COMMISSION OF THE EUROPEAN COMMUNITIES

MINES SAFETY COMMISSION

FIFTH REPORT

on

specifications and testing conditions relating to
fire-resistant fluids used for power transmission

Luxembourg, 15 November, 1974

(This report supersedes the fourth report dated 26 March, 1971)

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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 qide 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 harmonizing 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 machinery 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 connexion 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 incresed 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. COPPÉ
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 deal, 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 obligatory 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 in 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. COPPÉ
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 of 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 technical criteria of inflammability and technological criteria. Comparative tests, carried out in the laboratories of the Technischer Überwachungsverein (Essen), the Versuchsgrubengesellschaft (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.

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PART I - GENERAL

(relating to fire resistant fluids for use in mines meeting 'Community of Six' requirements: i.e., those considered in the Fourth Report)

1.1. CLASSIFICATION

Fire-resistant fluids used for hydraulic transmissions can be divided according to their use in the following categories:

A = Emulsions of the oil-in-water type containing a maximum of 20% combustible matter.
Temperatures of use between + 5° and + 55 °C.

B = Emulsions of the water-in-oil type containing a maximum of 60% combustible matter.
Temperatures of use between + 5° and + 60 °C.

C = Aqueous solutions of polymers containing a minimum of 35% water.
Temperatures of use between - 20° and + 70 °C.

D + Fluids containing no water.
Temperatures of use between - 20° and + 150 °C.

1.2. TECHNICAL FLAMMABILITY CRITERIA

The flammability of the fluids is determined by tests described in PART III 3.1. and 3.2.

1.2.1. Determination of flammability of fluid atomized under pressure

The fluid for testing is atomized under a pressure of 70 bars; the jet is directed on to a precisely-defined oxyacetylene flame.

The operating conditions are described in PART III 3.1.

1.2.2. Determination of flame propagation in a mixture of coal dust and fluid

Flame propagation in a mixture of 75 g coal dust and 37.5 cm^3 of fluid is measured by heating one end of a test bar of minimum length 150 mm, width 20 mm and thickness 3 mm, in a flame produced by a propane gaz burner at about 1,000 °C.

The operating conditions are described in PART III 3.2.

1.3. HEALTH CRITERIA

The fire-resistant product must satisfy the following toxicological criteria:

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

The analytical procedure is described in PART IV 4.1. and 4.2.

However, whatever the results of this test, it cannot predict deleterious effects of allergic or other origin, in the long term or upon repeated exposure.

Hence approval can only be provisional, and any final acceptance can only be given after a period of practical testing.

1.4. TECHNOLOGICAL CRITERIA

a) Determination of softening point

The softening point, at which the product regains a degree of fluidity on emerging from the solid phase must lie sufficiently below the lower temperature limit at the place of use.

The apparatus used and method of operation are described in PART III 3.3.

b) Determination of viscosity

Fire-resistant fluids must be capable of being pumped in hydraulically operated equipment at ambient temperatures which may vary, according to the application, between - 20 °C and + 50 °C. The viscosity will be measured in accordance with the following table.

The viscosity is determined by means of the viscosimeter and the corresponding thermostats, which are described in PART III 3.4.

Data for viscosity measurements

Temperature	-20 °C	0 °C	+ 20 °C	+50 °C	+100 °C
Forms	-	-	A	A	-
	-	-	B	B	-
	C	C	C	C	-
	D	D	D	D	D

c) Determination of vapour pressure

Water-free fire-resistant fluids for use in hydraulic transmissions must not exhibit at 200 °C a vapour pressure substantially higher than that of currently used mineral oils of 140 °C.

Fluids containing water are not subject to this test at present.

The vapour pressure must be determined by means of the vapour-pressure measuring apparatus described in PART III 3.5.

d) Determination of pH

The pH of fire-resistant fluids of forms A and C serves as an index of the danger of contact with the skin. For fluids of forms B and D, the neutralization number must be determined.

Measurement of the pH must be carried out by the method described in PART III 3.6. The neutralization number must be determined by conventional methods (e.g. ASTM).

e) Determination of shear strength

The determination of shear strength, as an index of resistance to mechanical stresses, is essential for fluids for hydraulic transmissions, with the exception of form A fluids having a viscosity of less than 10 centistokes at 20 °C.

The measurement must be carried out by the method described in PART III 3.7.

f) Determination of anti-corrosive power

The anti-corrosive power of fire-resistant fluids is to be determined by the method described in PART III 3.8. for metals and alloys used in the construction of mining equipment which may contain the fluids listed above, particularly: steel, cadmium, copper, zinc, aluminium, brass (70/30).

g) Determination of the ageing of fluids

The methods for determining resistance to ageing are given:

a) for water-free hydraulic fluids (form D), in PART III 3.9.

b) for aqueous hydraulic fluids (forms A, B and C), in PART III 3.10.

Ageing is determined at 95 °C using copper and iron as catalysts, liquid oxygen being supplied continuously.

h) Determination of the behaviour of packings and seals

In order to avoid losses of fire-resistant fluids through packings and seals, the rate of variation in the volume of the packing or seal material must be as small as possible. The method is to be applied to all forms of fluids at 60 °C. Fluids of form D are tested at 150 °C.

Measurement of the variation in the volume of packings and seals and of the hardness is described in PART III 3.11.

i) Determination of wear protection

Tests have proved that it is not at present possible to evaluate anti-wear protection by use of a single method for all forms of fluids and all the various applications encountered in practice.

Teststing stations usually carry out the tests advocated in PART III 3.12. and 3.13. based on the 4-ball machine and the Vickers pump test bench. Work is proceeding on the development of other methods for testing wear with rolling contacts.

k) Determination of foaming tendency

Fire-resistant fluids should have the lowest possible foaming tendency.

The method of determination is described in PART III 3.14.

l) Determination of emulsion stability

Emulsion stability is determined for forms A and B only by the method described in PART III 3.15.

m) Miscibility and compatibility

For economic reasons it should be possible:

a) to mix, by virtue of miscibility, fresh, unused products of the same composition but of different origin, at least with forms C and D;

b) to mix, by virtue of compatibility, a new product with a similar lubricant of the same group already in use but of different origin.

