properties of types HFC and HFD fluids intended for hydrostatic applications are described in section 5.3. In this section are included those tests for the thermal stability of type HFD fluids intended for hydrokinetic applications.

Three tests are described. Section 6.7.1 details a method of test for thermal stability which may be carried out in a laboratory using conventional laboratory glassware. Although designated a "discretionary test", defined acceptance criteria are included as a guide to the interpretation of the results of the various tests.

The tests described in sections 6.7.2 and 6.7.3 differ in that they involve the testing of fluids for their behaviour in a traction type and a scoop controlled coupling respectively. These tests, therefore, bear a direct relationship with the intended conditions of use of these fluids. As with the test described in section 6.7.1, acceptance criteria are given as a guide to the interpretation of the results.

6.7.1 METHOD OF TEST FOR THERMAL STABILITY OF TYPE HFD FLUIDS

6.7.1.1 Scope

This test shall be applied to type HFD fluids which are intended for use in hydrokinetic applications to assess the degree of fluid degradation which may occur at high temperatures.

6.7.1.2 Principle

Samples of the fluid are subjected to heat ageing for various periods of test at three different test temperatures. Air is bubbled through the fluid during the period of test. Various fluid properties are measured before and after the heat ageing treatment.

6.7.1.3 Apparatus

a) Heating vessel - 2 litre round bottom flask with two necks. One neck shall be fitted with a thermometer, a temperature sensing element for use in conjunction with the temperature controller (c) and an air nozzle, as shown in figure 1. The other neck shall be fitted with a water cooled reflux condenser of effective length 350 to 450 mm.

b) Electric heating mantle - suitable for use with the heating vessel, recommended heating power 450 w.

c) Temperature controller and sensing element - suitable for regulating the output of the heating mantle at the temperature of test.

d) Air supply - capable of maintaining a continuous flow of 200 ± 30 ml/min and fitted with a metering control.

e) Air flow measuring instrument - suitable for measuring an airflow of 200 ml/min. A rotameter is recommended.

6.7.1.4 Sampling

A sample of not less than 10 litres of fluid shall be supplied for test. Care should be taken to ensure that this sample is representative of the bulk fluid.

6.7.1.5 Procedure

The following procedure shall be carried out for each of the test conditions given in the Table below using a fresh sample of fluid for each test condition.

Conditions of Testing

Temperature of Test	Periods of Test hours		
250°C	20*	30*	40*
149°C	72	144	216*
93.3°C	144	288	432*

* - see section 6.7.1.6

Determination of fluid properties

The heating vessel shall be filled with 850 ml of fluid. The thermometer and air nozzle shall be positioned as shown in figure 1 and the sensing element of the temperature controller shall be positioned close to the bulb of the thermometer. The reflux condenser shall be fitted to the heating vessel and the cooling water shall be passed through the condenser.

The fluid shall then be heated to the test temperature, using the heating mantle. On reaching the test temperature, air shall be bubbled through the fluid from the air nozzle at a rate of 200 ml/min and the timing of the period of test shall begin. The fluid shall be maintained within ± 2°C of the test temperature for the period of test by the operation of the temperature controller (see note 1). At the end of the test period the heating and air bubbling shall be discontinued and the fluid allowed to cool naturally to room temperature. The tests enumerated below for the determination of fluid properties shall then be carried out.

Note 1 : The maximum heater power (450 W) shall be used for most of the initial heating up period. However, in order to avoid an excessive initial temperature overshoot it is recommended that the heater power be reduced by means of a regulator shortly before the test temperature is reached. This reduction of power will also assist in reducing the temperature oscillations which may occur if an on-off type of controller is used.

6.7.1.6 Determination of fluid properties

The following fluid properties shall be measured for a sample of untreated fluid and for samples of fluid subjected to heat ageing for all of the test conditions given in the table above (see section 6.7.1.5).

1. Total Acid Number (or, if appropriate, the Total Base Number) by the potentiometric titration method described in section 6.2.
2. Kinematic Viscosity at 40°C and 100°C by one of the methods of test described in section 5.1.
3. Density at 15°C by the method described in section 6.1.

The fire resistant properties of the fluid shall be measured for a sample of untreated fluid and for samples of fluid subjected to heat ageing for those test conditions in the table which are marked with an asterisk (*). The Wick Test described in section 3.3.2. shall be used for this purpose.

6.7.1.7 Results

The results of all the determinations required by the above method of test together with the percentage changes in specific gravity and viscosity shall be reported.

6.7.1.8 Acceptance criteria

When tested in accordance with the above method type HFD fluids which are intended for use in hydrokinetic applications shall satisfy the following requirements :

Temperature of test	Time of test Hours	Total acid number maximum value	Kinematic viscosity at 40°C maximum percentage increase	Kinematic viscosity at 100°C maximum percentage increase	Density at 15°C maximum percentage change
250°C	20	80	1400	300	3.0
250°C	30	120	2100	450	4.0
250°C	40	160	2800	600	5.0
149°C	72	2.0	4.0	3.0	1.5
149°C	144	3.5	7.0	5.0	2.0
149°C	216	5.0	10.0	7.0	2.5
93.3°C	144	1.0	2.0	2.0	1.0
93.3°C	200	1.5	3.0	3.0	1.5
93.3°C	432	2.0	4.0	4.0	2.0

The Wick Test results shall satisfy the requirements specified in section 3.3.2. of this report.

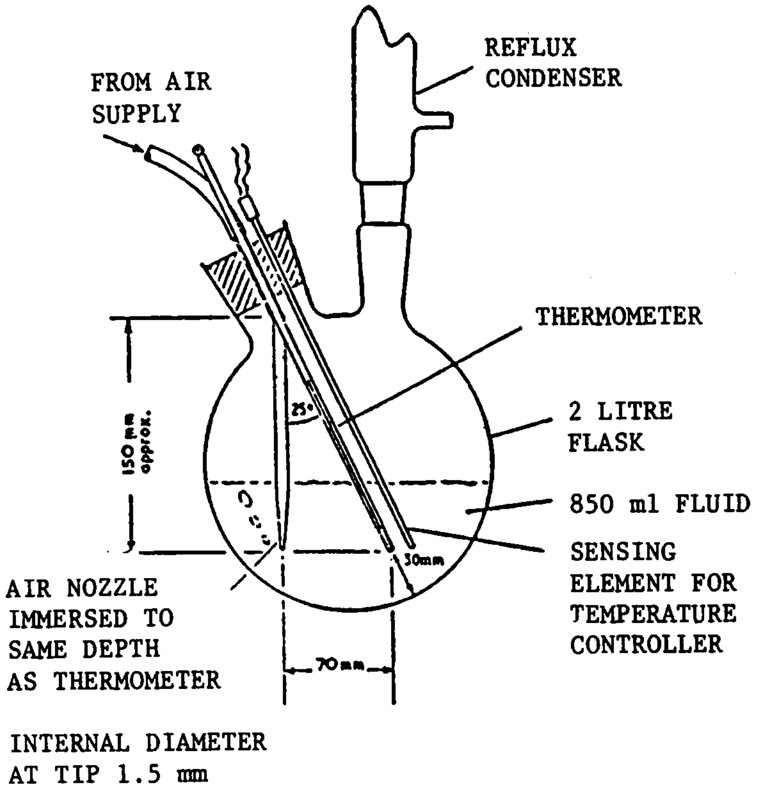


Figure 1 Apparatus for testing thermal stability of HFD fluids

6.7.2 METHOD OF TEST FOR THERMAL STABILITY OF TYPE HFD FLUIDS IN TRACTION COUPLINGS

6.7.2.1 Scope

The object of the test is to assess the suitability of a fluid for use in traction type couplings by examining the change in selected properties of the fluid during a controlled thermal cycling test. The test shall only be applied to type HFD fluids which are intended for use in hydrokinetic applications.

6.7.2.2 Principle

The thermal stability of a fluid in a traction type coupling is assessed by repeatedly cycling the fluid in a working coupling through the temperature range 80 to 130°C simulating the conditions occuring in a severe service application. Samples of the fluid are taken at intervals throughout the test, and at the end of the test, are examined for changes in viscosity, base number or acidity and fire-resistance.

6.7.2.3 Apparatus

a) One Fluidrive Engineering Co. Ltd. STC 450 coupling.

b) One 90 kW four-pole induction motor having a nominal speed of 154 rad/s (1470 rev/min) at full load capable of producing a pull-out torque in excess of 2.25 times full load torque : the speed at twice full load torque must exceed 147 rad/s (1400 rev/min).

c) One compressed-air operated disc brake capable of applying a resistive torque of at least 1350 Nm.

d) One continuously rated power absorber capable of applying a resistive torque of 700 Nm and dissipating in excess of 100 kW.

e) One torque transducer capable of measuring up to 2000 Nm to an accuracy of ± 2% of the measured value at a shaft speed of 154 rad/s (1470 rev/min).

f) One temperature transducer, FM telemetry link and decoder capable of measuring the temperature of the fluid within the coupling over the range 70 to 140°C to an accuracy of ± 2°C while the coupling is rotating at 154 rad/s (1470 rev/min).

g) One tachometer or equivalent device to measure motor shaft speed.

h) Solenoid-operated compressed-air valves and electronic timing circuits arranged to apply the disc brake for preset periods at preset intervals throughout the test.

The test rig is constructed so that the induction motor drives the fluid coupling, the output shaft of which is connected through the torque transducer to the disc brake and the power absorber.

6.7.2.4 Procedure

The traction coupling is filled with 11 l of the test fluid.

The drive motor is started and the disc brake applied to stall the output shaft for 20 s at regular intervals of 15 min. The duration of the stall is gradually increased until the fluid temperature at the end of each stall period is about 120°C. The resistance of the power absorber is then gradually increased until the fluid temperature immediately before each stall period is about 75°C. Further adjustment of the duration of the stall and the power absorber loading is then continued until a cycle is achieved in which the fluid temperature at the end of each stall is 130 ± 2°C and the temperature immediately before each stall is 80 ± 2°C. The thermal cycle is repeated continuously for a total period of 1,000 h.