No method of testing these two conditions had been developed at the time of publication of the report.

n) Determination of water content

This is done for inspecting the quality of deliveries and checking water absorption in use.

o) Determination of deaeration power

For the time being, the deaeration power of fire-resistant fluids may be determined for fluids of forms C and D. It is tested by means of the method described in PART III 3.16.

PART II - SPECIFICATIONS AND TEST CONDITIONS

(relating to fluids meeting 'Community of Six' requirements)

GENERAL

2.1. Conditions of approval

2.1.1. Fire-resistant fluids for hydraulic power transmissions and control, before being used in mines, must be granted a certificate confirming that they have undergone the following test cycle:

a) laboratory tests, described below (PART II 2.3. to 2.7. inclusive and PART III of the report)

b) long-term tests in normal service (PART II 2.8.).

2.1.2. The series of tests is to be carried out by a competent body, such as, at present, one of the following:

Germany	- Rheinisch-Westfälischer Technischer Überwachungsverein, Essen/Ruhr (Leitendes Fachinstitut) - Versuchsgrubengesellschaft mbH, Dortmund - Hygiene-Institut des Ruhrgebiets, Gelsenkirchen - Pharmakologisches Institut der Universität Hamburg, Hamburg
Belgium	- Institut national des industries extractives, Pâturages/Hainaut
France	- Laboratoire du Centre d'Études et Recherches des Charbonnages de France, Verneuil-en-Halatte - Laboratoire des Lubrifiants des Houillères du Bassin du Nord/Pas-de-Calais, Sin-le-Noble (Nord)
Luxembourg	
Italy	
Netherlands	

2.1.3. Authorization for the use of these fluids in coal mines is subject to the production of the certificate mentioned in (PART II 2.1., above.

2.2. Identification, classification and working temperature ranges

2.2.1. Fire-resistant fluids for hydraulic power transmission and control are identified by the following initials:

Federal Republic of Germany	HS
Belgium	THI
France	THI
Luxembourg	THI
Italy	
Netherlands	MH
United Kingdom	Fr

2.2.2. Four viscosity ranges have been provisionally laid down to cover the field of possible applications:

THI 1	1 to 1.5 centistokes at + 50 °C
THI 2	11 to 14 centistokes at + 50 °C
THI 4	20 to 40 centistokes at + 50 °C
THI 8	50 to 70 centistokes at + 50 °C

2.2.3. The categories of fire-resistant fluids for hydraulic transmissions at present available can be classified, on the basis of their use, in the following physical forms:

A - Emulsions of the oil-in-water type, containing a mximum of 20% combustible matter. Temperatures of use between + 5° and + 55 °C.

B - Emulsions of the water-in-oil type, containing a maximum of 60% combustible matter. Temperatures of use between + 5° and + 60 °C.

C - Aqueous solutions of polymers containing a minimum of 35% water. Temperatures of use between - 20° and + 60 °C.

D - Fluids containing no water. Temperatures of use between - 20° and + 150 °C.

2.2.4. The full identification of a fire-resistant fluid for hydraulic transmissions and controls will therefore be one of the following:

THI 1-A

THI 2-A, THI 2-C, THI 2-D

THI 4-A, THI 4-B, THI 4-C, THI 4-D

THI 8-B, THI 8-C, THI 8-D

2.2.5. Fluids of forms B, C and D are examined as-delivered. Form A fluids are examined after dilution according to the manufacturer's instructions, using the concentrate supplied and the test water defined in PART III 3.8. and 3.15.

SPECIFICATIONS AND TEST CONDITIONS

PART II Paragraphs 2.3. to 2.6. show the results to be obtained in the laboratory tests listed and described in PART II 2.7.

2.3. Flammability criteria

2.3. 1. Flammability of fluid atomized under pressure PART III 3.1.

The fluid must be given the rating "1" or "2" according to PART III 3.1.4. of PART III 3.1. in five consecutive tests.

(Note: Rating "2" may also be given if occasional flame peaks reach the screen when the burner is 1.20 m from the spray nozzle.

2.3. 2. Flame propagation in a mixture of fluid and coal dust (PART III 3.2.)

The flame must not spread appreciably the field of action of the burner flame. The arithmetic mean of the two sets of 10 measurements must not exceed 10 cm, 95% of the individual measurements (10 out of 20) being 13 cm or less; the anomalous measurement may be eliminated from calculation of the mean.

2.4. Health criteria

2.4.1. a) The toxicity tests of water-containing products are carried out by the techniques described in PART IV 4.1. Water-free products are examined by the techniques described in PART IV 4.2.

b) Products given a rating of 10 in any one test, or of 50 or over, after weighting of all results, will be rejected.

2.4.2. The toxicological laboratory responsible for performing the tests may communicate direct with the manufacturers of the fluids tested, subject to trade secrecy.

2.4.3. The toxicological laboratory performing the tests is the sole judge of the conclusions it submits to the competent bodies. These findings will be forwarded upon request to the manufacturers of fluids or to any toxicological laboratories which might be approved.

2.5. Physical criteria

2.5.1. Softening point (PART III 3.3.)

The softening point is determined before and after the shear test. The softening point of the new fluid serves only as a reference. The permissible limit values for modification of the softening point after the shear test are shown in Table 2.

2.5.2. Viscosities (PART III 3.4.)

These must correspond to the figures set out in Table I below:

TABLE I

Series	Form (for guidance only)	Kinematic visocisities in centistokes at				
		- 20 °C	± 0 °C	+ 20 °C	+ 50 °C	+ 100 °C
THI 1	A	No determinations				
THI 2	A			< 50	< 11/14	
	C	< 1 800	< 170	< 50	< 11/14	
	D	< 1 800	< 170	< 50	< 11/14	
THI 4	A			< 190	< 20/40	
	B			< 190	< 20/40	
	C	< 5 000	< 800	< 190	< 20/40	
	D	< 5 000	< 800	< 190	< 20/40	> 6
THI 8	B			< 360	< 50/70	
	C	< 12 000	< 1 800	< 360	< 50/70	
	D	< 12 000	< 1 800	< 360	< 50/70	< 10

The figures shown for - 20 °C and ± 0 °C are to be regarded as provisional limits.

2.5.3. Vapour presssure (PART III 3.5.)

At the present time this test is restricted to water-free fluids. The vapour presssure of such a fluid at 200 °C must not substantially exceed that of mineral oils. It must be below 0.5 bar.

2.5.4. pH (PART III 3.6.)

The pH must lie between 7.5. and 9.5. inclusive.

This measurement is carried out only on form A and C fluids. For form B and D fluids, the neutralization number is determined.

2.5.5. Shear strength (PART III 3.7.)

The characteristics of the fluid collected to be measured are given in Table II below. The variations from the values given by the same fluid before the shear test must not exceed the limits shown in Table II.

TABLE II

Characteristics measured	Fluid forms			
	A (1)	B	C	D
Kinematic viscosities at:				
- 20 °C	-	-	≦ ± 25 %	≦ ± 20 %
± 0 °C	-	-	≦ + 25 %	≦ ± 20 %
+ 20 °C	≦ ± 10 %	≦ ± 10 %	≦ ± 15 %	≦ ± 10 %
+ 50 °C	≦ ± 10 %	≦ ± 10 %	≦ ± 15 %	≦ ± 7 %
Softening point	≦ + 3 °C	≦ + 3 °C	≦ + 3 °C	≦ + 3 °C
pH	≦ ± 0.5	-	≦ ± 1	-
Neutralization number (mg KOH/g)	-	≦ ± 0.5	-	≦ ± 0.5
Reduction in water content, %	≦ 15	≦ 5	≦ 8	-

(1) Except THI 1 - A fluids

2.5.6. Anti-corrosive power (PART III 3.8.)

There should be not perceptible corrosion of metals or alloys used in the construction of the different machines: the reduction in weight must not exceed 10 mg per sample for metals of specific weight exceeding 7 g/cm^3 and 5 mg per sample for metals of specific weight below 7 g/cm^3. For cadmium and zinc the loss of weight must not exceed 20 mg per sample and for aluminium 10 mg.

No deposit should normally be observed on the samples: the increase in weight must not exceed 5 mg per sample.

Metal surfaces immersed in the fluid must not show any appreciable change in colour. The same applies to metal surfaces outside the fluid. If the colour does change, the reasons must be investigated.

2.5.7. Ageing (Annex IX-A and IX-B)

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

a) Fluids of forms B and D. Test duration 600 hours. Increase in neutralization number $\leq$ 2 mg (KOG/g for both B and D forms of fluid. P_1 is the percentage insoluble in benzene for the new fluids and P_2. The percentage after ageing for a period of 600 hours, P_1-P_2 must be $\leq$ 2% for form D fluids.

b) Fluids of forms A (except fluids THI I-A) and C. Test duration: 200 hours. After this time the pH must not be $\leq$ 4.

c) Metal coils must not become heavily fouled or corroded.

2.5.8. Behaviour of packings and seals (PART III 3.11.)

No appreciable deterioration must be observable in the material used for packings or seals; in particular there must be no variation in volume, hardening or modification of the surface (cracks).

The maximum permissible variations are:

a) Swelling: 4% of the volume of the test piece.

b) Shrinkage: 2% of the volume of the test piece.

c) Shore hardness: ± 4.

2.5.9. Wear protection (PART III 3.7. and 3.13.)

No permissibility criteria can be fixed at present. The 4-ball machine, and othe apparatus, is suitable for testing the regularity of manufacture of the fluids.

2.5.10. Foaming tendency (PART III 3.14.)

Fire-resistant fluids must not form greater volumes of foam than are allowed with conventional petroleum products, e.g. 300 ml.

2.5.11. Emulsion stability (PART III 3.15.)

This test is carried out only on forms A and B. The following limit values must be observed:

a) Cream level $\leq$ 5 mm after 600 hours at + 20 °C or at + 50 °C.

b) No separation of water and oil is allowed. However, an oil level of less than 15 mm diameter will not be regarded as separation.

2.5.12. Miscibility and compatibility

In principle, fire-resistant fluids within forms C and D should be mutually miscible in the unused state. Furthermore there should be compatibility within each form A, B, C and D, between a used fluid and fresh fluid of the same form used for topping-up.

2.5.13. Water content (standard method)

Water content serves as the criterion for identification and testing of changes in the fluid during use. To determine water content, xylene is used according to the distillation method AFNOR NFT 60-113 as fluid for entraining fluids of forms A and B, and benzene for form C. For form D the FISHER ASTM-D 1533 method is preferred.

2.5.14. Density (standard method)

The density is measured for purposes of identification. It should preferably be below 1.5.

2.5.15. Ash content (standard method)

Ash content is measured for identification purposes. The following values should preferably not be exceeded:

Form A : 4 %, determined on the concentraded flammable product;
Form B : 1.5%, determined on the fluid ready for use;
Forms C and D : 2 %, determined on the fluid ready for use;

The ash must in no case be abrasive.

2.5.16. De-aeration power (PART III 3.16.)

The de-aeration power is measured at 50°C for liquids in Group B, C and D, and for those in Group A, or up to 25°C. For the time being the value obtained is to be considered as serving for identification purposes; limit values will be laid down later, after longer practical experience.

2.6. Tolerances in the measurement of individual characteristics

Methods:

M 1 - PART IV 3.1. - Combustion of atomized jet over a flame: no tolerance
M 2 - PART III 3.2. - Flame propagation: ± 5 mm of the propagation length of the flame
M 3 - PART III 3.3. - Softening point: ± 3 °C
M 4 - PART III 3.4. - Viscosities: ± 10%
M 5 - PART III 3.5. - Vapour presssure: ± 10%
M 6 - PART III 3.6. - pH: ± 0.1 pH
M 7 - PART III 3.7. - Shear strength: ± 3% of viscosity values
M 8 - PART III 3.8. - Anti-corrosive power: ± 2 mg/sample
M 9 - PART III 3.9.-3.10. - Ageing: ± 0.5 mg KOH/g; ± 0.3 pH; ± 0.2% insolubles in benzene
M 10 - PART III 3.11. - Behaviour of packings and seals: ± 25% of mean value determined
M 11 - PART III 3.12.-3.11.- Wear protection: still being determined
M 12 - PART III 3.14. - Foaming tendency: ± 25%
M 13 - PART III 3.15. - Emulsion stability: ± 50% of the height of the separated layer
M 14 - - Miscibility and compatibility: no tolerance
M 15 - - Water content: not yet determined
M 16 - - Ash content: ± 20%
M 17 - PART III 3.16. - De-aeration power: ± 5 min.