Fluid samples of 0.5 l are taken from the new fluid and after each 100 h period during the test. The quantity of fluid removed from the coupling is replaced by an equal volume of new fluid to keep the total volume constant. The viscosity and base number or acidity of each fluid sample is determined. In addition 0.5 l samples are taken from the new fluid and from the fluid in the coupling after completion of the 1000 h test period ; these samples are checked for fire-resistant using the Wick Test detailed in section 3.3.2. of this report.

6.7.2.5 Results

The following data shall be recorded :

a) Fluid type, precise designation and batch number.

b) Total test duration.

c) The actual intervals at which fluid samples were taken.

d) For the new fluid samples and for each sample taken during the test :

  i) Kinematic viscosity in cSt at 40°C and 100°C by one of the methods detailed in section 5.1.

  ii) Fluid base number or acidity measured in mg of KOH/g in accordance with section 6.2.

e) For the Wick Tests for fire-resistance on the new fluid sample and on a sample taken at the end of the test in accordance with section 3.3.2. of this report :

  i) All six results for each of the five exposure times.

  ii) The average of each set of six results.

  iii) The largest of the averages in (ii) taken as the Mean Persistence of Flame.

6.7.2.6 Acceptance criteria

This test is carried out for information purposes only and no acceptance criteria are laid down. However, as a guide to the interpretation of test results it would be expected that the fluid would comply with the following :

a) Viscosity

The viscosity of the fluid in cSt at 40°C should not vary by more than 15% of the initial value during the 1,000 h period.

b) Base number or acidity

The total base number or acidity value of the fluid measured in mg of KOH/g should not show a reduction in base number or an increase in acidity of more than 1 mg of KOH/g during the 1,000 h test period.

c) Fire-resistance

The fluid shall remain fire-resistant on completion of the 1,000 h test period. The mean persistence of flame should be less than 60 s (in accordance with the requirements of section 3.3.2. of this report).

6.7.3 METHOD OF TEST FOR THERMAL STABILITY OF TYPE HFD FLUIDS IN SCOOP CONTROLLED COUPLINGS

6.7.3.1 Scope

The object of the test is to assess the suitability of a fluid for use in scoop control fluid couplings by examining the change in selected properties of the fluid during a controlled period of operation at a continuous high temperature.

The test shall only be applied to type HFD fluids which are intended for use in hydrokinetic applications.

6.7.3.2 Principle

The thermal stability of a fluid in a scoop control fluid coupling is assessed by subjecting the fluid to a period of operation in a working coupling loaded to cause the temperature in the external scoop circuit to be maintained at 121 ± 2°C for 300 h. Samples of fluid taken at intervals throughout the test and at the end of the test are examined for changes in viscosity, base number or acidity and fire-resistance.

6.7.3.3 Apparatus

a) One Fluidrive Engineering Co. Ltd. Size 18 Type SCR 4 scoop control fluid coupling.

b) One 120 kW four pole induction motor having a nominal speed of 154 rad/s (1470 rev/min) at full speed.

c) One continuously rated power absorber capable of applying a resistive torque of 800 Nm and of dissipating in excess of 120 kW.

d) One torque transducer capable of measuring up to 1000 Nm to an accuracy of ± 2% of the measured value at a shaft speed of 154 rad/s (1470 rev/min).

e) Two electronic tachometers arranged to read either input and output shaft speeds or slip.

f) Equipment to continuously indicate and record the fluid temperature in the external scoop circuit and to provide excess temperature tripping facilities.

The test rig is constructed so that the induction motor drives the coupling through the torque transducer. The coupling output shaft is connected to the power absorber.

6.7.3.4 Procedure

The scoop control coupling is thoroughly cleaned, flushed with the test fluid and filled in accordance with the instructions in the manufacturer's handbook capacity approximately 16 l.

The induction motor is started and the scoop control lever moved gradually to full insertion position. The resistive load of the power absorber is set to the maximum capability of the rig, consistent with not exceeding the full load rating of the coupling, so that the fluid temperature can be increased rapidly close to the test temperature of 121°C. The power absorber is then adjusted to produce a slip in the coupling sufficient to maintain the temperature of the fluid in the scoop circuit at 121 ± 2°C.

The test rig is operated continuously for 100 h and 0.25 l samples of fluid are taken from the scoop circuit every 25 h without stopping the coupling. As samples are taken the fluid is immediately replaced with an equal volume of new fluid to keep the total constant. The rig is then stopped for two days and a second 100 h duration test carried out under the same conditions. After a further pause of two days the procedure is repeated for a third 100 h period, so that at the termination of the test the fluid has been subjected to 300 h of operation in the coupling. The viscosity and base number or acidity of each fluid sample is determined.

In addition 0.5 l samples are taken from the new fluid and from the fluid in the coupling after completion of each 100 h phase ; these samples are checked for fire resistance using the wick test detailed in section 3.3.2. of this report.

6.7.3.5 Results

The following data shall be recorded :

a) Fluid type, precise designation and batch number.

b) Total test duration.

c) The actual intervals at which fluid samples were taken.

d) For the new fluid sample and each sample taken during the test :

   i) Kinematic viscosity in cSt at 40°C and 100°C by one of the methods given in section 5.1.

   ii) Fluid base number or acidity measured in mg of KOH/g in accordance with section 6.2 of this report.

e) For the wick tests for fire-resistance on the new fluid sample and on the samples taken at the end of each 100 h phase, in accordance with section 3.3.2.

   i) All six results of the five exposure times.

   ii) The average of each set of six results.

   iii) The largest of the averages in (ii) taken as the Mean Persistence of Flame.

6.7.3.6 Acceptance criteria

This test is carried out for information purposes only and no acceptance criteria are laid down. However, as a guide to the interpretation of test results, it would be expected that the fluid would comply with the following :

a) Viscosity

The viscosity of the fluid in cSt at 40°C should not vary by more than 15% of the initial value during the 300 h period.

b) Base number or acidity

The total base number or acidity value of the fluid measured in mg of KOH/g should not show a reduction in base number or an increase in acidity of more than 1 mg of KOH/g during the 300 h period.

c) Fire-resistance

The fluid shall remain fire-resistant on completion of the 300 h test period. The mean persistence of flame should be less than 60 s (in accordance with the requirements of section 3.3.2. of this report).

6.8 METHODS OF TEST FOR THE COMPATIBILITY OF COATINGS FOR POWER PACK TANKS WITH FLUIDS OF TYPES HFA, HFB AND HFC

Note : The tests described in this section are to enable an assessment to be made of the corrosive or destructive effects of fluids on the coated metal parts of power pack tanks and other hydraulic equipment (see also sections 5.10 and 5.9 for the effect of fluids on seals and packings and bare metals respectively).

The two tests described cover the use of fluids of types HFA, HFB and HFC. A test covering the use of type HFD fluid has not been included as the fluids of this type currently in use appear to be non corrosive to unprotected steel in the immersed state or in the vapour phase. Should a test for type HFD fluids be required at some later date, however, the test could be based on that described in section 5.9.2. but with the O-ring material chosen to be compatible with the fluid and the test temperature selected to reflect the conditions of use for that fluid.

6.8.1. METHOD OF TEST FOR COMPATIBILITY OF COATINGS OF POWER PACK TANKS WITH TYPE HFA FLUIDS AND EMULSIFYING OILS

Note : This method of test is based on the NCB Supplementary Test Methods for Fire-resistant Fluids.

6.8.1.1. Scope

The test provides a guide to the selection of coating systems for use on the internal surfaces of power pack tanks. Such tanks may contain emulsifying oil or made-up dilute emulsions.

6.8.1.2. Principle

The coating needs to be compatible with, and needs to protect the underlying steel from attack by, the emulsifying oil, its vapour, the emulsion at normal and low levels of oil concentration, the emulsion vapour (essentially water vapour) and the condensed emulsion vapour (essentially water).

The method provides a coating assessment in relation to these requirements by two separate tests. In the first the effects of the oil and oil vapour on the coated steel are examined. In the second the effects of distilled water and its vapour on the coated steel are examined. In each test coated specimens are maintained in appropriate environments for a period of time at a temperature corresponding to the maximum service temperature.

6.8.1.3. Apparatus

1) Mild steel tubes, 50 ± 0.01 mm in length, 20 mm outside diameter and 15 mm inside diameter.

2) Clamping device.

3) Boiling tubes.

4) Tube closures.

5) Thermostatically controlled oven, suitable for maintaining specimens at a temperature of 70 ± 1°C, and fitted with a glass door.

6) Mercury-in-glass total immersion thermometer, with a range of 45 to 105°C and an accuracy of 0.2°C.

7) Cork bung to fit boiling tube and with a central hole to accomodate the thermometer.

8) Non-destructive coating thickness gauge capable of measuring inside a circular area of 15 mm diameter.

9) Paint scratch tester.

6.8.1.4. Materials

1) Mild steel test panels coated with the coating system under test ; two of the panels of size 100 mm x 100 mm and two of size 100 mm x 25 mm.

2) Cold curing silicone rubber.

3) Distilled water.

4) Petroleum spirit or other solvent or solvents which are suitable for the emulsifying oils and which are compatible with the coating material.

5) 5 ml of each emulsifying oil.

6.8.1.5. Preparation

Inspect the test panels and reject any that are damaged, contaminated or otherwise not in an as-coated condition.

Measure the coating thickness at representative positions on the four test panels by means of the thickness gauge.

For each of the emulsifying oils to be included in the test, locate a mild steel tube on one of the larger test panels so as to form a number of cylindrical vessels with the test panel as base. Clamp the tubes to the test panel and apply cold curing silicone rubber around the base of each tube to form a joint. When the sealant has cured, remove the clamping device.

Mount the thermometer and cork bung in one of the boiling tubes into which distilled water has been introduced. The thermometer bulb should not touch the bottom of the tube and the level of the water should be well above the 70°C scale marking on the thermometer stem.

6.8.1.6. Procedure

Introduce 5 ml of each emulsifying oil in one of the tubes on the larger test panel. Mark the panel to identify the oil in each tube. Cover the tubes with the second test panel. Mark the panel to identify its position relative to the lower panel.

Place each of the two smaller test panels in a boiling tube in which distilled water has been introduced. The quantity should be such that approximately half the panel is immersed. Close each tube.

Place the oil test assembly, the two boiling tubes containing test panels and the boiling tube containing the thermometer close together in the oven. Position the items so that the temperature can be read from the thermometer. Maintain the specimens at a temperature of 70 ± 1°C for a period of 7 days.

At the end of the test period remove the oil test assembly and the boiling tubes from the oven. Remove the smaller test panels from the boiling tubes. Remove the top test panel from the oil test assembly and pour the oil from the tubes. Remove residual oil from the tubes by rinsing with petroleum spirit or other appropriate solvent and pull the tubes from the lower test panel.

Note : The absence of any effects of the solvent on the coating material must be checked by preliminary test if there is any doubt about the compatibility.

Visually examine each circular test zone on the lower test panel for the effects of each of the oils. Visually examine each circular test zone on the upper oil test panel for the effects of the vapour from each of the oils. Visually examine the two smaller test panels for the effects of the distilled water and its vapour and visually examine the water in each of the boiling tubes.

If the coating under test is metallic, measure the thickness in each test zone on each panel by means of the thickness gauge. The presence of corrosion product may prevent these measurements being made.

If the coating under test is organic, assess the loss of adhesion as nil, partial or complete by means of the paint scratch tester. The regions on the larger test panels distant from the test zones shall be taken as representative of the initial adhesion.

6.8.1.7. Results

The following should be reported :

a) The coating system identification.

b) The mean initial coating thickness (µm).

c) The visual effects of each oil, its vapour, distilled water and its vapour, on the coating and, if evident, on the underlying steel.

d) The visual changes in the distilled water used in the tests.

e) For metallic coatings, the mean loss in thickness produced by each oil, its vapour, distilled water and its vapour : where appropriate, total loss should be stated and any effect preventing satisfactory measurement should be described.

f) For organic coatings, the loss in adhesion produced by each oil, its vapour, distilled water and its vapour, expressed as nil, partial or complete.

6.8.1.8. Acceptance criteria

Any evidence of rusting of the underlying steel in the distilled water test indicates unsuitability of the coating. Acceptable metallic coatings may tarnish but there should be no obvious corrosion product and no significant loss of coating in any of the tests. Acceptable organic coatings should suffer no peeling or loss of adhesion in any of the tests.

6.8.2 METHOD OF TEST FOR COMPATIBILITY OF COATINGS OF POWER PACK TANKS WITH TYPES HFB AND HFC FLUIDS

Note : This method of test is based on the NCB Supplementary Test Methods for Fire-resistant fluids.

6.8.2.1. Scope

The test provides a guide to the suitability of coating systems for use on the internal surfaces of power pack tanks containing fluids of types HFB or HFC.

6.8.2.2. Principle

The coating needs to be compatible with, and needs to protect the underlying steel from attack by, the fluid, the fluid vapour (essentially water vapour) and the condensed fluid vapour (essentially water).

The method provides an assessment of a coating in relation to these requirements by exposing different regions of a coated steel sample to a range of fluids and their vapours and to distilled water and its vapour.

At the test temperature (the maximum temperature expected in service) the period of exposure is sufficient to indicate incompatibility. The test does not evaluate the service life of a compatible coating.

6.8.2.3. Apparatus

1. Heat resistant glass plate 500 mm (or plates with lengths totalling 500 mm) x 100 mm x 6 mm, with a row of thirteen holes each of 7 mm diameter aligned parallel to the 500 mm dimension (figure 1). The centre of each hole is 30 mm from the 500 mm edge and 38 mm from the next hole. The two end holes are each about 20 mm from the 100 mm edge.

2. Clamping frame (figure 2).

3. Nitrile rubber O-rings of hardness 65° BS or less, thirteen in number, each of nominally 64 mm outside diameter and 3 mm cross-section.

4. Cork bungs, thirteen in number, to fit the holes in the glass plate.

5. Pipette or syringe.

6. Flexible syringe.

   Note : This may be constructed using polythene tubing, semi-rigid grade (softening point approximately 115°C).

7. Thermostatically controlled oven suitable for maintaining specimens at 70 ± 1°C.

8. Non-destructive coating thickness gauge capable of measuring inside a circular area of 15 mm diameter.

9. Paint scratch tester.

6.8.2.4 Materials

1) Mild steel test panel, 500 mm x 100 mm x 2.0 to 2.7 mm, coated with the

coating system under test.

Notes

a) This size of panel allows the simultaneous testing of the coating with up to thirteen fluids.

b) Although one side only of the test panel need be coated it is advisable to prepare both sides to minimise distortion arising from, for example, grit-blasting on one side only and consequent difficulties in assembly of the apparatus.

c) It is the coating together with its associated method of surface preparation and application that is tested by the method. The coating manufacturer will define the proper method of surface preparation and application and will make the information available at the outset. A declaration of the method of preparation of the test panel will be required before the panel is tested. A decision not to proceed with the test may be taken if the panel has been prepared in such a way that practical application to power pack tanks would not be possible.

2) Distilled water.

3) 20 ml of each fluid.

4) Paper tissues.

6.8.2.5 Preparation

Inspect the test panel and reject it if it is damaged, contamined or otherwise not in an as-coated condition.

Measure the coating thickness at representative positions on the panel by means of the thickness gauge.

Assemble the panel, the glass plate, the O-rings and the clamping frame so that each O-ring forms a compartment between the panel and the glass plate. Each O-ring will be deformed to produce an elongated compartment which should be positioned to correspond with one of the holes, used for filling the compartment, in the glass plate. Figure 3 shows the apparatus assembled. Tighten the clamping frame sufficiently to produce effective seals between the O-rings and the panel and plate, but note that overtightening may fracture the glass plate.

6.8.2.6 Procedure

With the apparatus mounted so that the glass plate is vertical and the filling holes are towards the upper ends of the compartments, introduce a sample of one of the fluids into each of the compartments, using the pipette or syringe. The quantity of each fluid should be sufficient to bring the level to the lower edge of the filling hole. In a similar manner introduce distilled water into one of the compartments. Mark the frame to identify the fluid in each compartment. Lightly position a cork bung in each of the filling holes.

Place the assembly in the oven and after bringing the temperature to 70 ± 1°C tighten the cork bungs in the filling holes. Maintain the temperature at 70 ± 1°C for 7 days.

At the end of the test period remove the assembly from the oven. Visually examine the fluids and the distilled water in the compartments. Empty the compartments using the flexible syringe and dismantle the clamping frame, glass plate, O-rings and test panel. Gently mop the test panel dry using paper tissues.

Visually examine the test panel for the effects of each fluid and each vapour. Any effects of a fluid at different levels within the immersed zone should be examined in relation to any previously observed separation or layering in the fluid.

If the coating under test is metallic, measure the thickness in each immersed test zone and vapour test zone on the panel by means of the thickness gauge. The presence of corrosion product may prevent these measurements being made.

If the coating under test is organic, measure the thickness in each test zone that shows no blistering or peeling of the coating. Assess the loss of adhesion in each test zone as nil, partial or complete, on the basis of any blistering or peeling or, in the absence of such effects, by means of a paint scratch tester. The regions on the test panel away from the test zones shall be taken as representative of the initial adhesion.

6.8.2.7 Results

The following should be reported :

a) The coating system identification, the coating manufacturers' recommended method of surface preparation and coating application and any departure from these recommendations in the preparation of the test panel.

b) The mean initial coating thickness.

c) The visible effects of each fluid, its vapour, distilled water and its vapour on the coating and, if evident, on the underlying steel.

d) The visible changes in each fluid and the distilled water.

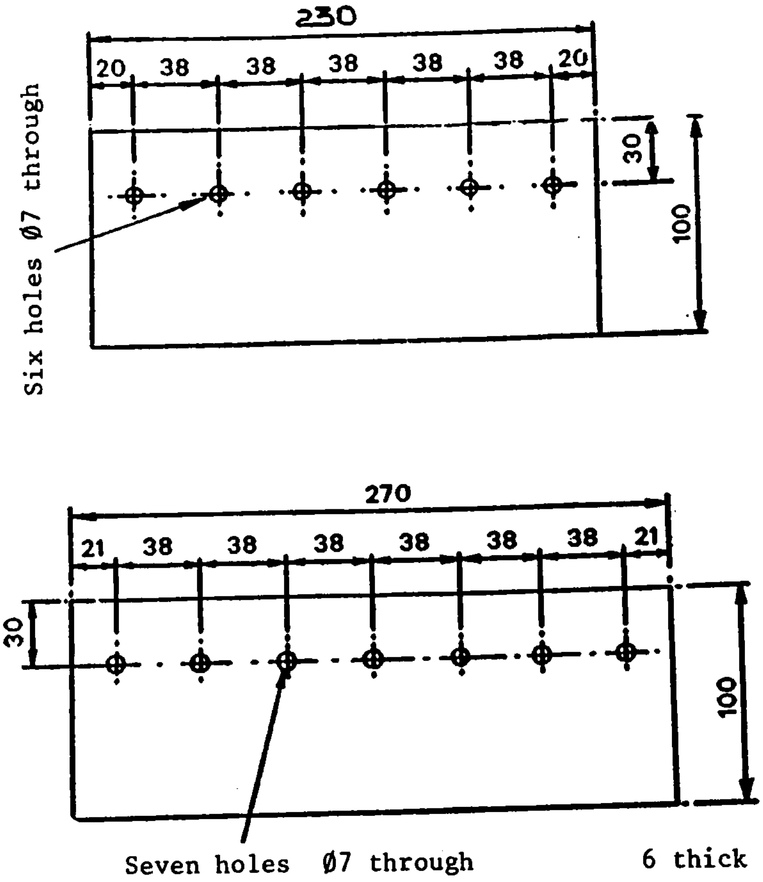
e) For metallic coatings, the mean loss in thickness produced by each fluid, its vapour, distilled water and its vapour ; where appropriate total loss should be stated and any effect preventing satisfactory measurements should be described.

f) For organic coatings, the mean increase in thickness produced by each fluid, its vapour, distilled water and its vapour, except for those fluids or vapours causing blistering or peeling.

g) For organic coatings, the loss in adhesion produced by each fluid, its vapour, distilled water and its vapour, expressed as nil, partial or complete.

6.8.2.8 Acceptance criteria

Any evidence of rusting of the underlying steel indicates unsuitability of the coating. Acceptable metallic coatings may tarnish but there should be no obvious corrosion product and no significant loss of coating. Acceptable organic coatings should suffer no blistering, peeling, loss of adhesion or increase in thickness greater than about 20%.