2.7. List of analysis and test methods

Tests	Method described in the following paragraphs of PART III	Germany	Belgium	France	Netherlands	U.S.A.	U.K.
3.1. Spray combustion	3.1.			NF. 48 618			NCB 510/1970
3.2. Flame propagation	3.7.			NF. 48 620			- " -
5.1. Pour point	3.3.			NF. 48 611			IP 16/67
5.2. Viscosity	3.4.	DIN 53015					IP 71/66
5.3. Vapour pressure	3.5.			NF. 48 613			
5.4. pH	3.6.			NF. 48 617			
5.5. Shear strength	3.7.	DIN 51312					
5.6. Anti-corrosive power	3.8.						IP 133/67
5.7. Ageing of water-free fluids	3.9.	DIN 51587			NEN 3024	ASTMD 943-54	
water-containing fluids	3.10						
5.8. Behaviour of packings and seals	3.11	DIN 53521					NCB 463
5.9. Wear protection	3.12.a.3.13.	DIN 51350 or 51386 E		NF.E. 48 617			
5.10. Foaming tendency	3.14.					ASTMD 892-63	
5.11. Emulsion stability	3.15			NF. 48 615			NCB 570
5.12. Miscibility and comptability		DIN 51582 DIN 51777					
5.13. Water content				T 60-113		ASTMD 1533	NCB. 570
5.14. Density		DIN 51757	NBN 52011 and 52015	T 60-101	N 907		
5.15. Ash content		DIN 51575	NBN 52044	T 60-111			
5.16. De-aeration power	3.16.	DIN 51381		NF. 48 614			
4.2. Toxicological test: fluids containing water (A+C)	PART IV 4.1.			NF.E. 48 621			
fluids containing no water (D)	PART IV 4.2.			NF.E. 48 621			
Flash point							IP 34/67

2.8. Tests in practical service

1. These tests are carried out in a mine at the request of the competent body, with the authorization of the competent departments.

   The conditions for carrying out the test are to be agreed between the parties mentioned above.

2. The supplier of the test fluid will be allowed to attend the tests only if expressly agreed. In such case the competent body must be informed.

3. These tests must continue for at least 6 months.

2.9. Withdrawal of approval

At the request of the competent body, authorization for use in mines may be withdrawn by the issuing authority.

PART III - DESCRIPTION OF TEST PROCEDURES

(relating to fire resistant fluids meeting 'Community of Six' requirements).

3.1. DETERMINATION OF IGNITABILITY OF FLUID ATOMIZED UNDER PRESSURE

3.1.1. 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.

3.1.2. APPARATUS

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

b) Pressurizer for atomizing the fluid (Diagram A or B in plate -a- below):

Diagram A: A cylinder of compressed nitrogen with a pressure-reducing valve set to 70 ± 3 bars. 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.

Diagram B: A pressure generator, consisting of a pressure pump connected to the spray nozzle via a metal pipe on which are mounted the presssure gauge and a pressure regulator, which discharges excess fluid back into the tank.
The pressure regulator is calibrated to 70 ± 3 bars. The spray nozzle is connected directly to a valve.

c) A test spray nozzle as shown in drawing (see plate -b- below).

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 $\leq$ 0.4 mm mesh sieve (144 meshes per cm^2), with a minimum diameter of 10 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 -c-).

Each of the two gases leaves its container under presssure 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 10 bars gauge pressure; the range for the acetylene manometer is 0 to 1.6 bars.

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

The oxyacetylene welding torch, Rex No. 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 litres/minute of oxygen and 15 ± 1 litres/minute of acetylene are discharged, at a pressure of 5.0 bars 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 screen 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.

3.1.3. TEST PROCEDURE

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

When the temperature of the fluid is 65 ± 5 °C if the apparatus shown in diagram A is used, or 65 ± 2 °C with the apparatus in diagram B, and when its pressure has been set at 70 ± 3 bars, 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 fromt 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/sec. The flame is then kept stationary for 5 sec. at the maximum distance of 1.20 m. Thus the whole test takes 35 sec. 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 diagram B, the test apparatus must be flushed carefully with the fluid under test.

3.1.4. EXPRESSION OF TEST 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).

Note 1: Rateing '2' may also be given if occasional flame peaks reach the screen when the burner is 1.20 m from the spray nozzle.
Note 2: This method is based on the American method AMS-3150 C. The atomizing pressure and the dimensions of the nozzle are as defined therein.

-(a)-

Annex I

DETERMINATION OF IGNITABILITY OF FLUID ATOMIZED UNDER PRESSURE

Diagram A

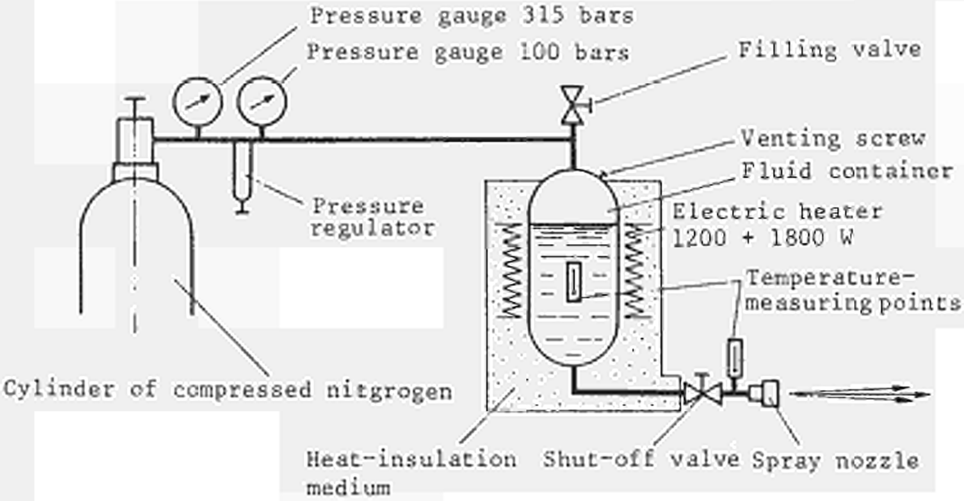
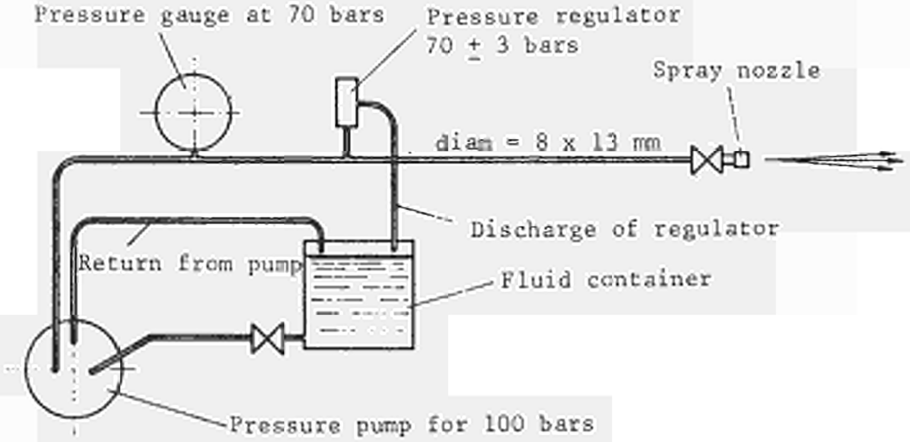