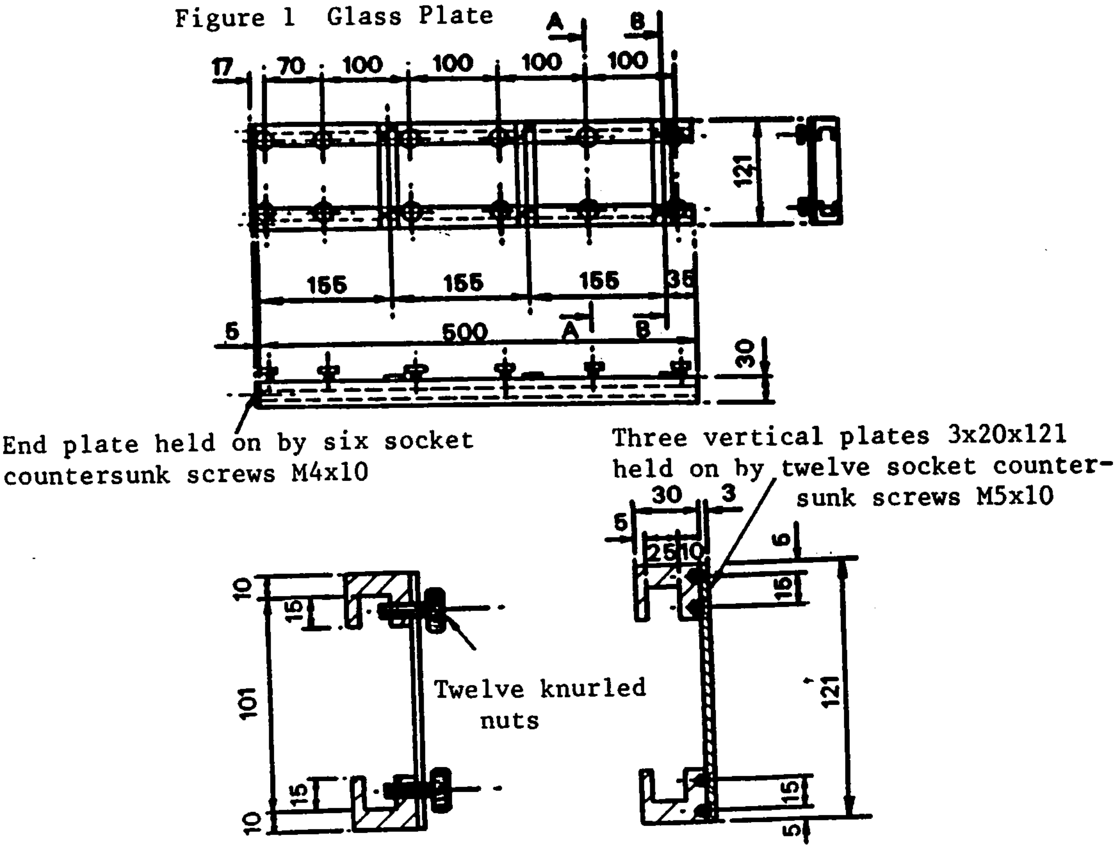
Six holes Ø7 through

Seven holes Ø7 through

6 thick

All dimensions in millimetres

Figure 1 Glass Plate



End plate held on by six socket countersunk screws M4x10

Three vertical plates 3x20x121 held on by twelve socket countersunk screws M5x10

Twelve knurled nuts

Figure 2 Clamping frame

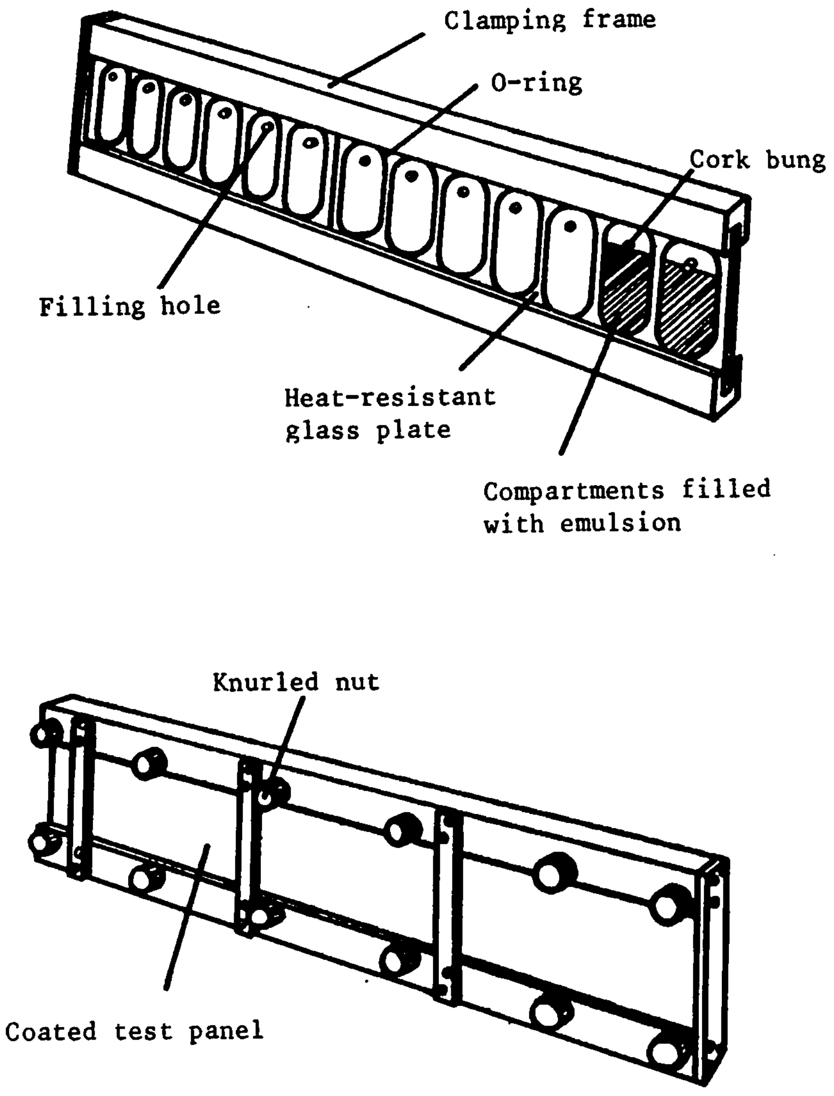


Figure 3 Assembled apparatus

6.9 METHODS OF TEST FOR THE LUBRICATING PROPERTIES OF FLUIDS

No single test is currently available which, by itself, is capable of covering all aspects of lubrication and indicating the mechanical suitability of a fluid for all applications. A number of tests are available, however, which relate to the different types of contact, relative movement and wear and the user and/or manufacturer is advised to select those tests which most closely reproduce the working conditions of the principal applications of the fluids in service.

The best known of these mechanical tests are listed in the table. Three of these tests are described in sections 6.9.1 to 6.9.3 as example of the types of test which are currently being used for the assessment of lubricating properties. They have not been included to specially advocate their use.

Type of contact	Type of Relative Movement	Type of Wear	Type of Test	Standard Test Methods (2)
Point or line (Hertzian)	Rolling	Fatigue	Four-ball machine with rolling balls	NCB Supplementary Method No 8 (1) NCB Supplementary Method No 11 IP 300/79
			Unisteel machine (balls)	NCB Supplementary Method No 9 NCB Supplementary Method No 11 IP 305/79
			CERCHAR ball and needle roller machine	
			RW-TUV/SKF needle roller machine	
			INSA rotating disc film thickness apparatus MRDE/SKEFCO needle roller machine	
	Sliding	Adhesive wear. Welding	Four-ball machine	NCB Supplementary Method No 6 IP 239/79 DIN 51350 NF E48-617 ASTM D2596
			INSA rotating disc film thickness apparatus	
			Tribolub machine	
	Sliding and Rolling	Adhesive wear. Scoring Scuffing Seizure	FZG Spur gear machine	DIN 51 354 IP 334/77 CEC L-07-A-71
Surface (conforming)	Sliding	Adhesive wear.	Tribolub machine	
			Vickers vane pump	CETOP RP67H (1) IP 281/77 BS 5096:1974 DIN 51389 NF E48-617 ASTM D2882

<u>Notes</u> : (1) These tests are described in sections 6.9.1 to 6.9.3 as examples.

(2) Listing of several methods within the same category is not intended to imply that the methods are identical or technically equivalent.

6.9.1 METHOD OF TEST FOR LUBRICATING PROPERTIES OF FLUIDS BY MEASUREMENT OF THEIR EFFECT ON ROLLING FATIGUE RESISTANCE OF BEARING STEEL USING THE ROLLING 4-BALL MACHINE

6.9.1.1 Scope

The test provides a simple and relatively quick method for assessing the tendency of fire resistant fluids to cause fatigue failures in rolling contacts. The test method is essentially the same as Test Method IP 300/79 and the units adopted are as in the IP method.

6.9.1.2 Principle

The conventional Four-ball Machine, conforming to IP 239/79, is modified so that the three lower balls are allowed to rotate in a special race* when brought into loaded contact with the single upper ball. The fluid under test is used to lubricate the balls. The upper ball is regarded as the most important test piece and it is the pitting of this ball which is considered to be the main criterion of the test. Twenty-four runs are made per test and the time to pitting of the top ball recorded for each run.

6.9.1.3 Apparatus

1) Four-ball Extreme-pressure Lubricant Testing Machine modified as below.

2) Ball-cup Housing ; this replaces the standard ball-cup assembly IP 239/79 and contains a race* in which the bottom three balls are free to rotate. For the measurement of temperature of the test oil a sensing thermocouple may be situated in the ball housing so as to come into contact with the test oil. A fluid drain hole is provided through the spacing block below the cup. A drawing of the modified cup assembly of the Four-ball Machine is shown in figure 1.

3) Fitting Detector : a device which detects the onset of pitting and subsequently shuts down the machine drive motor and test time recorder. The primary sensing element can be a commercially available accelerometer fitted to a pot magnet. The associated equipment consists of a suitable amplifier and control relays. The sensitivity of the equipment may be either adjustable or permanently fixed to cause shut-down at any desired degree of pitting. If an accelerometer is used then a steel disc is incorporated on the handle of the ball housing for mounting the magnet.

4) Time Lapse Recorder : electrically operated.

5) Fluid Reservoir : fitted with drip feed.

6.9.1.4 Materials

1) Test balls : En31 Acid open-hearth steel 0.5 in (12.7 mm) diameter SKF Grade A. These balls conform to AFBMA Grade 10 dimensions.

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* The special race is obtainable from Creep and Tribology Division, Department of Trade and Industry, National Engineering Laboratory, East Kilbride, Glasgow.

2) Solvents : acetone conforming to BS 509, petroleum ether 60/80 conforming to IP Specification, or white spirit conforming to BS 245. An additional solvent may be required which is appropriate to the type of fluid under test.

6.9.1.5 Preparation

Before starting a test, run the motor unloaded for a minimum of 15 minutes. Clean the ball housing, chuck, splash ring, and four new steel balls in one, or a combination, of the solvents as found necessary and dry with a clean soft lint-free cloth or clean dry air before each run (Note 1).

Note 1 : The object of this cleaning procedure is to remove from the metal components all traces of oil, grease or protective compounds. The choice of solvent will depend on the type of material present but as a rough guide acetone should be used for aqueous-based or glycol-containing fluids, petroleum ether for mineral oils and white spirit for greases. Where difficulty is experienced using a single solvent a combination of two or more may be found effective.

6.9.1.6 Procedure

Fit one ball into the chuck and ensure that it cannot be rotated by hand within the chuck. Reject chucks in which the ball is loose. Fit the chuck into the taper in the end of the motor spindle.

Suspend the weight hanger, which weighs 1 kg, at a distance of 20 units from the spindle centre.

Place three cleaned and dried balls in the ball housing and mount the ball housing centrally under the motor spindle in contact with the upper ball. Place the mounting pad between the thrust bearing and the ball housing so that when the load is applied, the ball housing seats squarely on the mounting pad and is free to rotate with it.

Fill the fluid reservoir with the test fluid, locate the feed pipe above the ball housing and allow fluid to pass into the housing and out of the drain. Adjust the feed rate to give six to eight drops per minute.

Release the load lever and apply the 20 kg load very gently to the balls, taking care to avoid shock loading as this may deform the balls permanently. Check that the three lower balls centre themselves against the upper ball and spin the motor spindle by hand to ensure correct assembly, when a click will be heard. Place two 2-kg discs on the weight hanger, thereby giving a total load of 100 kg.

Assemble the vibration cut-out device, e.g. if this is an accelerometer place its mounting magnet on the handle of the ball housing. Reset the time recorder to 0.5 minute before the zero position and press the amplifier reset button. If the sensitivity of the pit detector is adjustable, turn the sensitivity down to zero.

During the first 0.5 min from start up, apply a further 500-kg load (i.e. five 5-kg discs) in order to bring the total applied load up to 600 kg. The discs must be applied very gently on the weight hanger.

When fully loaded, carefully increase the sensitivity of the pit detector, if adjustable, to a reading on the amplifier dial which will cause the machine to be shut down at the desired degree of pitting. After approximately 5 min of steady running the sensitivity should be trimmed.

The machine may now be left running unattended, until the top ball 'pits'. On automatic shut-down of the machine the time recorder will indicate the total running time.

Record the time to shut down to the nearest minute, and remove the load.

Remove the ball housing and mounting pad from the machine, and the upper ball chuck from the motor spindle.

Wash the ball in the solvent and inspect it for pitting. If on inspection there is no 'pit' on the top ball this run will be invalid. One of the lower three balls will probably have a 'pit' on its surface, therefore these balls should be removed from the ball housing, washed in the solvent and inspected for pitting (Note 1). In the event of a machine shut-down occurring without a 'pit' on any of the balls, this should be reported.

Store the four balls used in the run in a suitable container.

Clean the equipment as described in section 6.9.1.5 'Preparation'.

Repeat the above sequence of operations, using four new balls and a fresh sample of test fluid for each run until 24 runs have been completed, each terminating in pitting of the top ball.

6.9.1.7 Calculations

List the results of the 24 tests in order of increasing time to failure. Give each test result a value of 'estimated cumulative percentage failed' commencing with 4% for the lowest time, increasing in increments of 4% up to a final value of 95% for the highest time. A less simple derivation of the estimated cumulative percentage failed, but one which is more satisfactory on theoretical grounds has been given by Johnson* and may be employed alternatively.

Plot the estimated cumulative percentage failed against the failure time on Weibull co-ordinates and fit a straight line to the points. From the line read off the 10% life, L_{10} and the 50% life L_{50}. Convert the values of L_{10} and L_{50} to millions of revolutions by multiplying by the machine shaft speed in millions of revolutions per hour, i.e. 0.087. Calculate the Weibull slope, a, from

$$a = 0.8187 \;/\; \left\{\log_{10}(L_{50}/L_{10})\right\}$$

Calculate the 90% confidence limits on L_{10} and L_{50} by the method given by Johnson*.

The straight line may be fitted to the points by eye. Alternatively the points may be fitted to a Weibull distribution by a mathematical procedure and L_{10}, L_{50} and a together with the appropriate confidence limits, derived directly.

6.9.1.8 Report

The following should be reported :

a) Test fluid identification

b) Test load (600 kg)

c) Number of tests (24)

d) The life parameters characteristics of the fluid performance, L_{10} and L_{50} (millions of revolutions), together with their 90% confidence limits.

e) Weibull slope, a.

6.9.1.9 Precision

The precision of this method has not yet been established.

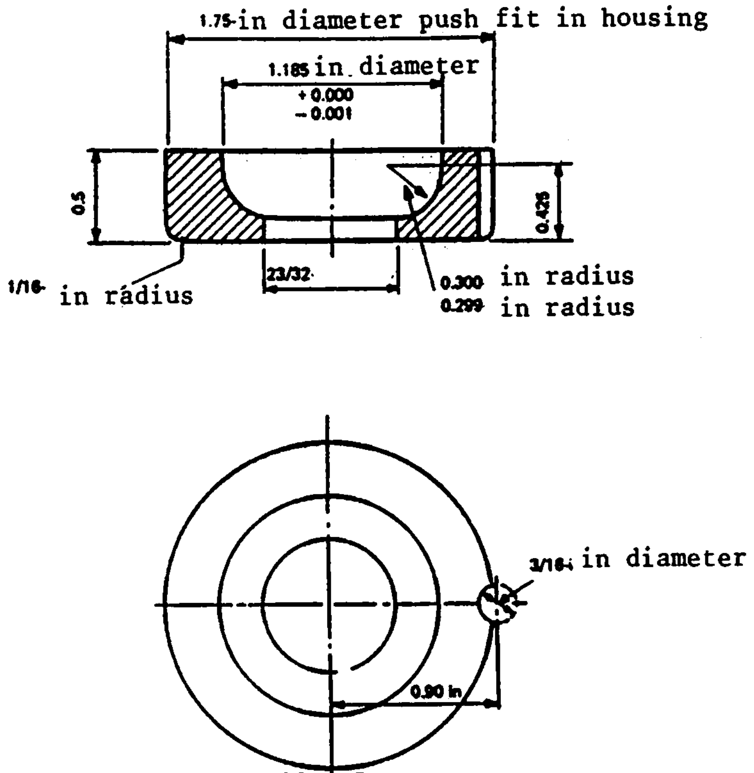
6.9.1.10 Interpretation of results

The value of L_{10} represents the life at which 10% of a large number of test pieces, lubricated with the test fluid, would be expected to have failed. The value of L_{50} relates in a corresponding manner to failure of 50% of test pieces. Fluids may be compared on the basis of L_{10} or L_{50} values. The advantage of using L_{10} is that the definition is similar to the definition of nominal bearing life as used by bearing manufacturers for specifying bearing performance with mineral oil lubrication. On the other hand the confidence band on L_{10} is wider than that on L_{50}.

The value of a is determined by the ratio of L_{50} to L_{10}. It is inversely related to the observed spread of test piece lives and, for a given number of tests, it determines the confidence limits on L_{10} and L_{50}.

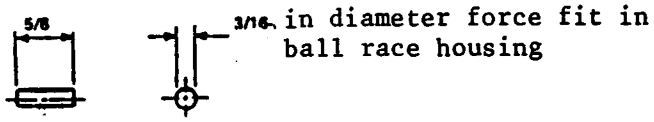
Though fluid comparisons can be made on the basis of the results it is recommended that the results be used as part of a more comprehensive assessment detailed in NCB Supplementary Test Methods for Fire-resistant Fluids.

* Johnson, L.G. The Statistical Treatment of Fatigue Experiments, Elsevier 1964.



Ball Race

One required. Tool steel (X9), harden and tamper



Dowel

One required. Silver steel

Figure 1 Four-ball machine, modified cup assembly

6.9.2 METHOD OF TEST FOR LUBRICATING PROPERTIES OF FLUIDS IN SLIDING CONTACT CONDITIONS

6.9.2.1 Scope

The test provides measurements of the extreme-pressure and anti-wear properties of fire resistant fluids by means of the Four-ball Machine. The test conditions are not intended to simulate particular service conditions but to provide information over a range of standard conditions for purposes of quality control and fluid comparisons. The test method corresponds with part of Test Method IP 239/79 and the units adopted are as used in the IP Method. This method is similar to but not identical to the method detailed in DIN 51350.

6.9.2.2 Principle

A single ball is rotated in loaded contact with three fixed balls, the fluid under test being used to lubricate the balls.

6.9.2.3 Apparatus

1) Four-ball Extreme-pressure Lubricant Testing Machine*: consisting essentially of a device by means of which a bearing ball may be rotated in contact with three fixed bearings balls that are immersed in the sample. Different loads are applied to the balls by means of weights on a load lever. No provision is made for the control of temperature. The upper rotating ball is held in a special chuck at the lower end of the vertical spindle of a constant-speed electric motor, operating at 147 to 157 rad/s (1450 to 1500 rev/min). The lower fixed balls are held in position against each other in a steel cup by means of a clamping ring and locking nut. The cup assembly is supported, above the load lever, by a disk which rests on a thrust bearing, thus allowing horizontal displacement and automatic alignment of the three lower balls against the upper ball. The frictional torque exerted on the three lower balls can be measured by means of a calibrated arm attached to the cup assembly, which is connected to the spring of a friction recording device.