Diagram B



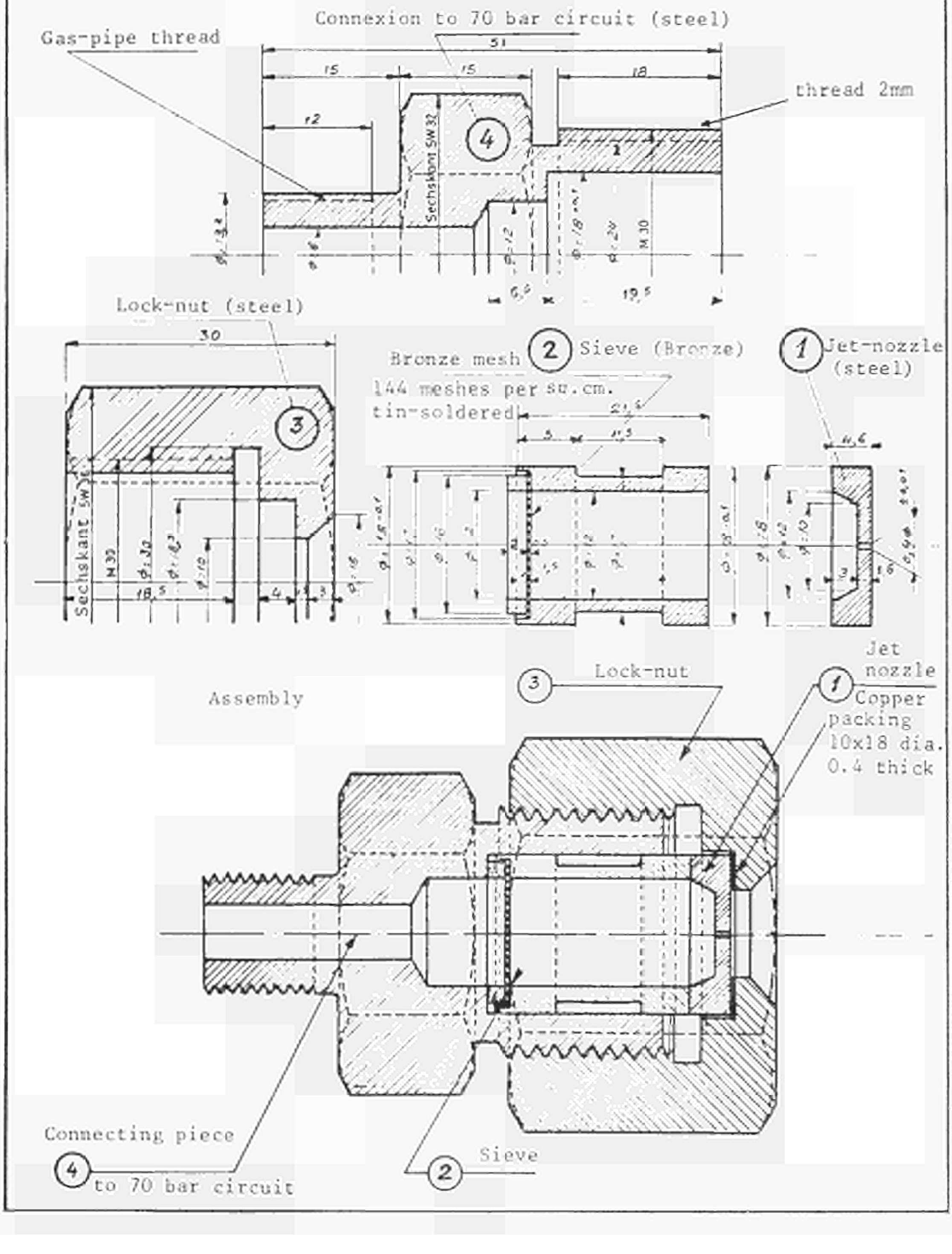
-(b)-

Annex I

DETERMINATION OF IGNITABILITY OF FLUID ATOMIZED UNDER PRESSURE

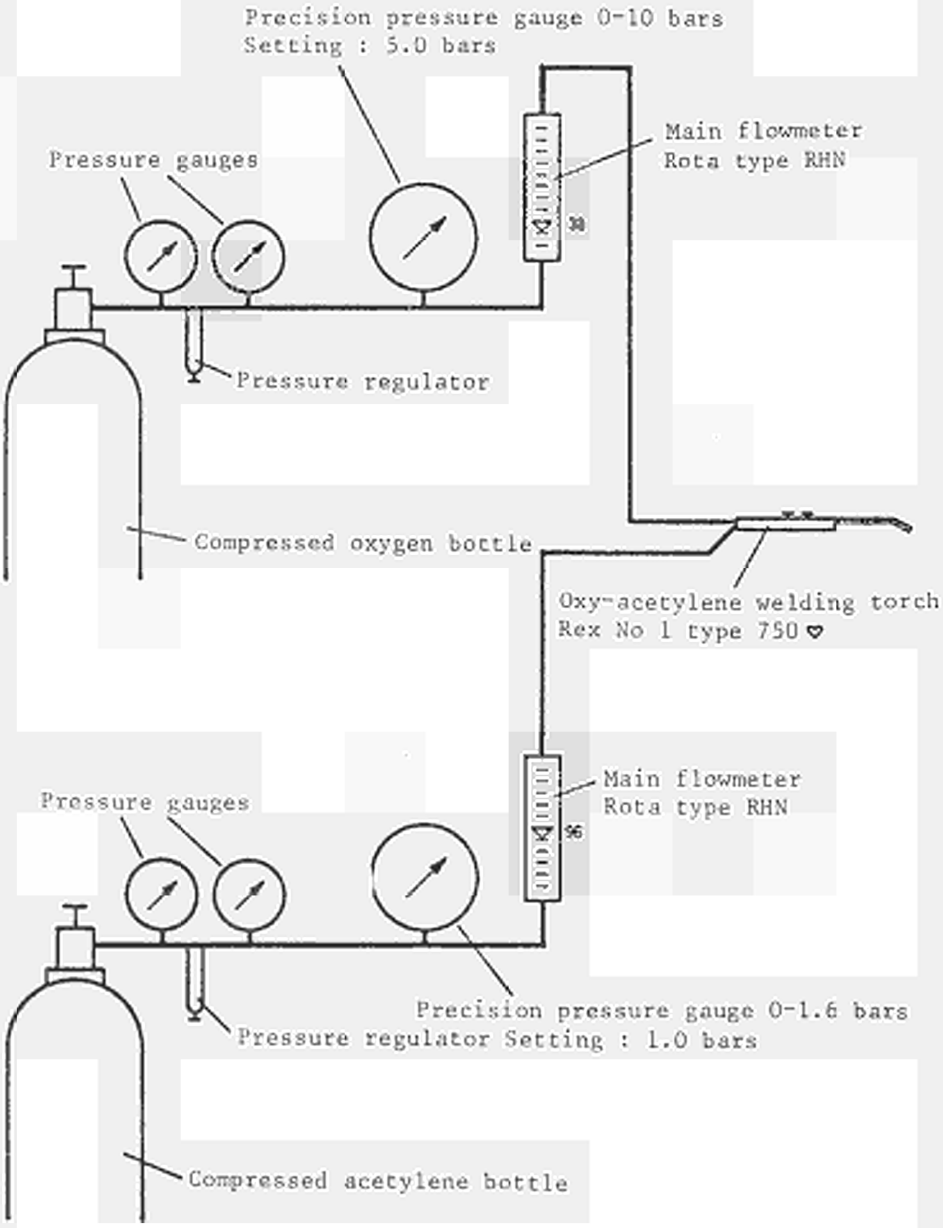
Components of test nozzle

Scale 2:1



Annex I

DETERMINATION OF IGNITABILITY OF FLUID ATOMIZED UNDER PRESSURE



FLAME-PROPAGATION TEST IN A MIXTURE OF THE FLUID WITH COAL DUST

3.2.1. PRINCIPLE

The propagation of a flame is measured in a mixture of 75 g of coal dust and 37.5 cm^3 of fluid. The test is carried out in an enclosure at ambient temperature without artificial ventilation.

3.2.2. APPARATUS REQUIRED

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 test apparatus (support for the test piece plate, support for the termo-couples 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.

3.2.3. COAL DUST

The coal dust used for the tests is taken from Montrambert coal *), prepared and supplied by the Centre d'études et de recherches des Charbonnages de France, Verneuil-en-Halatte (Oise). The average characteristics of this dust are follows:

Moisture	1.4 % (tolerance ± 20 % of 1.4. %)
Ash, dry basis	10.3 % (tolerance ± 10 % of 10.3. %)
Volatile matter, dry basis	27.1 % (tolerance ± 10 % of 27.1. %)
Particle size	0 to 100 mircrons

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

*) Note: This scam is now nearly exhausted and an alternative Source of dust is being sought.

3.2.4. PREPARATION OF THE TEST MIXTURE

The test mixture must be in proportions of 75 g coal dust to 37.5 cm^3 of the fluid to be tested. Two mixtures are prepared, each sufficient for 10 determinations.

In principle a procelain 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.

3.2.5. PREPARATION OF 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 text mixture must be stirred once again. The test piece is at least 15 cm long and 20 mm thick. The test piece plates bear a datum mark 15 cm 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.

3.2.6. TEST PROCEDURE

The gas pressure is set at 650 mm/w.g. 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 3.5 cm.

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, ½ hr 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. The tip of ther burner is 45 mm away from the undersurface 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.