   Note 1 : The UK-manufactured EP machines normally operate at 147 to 157 rad/s (1450 to 1500 rev/min). Machines made in other countries may operate at other speeds.

   It is important to distinguish between the Four-Ball Extreme-pressure Lubricant Testing Machine and the Four-Ball Wear Tester. The Four-ball Wear Tester is limited to loads up to 50 kg. The Four-ball Extreme-pressure Lubricant Testing Machine is designed for testing under more severe conditions up to 800 kg.

2) Loading weights : supplied for the applications of loads from 6 to 800 kg.

3) Microscope : measuring or projecting, to read to 0.01 mm.

4) Stopwatch : reading to 0.2 s.

5) Test balls : steel, 0.5-in diameter SKF grade 1, normal lime-polished balls.

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* The sole manufacturer in the UK of the machine is Stanhope-Seta Ltd, Park Close, Englefield Green, Egham, Surrey.

When ordering state 'for use with Four-ball Machine'.

6) Erecting plate : bolted firmly to the bench to facilitate assembly or removal of lower balls in ball cup ; it consists of a plate with three pins on which the cup can be located without turning when the locking nut is tightened or loosened.

6.9.2.4 Materials

Solvents : acetone conforming to BS 509, petroleum spirit 60/80 conforming to IP Specification, or white spirit conforming to BS 245. An additional solvent may be required, appropriate to the type of fluid under test.

6.9.2.5 Preparation

Before starting a series of tests, run the machine unloaded for a minimum of 15 min. Clean all appropriate parts of the machine with the solvent and dry with a clean soft lint-free cloth or clean dry air. Clean four new steel balls for each run in the same way.

6.9.2.6 Procedure

Place the ball cup on the erecting plate, put three balls into the cup, hold them in position with the clamping ring, and secure the assembly by tightening the locknut. Pour in sufficient sample of fluid (approx. 8 ml) to cover the balls to a depth of 3 mm.

Fit a clean ball into the upper ball chuck and check that it cannot be rotated by hand within the chuck. Reject chucks in which the ball is loose. Fit the chuck into the taper in the end of the motor spindle.

Mount the ball-cup assembly centrally under the spindle in contact with the fourth ball. Place the mounting disk between the thrust bearing and the cup so that when the cup is lowered into position it seats squarely on the disk and is free to rotate with it.

Suspend the weight-pan, which weighs 1 kg., from the appropriate notch on the load lever and place the required weights on it to give the desired load.

Release the load lever and apply the load slowly to the balls, taking care to avoid shock loading as this may deform the balls permanently. Check that the three lower balls centre themselves against the upper ball.

Start the main motor and the stop watch.

Allow the machine to run for the appropriate length of time, stop the motor and remove the load from the balls by raising the lever arm and locking it in position.

Remove the cup from the machine. Pour out the fluid sample from the cup and rinse the assembly with solvent, remove the balls (Note 2), wash again in solvent if necessary, dry and place in a suitable container for safe keeping and subsequent measurement of scar diameters.

Note 2 : When the test requires measurement of scar diameters, this may be done either before or after removing the balls from the cup according to the type and optical axis of the microscope used. It is helpful to mark the surface of the balls with an electric etcher to indicate the position of the wear scars, particularly when these are very small, otherwise there may be difficulty in finding the scars again.

Clean the locknut, lock ring, etc., in preparation for the next test. Remove the upper ball chuck from the machine and knock the ball out of the chuck by means of the hardened-steel pin and a hammer. Clean the chuck thoroughly.

Repeat the above procedure, using four new balls and a fresh sample for each run, for all the determinations and different loads required to complete a particular test.

6.9.2.7 Tests

1) Mean Hertz Load (MHL)*

This procedure is described with reference to the data sheet shown in figure 1.

Carry out a series of runs, applying a starting load of 40 kg (marked 'base') and allowing the machine to run under load for 60 ± 2 s. Make the subsequent runs at the successively higher loads shown on the data sheet until the welding of the four balls occurs.

Care should be taken to switch-off immediately welding occurs to prevent excessive wear in the chuck since, at this stage, the chuck will spin round on the top ball.

Make two check runs at the welding load, if the welding does not occur on both of the check runs, make a run at the next higher load and check if welding occurs.

If the verified welding load occurs at or below 355 kg make enough additional runs at the successively lower loads below 40 kg to provide a total of 20 runs exclusive of the welding load. If welding occurs above 355 kg, do not make any additional runs below 40 kg.

After each run, measure the two wear scar diameters to the nearest 0.01 mm in the direction of rubbing and at right angles to it on each of the three lower balls and record them in columns 1 to 6 of the data sheet. A measuring or projecting microscope is suitable for this purpose.

At light loads it is advisable to mark the region of the scar before removing the balls from the cup for measurement. Alternatively, scar diameters may be measured with the balls in the cup; in which case care should be taken to ensure that the scars are viewed normal to the scar surface.

Several different types of wear scar may be found. At low loads before seizure has occurred the wear scar is normally circular with well defined edges and the measurement of two of its diameters presents no difficulty.

At light loads after seizure the wear scar is again approximately circular, but the edges are frequently ragged and sometimes may be obscured by metal, worn off during the run, adhering to the scar on the trailing edge. This obstruction may be easily removed with a penknife to reveal the true edge.

2) Wear Load Curve. Welding Load and Initial Seizure Load (ISL).

Carry out 60 ± 0.2 s runs at a series of loads in 10 kg steps up to 100 kg, then in 25 kg steps up to 300 kg and finally in 50 kg steps up to 800 kg. The highest load will be the welding load, which is determined by additio-

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*ASTM Method D2596 and US Federal Test Method Standard 791D, Method 6503.1, describe similar test procedures.

nal runs to the nearest 10 kg, one run being made 10 kg below the welding load.

Alternatively, the results from a Mean Hertz Load determination may be used to plot a wear load curve.

In either case, additional runs may be necessary to define more precisely the shape of the curve.

Measure the wear scar diameters in accordance with the procedure detailed for Mean Hertz Load.

3) Wear Test

Carry out a 1 h run at a load of 15 kg.

Measure the wear scar diameters in accordance with the procedure detailed for Mean Hertz Load.

A shorter test time may be used depending on the observation of any changes in the test fluid due, for example, to evaporation of water. A higher load may be used depending on the values of welding load and initial seizure load for the fluid.

6.9.2.8 Calculations

1) Mean Hertz Load

Where the spherical balls are in contact under load the Hertz diameter is the theoretical diameter of the circular area of contact, produced by elastic deformation, according to Hertz.

A plot of Hertz diameter against load is known as the Hertz line ; on a log basis this is a straight line. Two suitable reference points for constructing the line in relation to conditions in the Four-ball Machine are at loads of 40 kg and 316 kg, at which the diameters are 0.300 mm and 0.597 mm respectively.

Using the data sheet (figure 1) calculate the Mean Hertz Load by means of the following equation :

$$\mathrm{MHL} = \frac{A + B}{20}$$

where A is the sum of the corrected loads at 316 kg and below, and B is the average of all the corrected loads made above 316 kg.

The corrected load of the last column of the data sheet is obtained for each run by reducing the actual load in the proportion of the ratio of the Hertz diameter at that load to the measured wear scar diameter, thus :

$$\text{corrected load} = \text{actual load} \times \frac{\text{Hertz diameter}}{\text{measured diameter}}$$

Since the actual loads and the corresponding Hertz diameters are known their products Wd_H have been shown as factors (figure 1).

2) Wear-load Curve and Welding Load

Plot the log of the mean of the six wear scar diameter measurements at each load against the log of the load.

3) Initial Seizure Load

On the Wear Load Curve the Initial Seizure Load is indicated by a sharp increase in wear.

6.9.2.9 Report

The following should be reported :

a) The test fluid identification

b) The Mean Hertz Load to the nearest 1 kg as MHL (60 s)

c) The Wear Load Curve and the Hertz line

d) The Welding Load to the nearest 10 kg as WL (60 s)

e) The Initial Seizure Load to the nearest 5 kg as ISL (60 s)

f) The Mean Wear Scar Diameter from the wear test to the nearest 0.01 mm as MWSD (1 h at 15 kg).

6.9.2.10 Precision

The following criterion should be used for judging the acceptability of results (95% confidence).

Reproducibility - the results submitted by each of two laboratories should not be considered suspect unless they differ by more than the values given below :

Method	Reproducibility
Initial Seizure Load 60 s	0.52x
Mean Hertz Load 60 s	18.2 (tentative estimate)
Welding Load 60 s	60.1

where x is the mean of two results.

6.9.2.11 Interpretation of results

The precise contact conditions existing in the four-ball configuration are rarely reproduced in practical applications, therefore care should be taken in the interpretation of results. Tests have established typical values for the various types of fire-resistant fluids so that results can be used on a comparative basis. The Mean Hertz Load expresses in a single figure the overall wear-load diagram for the fluid. The Welding Load indicates the extreme-pressure characteristics of the fluid while the Initial Seizure Load provides a measure of the film strength. The mean wear scar after 1 h at 15 kg has been found to correlate with wear in pump tests better than have the results from the 60 s tests.

Applied load (kg)	Wear Scar Diameters (mm)						Average Diameter (mm)	Wd_H Factor	Corrected Load $\frac{Wd_H\ Factor}{Av.\ Diameter}$
	1	2	3	4	5	6			
6								0.954	
7								1.120	
8								1.400	
9								1.638	
10								1.880	
11								2.134	
13								2.665	
14								2.940	
16								3.520	
18								4.122	
20								4.470	
22								5.390	
25								6.375	
28								7.420	
32								8.864	
38								10.37	
Base-40								11.96	
45								14.00	
50								16.10	
56								18.70	
63								21.86	
71								25.70	
79								29.62	
89								34.71	
100								40.50	
112								47.15	
126								55.19	
141								54.01	
158								74.58	
178								87.40	
200								102.2	
224								118.8	
251								138.3	
282								161.6	
316								188.0	
								Total A	
355								219.4	
398								255.5	
447								298.1	
501								347.2	
562								404.6	
631								472.6	
708								550.8	
794								641.6	
								Average B	

$$\boxed{\text{Mean Hertz Load} = \frac{A + B}{20}}$$

Figure 1 Mean hertz load data sheet

6.9.3 METHOD OF TEST FOR ANTI-WEAR PROPERTIES USING THE VICKERS PUMP TEST RIG

6.9.3.1 Scope

This method is used to find the wear behaviour of flame-resistant hydraulic fluids in vane pumps.

6.9.3.2 Principle

The weight loss caused by wear in the rubbing parts of the pump is found under mechanical-dynamic conditions such as are met with in hydraulic pumping. This weight difference is related to the running time for evaluation purposes.

6.9.3.3 Apparatus

The following components are required :

a) Vickers vane pump, V 105 C-10 (V 104 C-10) driven by an electric motor 11 KW, 1460 ± 30 rev/min.

b) Pressure governor valve.

c) Fluid cooler with cold-water connection and regulator.

d) Reflux filter, 25 cycles (filter insert must be compatible test fluid) porc size : 10 µm

e) Flowmeter : range 0 to 45 l/min.

f) Fluid container with minimum capacity of 70 l made from material resistant to all test fluids (stainless steel, proposal shown in figure 2).

g) Tube from container to pump, dia 25 mm x 2 mm

h) Tube from pump to container, dia 19 mm x 2 mm

i) Manometer, thermometer and stop valve (ballcock).

j) Multi-colour compensation recorder
Multi-colour compensation recording of this kind makes it easier to read off the factors.

k) Timer.

l) Balance, load capacity at least 200 g, reading every 0.1 mg.

m) Torque wrench 0 to 20 Nm

n) Wear insert for pump.

Pump consists of rotor, ring, vanes, two bushes and pin.

Wear insert components must satisfy following conditions :

Hardness :
Ring : 60 to 63 Rockwell C
Rotor : Surface hardness at least 81 Rockwell A (Vickers 6850 N/mm)
Core hardness 30 to 42 Rockwell C
Vanes : 60 to 63 Rockwell C

Chemical analysis in % by weight

Element	Ring	Rotor	Vanes
C	0.95 - 1.10	0.18 - 0.23	0.78 - 0.88
Mn	0.25 - 0.40	0.70 - 0.90	0.20 - 0.40
P	0.025 max	0.035 max	0.030 max
S	0.025 max	0.040 max	0.030 max
Si	0.20 - 0.35	0.20 - 0.35	0.20 - 0.40
Ni	0.25 max	0.40 - 0.70	-
Cr	1.30 - 1.60	0.40 - 0.60	3.75 - 4.50
Mo	0.08 max	0.15 - 0.25	4.50 - 5.50
W	-	-	5.50 - 6.75
W	-	-	1.60 - 2.20
Cu	0.25 max	0.35 max	-

Dimensions:

The ring must be 0.018 to 0.036 mm wider than the rotor, and the rotor must be 0.0025 to 0.015 mm wider than the vanes.

o) Safety devices :

The following are recommended :

a) Motor switch.

b) Temperature monitor which cuts out the motor when fluid temperature drops more than 5°C below the ideal temperature.

c) Pressure governor valve (contact pressure gauge) which cuts out the motor when pressure downstream of pump deviates by more than 20 bar either wide of ideal value.

d) Flow monitor which cuts out motor when pump volume deviates more than 25% either way of ideal value.

e) Level monitor which cuts out the motor when the fluid volume in the container during the test decreases by more than 10%.

The various components which are all commercially available, are assembled as shown in figure 1.

All inside coatings must be completely removed before assembly, and the packings must be replaced by Viton grade packings. The filter insert must be tested to establish whether the substances of which these are made are resistant to the test fluids. Care should be taken during assembly to ensure that the tube between the outlet from the container and the pump intake is devoid of curves. The pump intake should lie 500 to 700 mm below the meniscus in the container. The stopcock should be of the ballcock type. Measuring stations are as in figure 1.

The timing of the experiment is carried out using a timer operated in steps less than 1 min. It is a good idea to install a pre-selector which cuts out the motor after a pre-set time.

6.9.3.4 Solvents

The solvents used to clean the rig and the wear components must be devoid of high-pressure properties which necessarily excludes carbon tetra-chloride. In the case of type HFD fluids, an easy-evaporating petroleum distillate should be used. Prior cleaning when testing aqueous hydraulic fluid should be with 1 : 2 propyleneglycol.

6.9.3.5 Preparation

a) Cleaning the wear components

It is essential that these components should be cleaned before weighing to ensure that the weight loss is accurately determined. Experiments have indicated that straightforward washing with solvent does not always provide reproducible cleanness. Ultrasonic cleaning has been found effective, preferably over a duration of 30 to 60 s.

b) Temperature control (viscosity)

As the operative viscosity is critical for the hydro-dynamic component, the viscosity of the pressure fluid upstream of the pump must be kept constant. Automatic viscosity control involves continuous monitoring of viscosity, Normally speaking, viscometers working on the rotation principle are the only ones suitable for this duty. In this particular principle, the shear gradient across the rotation body influences the measured viscosity value. Depending on the composition of the pressure fluid, especially in the case of fluid involving polymerisation products, this can have an appreciable effect. For this reason, a temperature regulating principle has been installed, to replace direct control of the viscosity. The temperature probe at the cooler outlet controls the coolant delivery rate.

c) Establishing the test temperature

In order to be able to compare the performances of fluids of different types and of different viscosities, the temperature of the fluid under test should be adjusted so that its viscosity is as shown in the following table :

Type of fluid	ISO viscosity grade	Viscosity of fluid at test temperature
HFD	-	13 cSt
HFA, HFB and HFC	10	9 cSt
	15	12 cSt
	22	17 cSt
	32	23 cSt
	46	33 cSt
	68	48 cSt

d) Establishing the operating pressure

Normally speaking, the operating pressure is twice the permitted operating pressure for this type of pump, i.e. 140 bar for type HFD fluids. For fluids of types HFA, HFB and HFC, the test is carried out at a pressure of 105 bar.

e) Duration of test

The pump tests should not last longer than 250 h.

f) Requirements for wear parts

The wear parts must be tested before assembly to check for tolerances. Each rotor must be ultrasonically tested before installation to check for possible cracks. For this purpose, a 12 MHZ subminiature probe is used to test all points on end and casing surfaces between the rotor slots in both radial and axial directions.

6.9.3.6 Procedure

Before the actual test run, all the parts of the rig coming into contact with the fluid must be carefully cleaned. When testing fire-resistant fluids containing water (types HFA, HFB and HFC) the cleaning fluid should be 1,2 Propyleneglycol. For anhydrous fluids, cleaning should be with benzine. The cleaning fluid is then drained and all the tube connections freed. A new element is installed in the filter and this is then flushed with the test fluid. The flushing fluid is subsequently discarded.

A filling of 60 l of fluid is required for the test.

A new wear insert (Vickers Order No. 429 126) is used for each test. The wear components are first cleaned roughly using a benzine soaked rag, and subsequently using a rag steeped in acetone. The final cleaning is carried out in an ultrasonic bath and, after being weighed, the wear components are left overnight in the test fluid.

In the case of water-containing fluid, the shaft and the ball bearings exposed to higher loads are replaced as often as possible.

For the pump test, the wear components are installed (check on the direction of rotation when installing the vanes) and the test stand is run for one hour at zero pressure and with the coolant off (contact time). The difference between the level of fluid in the container and the pump intake should be at least 500 mm and the fluid has to be replenished if need be. At the end of the contact time, the pressure is raised to 20 bar in 60 s. At this pressure, the pump is operated for 10 minutes and the pressure is subsequently raised in steps of 20 bar at 10 min intervals (running-in) within a period of 60 s. The coolant water is not switched in until the fluid temperature reaches the established test value. For fluids of types HFA, HFB and HFC, maximum operating pressure of bar is reached after 50 minutes, and maximum operating pressure of 140 bar is reached after 70 minutes in the case of type HFD fluids. Lid-mounted screws are used to regulate the flow volume to 28 ± 2 l/min. The torque wrench is used to adjust the lid screws by uniformed amounts so as to keep the lid planer/parallel.

Operating temperature must not fluctuate by more than ± 2°C. After 30 minutes running, the pressure is measured in the pump suction pipe and should not exceed a vacuum pressure of 0.05 bar.

The test is terminated after 250 h. The weight loss in the wear components is carried out after cleaning as described in section 6.9.3.5.

6.9.3.7 Results

Wear protection capacity is evaluated by the weight loss in the wear components (ring and vane) depending on the duration of the tests (including running in time).

6.9.3.8 Reproducibility

Differences arising when the test is repeated are caused not only by the fluid itself but also by the manufacturing tolerances of the wear components and the degree of exactness with which the test conditions are observed. Incorrect installation of the wear components or incomplete venting of the system can produce outliers in the results. Where weight losses are high, above 1,000 there will necessarily be large absolute differences between two individual results. At these high orders of weight loss, the fluid cannot only be assessed by the absolute value of this weight loss in the wear components. Consideration must also be given in these cases to the running period achieved until the appearance of running noise, pressure loss, reduction in pressure fluid or possible axle breakage.