3.2.7. RESULTS AND TOLERANCES

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

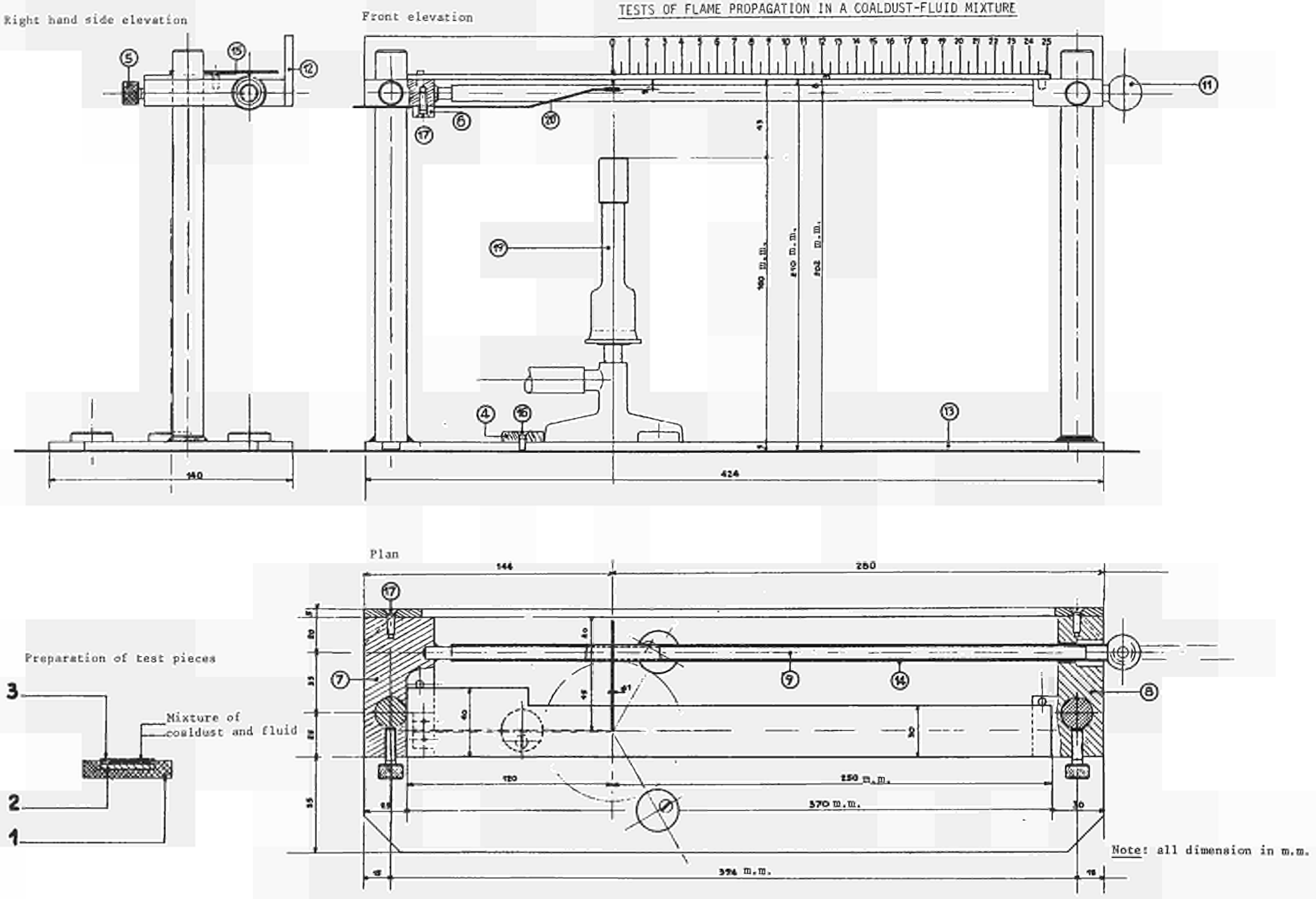
3.2.8. NOTES

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

b) The steel plates for the test piece 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.

TESTS OF FLAME PROPAGATION IN A COALDUST-FLUID MIXTURE

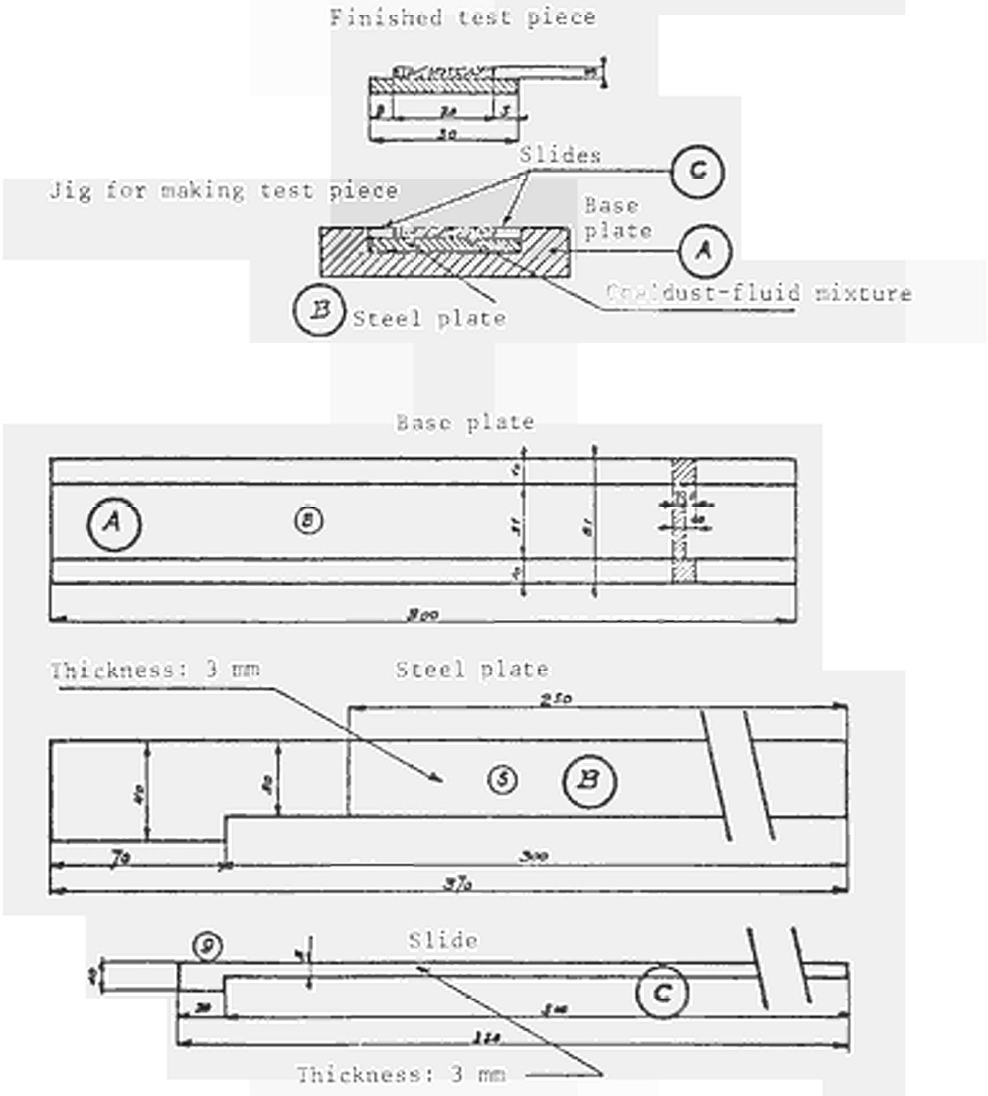


Note: all dimension in m.m.