ASSESSMENT OF ANTI-WEAR PROPERTIES

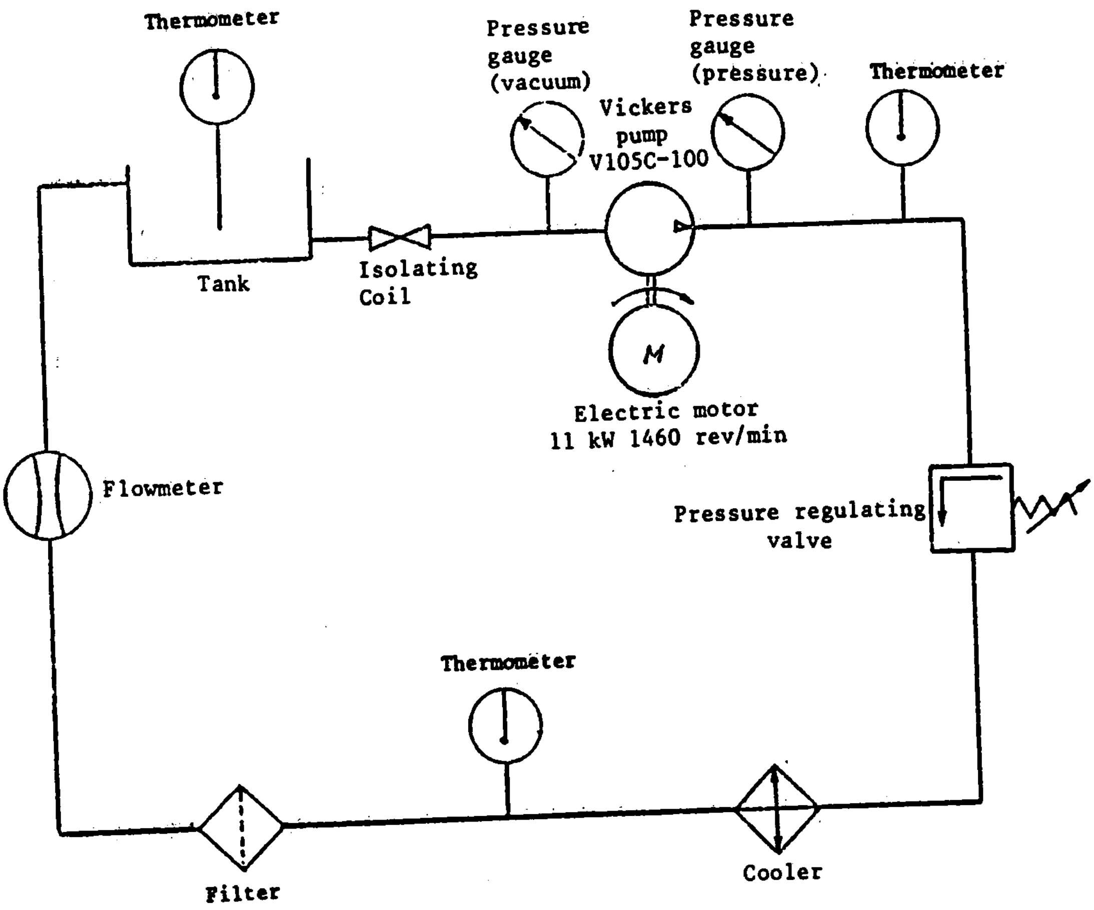

Figure 1 Diagram of test assembly

6.10 THE CLOSED FLASH POINT TEST

Emulsifying oils and fluid concentrates for type HFA and HFAS fluids, respectively, only become fire-resistant when diluted with a proper concentration of water (normally they are used with less than 5% of oil or fluid concentrate in more than 95% of water). Member States of the European Community have rules which prevent fluids having flash points below a value which varies according to the State from 55°C to 70°C from being taken underground in coal mines. It is, therefore, necessary to determine the closed flash point of such fluids. For this purpose, the methods specified in ISO 2719 and DIN 51758 are recommended. Relevant details are given below :

6.10.1 Scope

Primarily for emulsifying oils and fluid concentrates where its use is mandatory. The same method, however, can also be used to determine the flash point of type HFD fluids.

6.10.2 Principle

The test method uses the Pensky Martens closed cup apparatus as described in the above mentioned ISO standard. A sample of the fluid is heated in an enclosed cup and a small igniting flame is directed into the vapour space in the cup at regular intervals. The closed flash point is the lowest temperature at which application of the flame causes the vapour to ignite.

6.10.3 Apparatus

a) A Pensky Martens closed cup apparatus as described in ISO 2719.

b) Thermometers - partial immersion type conforming to the appropriate spesification in table 1 below :

- low range for samples giving a flash point between 12°C and 110°C
- high range for samples giving a flash point between 107°C and 370°C

Table 1. Thermometer specifications

	Low range	High range
Range	-7 to +110°C	+90 to +370°C
Immersion	57 mm	57 mm
Graduation	0.5°C	2°C
Longer lines at each	1°C	10°C
Numbered at each	5°C	20°C
Scale error not to exceed	0.5°C	1.5°C

	Low range	High range
Expansion chamber permitting heating to	160°C	370°C
Overall length	287 ± 5 mm	287 ± 5 mm
Stem diameter	6 to 7 mm	6 to 7 mm
Bulb length	9 to 13 mm	8 to 10 mm
Bulb diameter	stem	4.5 to 6 mm
Distance from bottom of bulb to line at :	0°C 85 to 98 mm	110°C 86 to 99 mm
Distance from bottom of bulb to line at :	100°C 221 to 237 mm	360°C 227 to 245 mm
Stem enlargement :		
diameter	7.5 to 8.5 mm	7.5 to 8.5 mm
length	2.5 to 5.0 mm	2.5 to 5.0 mm
distance to bottom	64 to 66 mm	64 to 66 mm

6.10.4 Preparation of Sample

Samples of very viscous materials may be warmed until they are reasonably fluid before they are tested. However, no sample should be heated more than is absolutely necessary. A sample shall never be heated above a temperature 17°C below its expected flash point.

Samples containing dissolved or free water may be dehydrated with calcium chloride or by filtering through a qualitative filter paper or a loose plug of dry absorbent cotton. Warming the sample is permitted, but it shall not be heated for prolonged periods or above a temperature 17°C below its expected flash point.

6.10.5 Procedure

Thoroughly clean and dry all parts of the cup and its accessories before starting the test, being sure to remove any solvent which has been used to clean the apparatus. Fill the cup with the sample to be tested to the level indicated by the filling mark. Place the lid on the cup and set the latter in the stove. Ensure that the locating or locking device is properly engaged. Insert the thermometer. Light the test flame and adjust it to a diameter of approximately 4 mm. Heat at such a rate that the temperature as indicated by the thermometer increases 5 to 6°C/min. Turn the stirrer at 90 to 120 rev/min (9.4 to 12.5 rad/s) stirring in a downward direction. (DIN 51758 specifies a stirring speed of 60 rev/min or 6.2 rad/s).

If the sample is expected to have a flash point of 104°C or below, apply the test flame when the temperature of the sample is not higher than 17°C below the flash point and thereafter at a temperature reading that is a multiple of 1°C. Apply the test flame by operating the mechanism on the cover which controls the shutter and test flame burner so that the flame is lowered into the vapour space of the cup in 0.5 s, left in its lowered position for 1 s, and quickly raised to its high position. Do not stir the sample while applying the test flame.

If the sample is expected to have a flash point above 104°C, apply the test flame in the manner just described at each temperature that is a multiple of 2°C, beginning at a temperature not higher than 17°C below the flash point.

Record as the flash point the temperature indicated on the thermometer at the time the test flame application causes a distinct flash in the interior of the cup. Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame at applications preceding the one that causes the actual flash.

6.10.6 Results

Correction for barometric pressure.

Observe and record the barometric pressure. Calculate the flash point corrected to standard barometric pressure of 1,013 bar by adding algebraically to the observed temperature the correction given in degrees Celsius by the formula :

$$\frac{1{,}013 - p}{0{,}033} \times 0.9$$

where p is the barometric pressure expressed in bars.

Round off the values thus obtained to the nearest whole number. The corrected flash point should be reported as the Pensky-Martens closed cup flash point.

6.10.7 Acceptance criteria

The value of the flash point, corrected in accordance with section 3.4.6. shall not be less than the minimum value specified by the Member State in which the fluid is to be used.

Where duplicate results are obtained by the same operator using the same apparatus, they shall be considered suspect if they differ by more than 2°C in the case of flash points of 104°C or less or 5.5°C in the case of flash points over 104°C.

Where test results are submitted by each of two laboratories, the results shall be considered suspect if they differ by more than 3.5°C in the case of flash points of 104°C or less or 8°C for flash points greater than 104°C.

APPENDIX I

LIST OF PROPERTIES CONSIDERED BY THE COMMITTEE OF EXPERTS ON FIRE-RESISTANT FLUIDS DURING THE PREPARATION OF THE SIXTH REPORT ON CRITERIA AND SPECIFICATIONS

FLUID PROPERTY CONSIDERED	TEST METHOD INCLUDED IN REPORT	TEST METHOD REJECTED	NOTES
Ageing HFC and HFD)	0		(1)
Ash content		0	
Colour		0	
Compatibility with coatings:			
- HFA fluids and emulsifying oils	0		(2)
- HFB and HFC fluids	0		(2)
Compatibility with sealing materials	0		(2) (3) (4)
Compressibility		0	
Contamination		0	
Corrosion prevention:			
- Fluids	0		(1) (3)
- Emulsifying oils	0		(1)
Deaeration capacity	0		(1)
Density	0		(2)
Fatigue - see Lubricating properties			
Filtrability	0		
Flammability - see resistance to flame			
Flash point of emulsifying oils	0		(1)
Foaming	0		(1)
Formulation		0	(5)
Health criteria	0		(1)
Identity control	0		(8)
Lubricating properties	0		(2) (6)
Miscibility		0	
Neutralisation number HFB and HFD)	0		(2)
Oil content of HFA fluids	0		(2)
Pour point	0		(2)
pH value:			
- of HFA and HFC fluids	0		(1)
- of water	0		(1)
Pumps - see Lubrication			
Refractive index		0	
Resistance to flame:			
- Spray ignition	0		(1) (3) (4)
- Flame propagation	0		(1) (3)
Stability of emulsions:			
- HFA fluids	0		(1)
- HFB fluids at ambient temperature	0		(1)
- HFB fluids at medium temperature	0		(1)
- HFB fluids at low temperature	0		(1)
Shear strength	0		(1)
Specific heat		0	
Storage life		0	(5)
Thermal expansion		0	
Thermal stability of HFD fluids	0		(2) (3) (7)
Vapour pressure		0	
Viscosity	0		(1) (3)
Viscosity index for HFD fluids	0		(2)
Water content of HFB and HFC fluids	0		(1)
Wax deposition		0	
Wear - sliding - see Lubrication			

Notes :

(1) A test forming part of the essential basis of acceptance or approval - see Parts III, IV and V of this report.

(2) A discretionary test for information purposes - see Part VI of this report.

(3) Alternative test methods are described.

(4) Test methods described included a method of test under development and assessment.

(5) A test method is not included but appropriate general requirements are mentioned in the report.

(6) A general survey of acceptable methods of test is given together with selected examples of test methods suitable for :

a) rolling fatigue;
b) sliding wear; and
c) use in a Vickers pump.

(7) Test methods described include tests under practical operating conditions in fluid couplings.

(8) See sections 4.2.2. and 4.3.2.

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