-(b)-

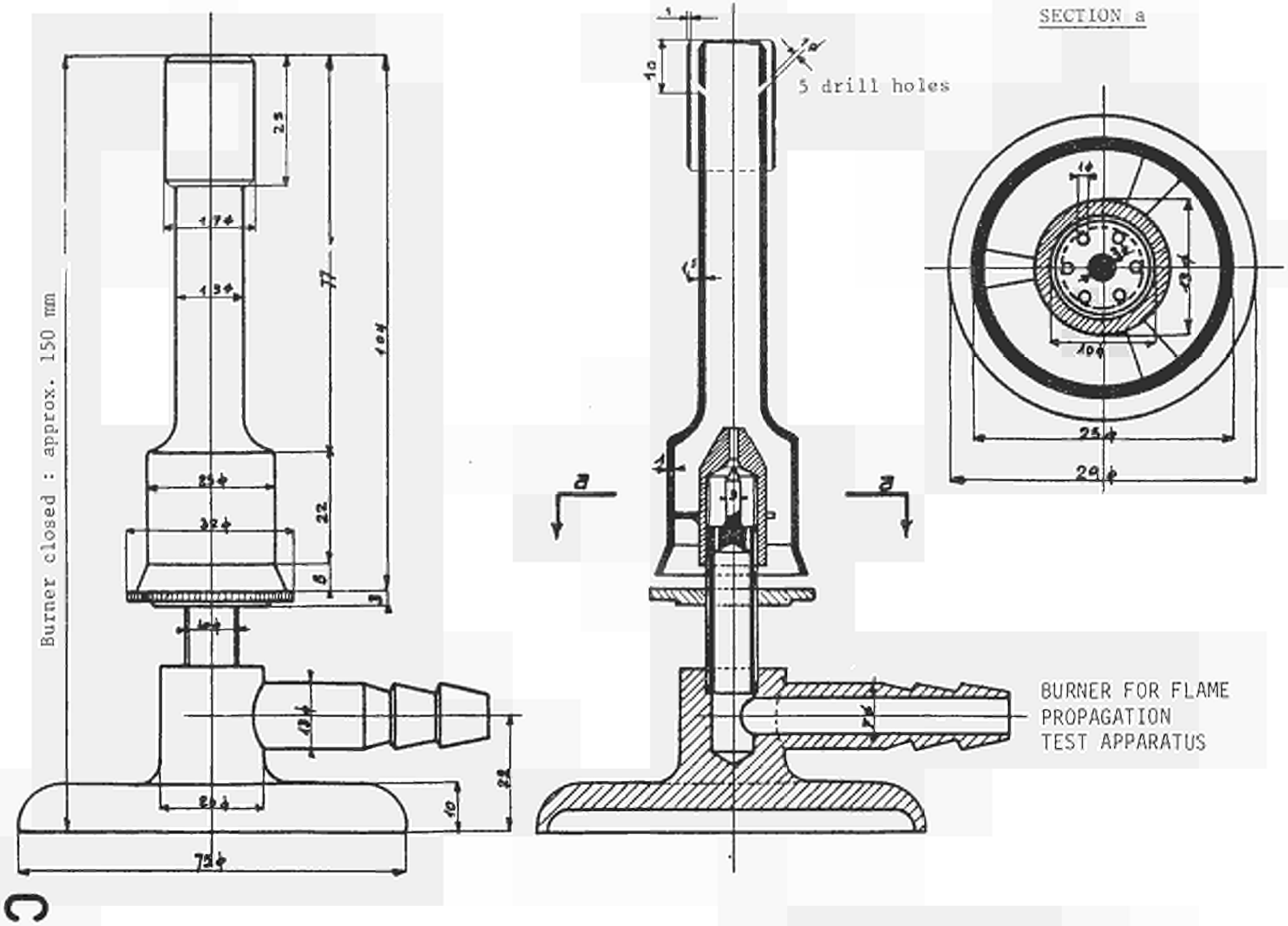
MAKING OF TEST PIECE

Scale 1:1 and 1:2



B

Note: all dimensions in mm.



BURNER FOR FLAME
PROPAGATION
TEST APPARATUS

Note: all dimension in mm.

DETERMINATION OF THE SOFTENING POINT (POUR POINT)

3.3.1. INTRODUCTORY REMARKS

With the appartus used, the softening point can be determined from a thin layer of fluid; this precaution is ncessary because of the poor conductivity of solidified substances. The test is carried out in an air bath of accurately-known temperature and under thermal conditions which allow for the structural changes occurring as a result of variable cooling and avoid any irregularities which might result from these changes. Variations of temperatur between successive tests should, as a general rule, not exceed a few tenths of 1 °C.

Particular attention is drawn to the fact that tests to determine viscosity at low temperatures and tests to determine softening points are essentially different. Each one is a particular test and its results are applicable only to the corresponding type of test.

3.3.2. PRINCIPLE

The softening point is the temperature at which the fluid, after having been thoroughly and rapidly solidified by specific thermal treatment and then gradually reheated, loses its rigidity sufficiently to release a movable object trapped in the fluid as it solidified, under a constant load.

3.3.3. APPARATUS

The apparatus illustrated in the accompanying diagram consists of a ring-shaped container A (representing the moving element) intended to hold the fluid under investigation. The dimensions of the container are: inside diameter of the inner wall 15 mm; inside diameter of the outer wall 21 mm:, inside radial width 3 mm; wall thickness 0.4 mm; depth 11 mm; and bottom thickness 1.5 mm. This container is fastened to a hollow metal rod C, topped by a white marker D, by means of a perforated disc B. This vertical rod can be inserted in a brass tube E forming a sleeve of inside diameter 15 mm, outside diameter 16 mm and length 234 mm; at its lower end is a brass cylindrical component F of inside diameter 17 mm, outside diameter 18 mm and height 31.5 mm, which acts as a plunger when pushed into the ring-shaped container A. This cylinder F is pierced by two openings G diametrically opposed and measuring 12 mm high by 5 mm wide; the bottom of these openings is 13.5 mm above the bottom of cylinder F. Rod C is centred and guided by component H and a head I with a play of about 0.1 mm at the bottom and top of sleeve E. In order to reduce the conductivity of the metal and to avoid the formation of frost, sleeve E is perforated by six rows of two holes of diameter 10 mm, as shown in the figure; rod C is also perforated by 17 rows of 2.5 mm diameter holes. The middle of the perforated section of rod C is at the level of the top of the head I. A pin J can be inserted in the hole of rod C tangentially to the top of head I and consequently it can lock the moving assembly in the sleeve; a rigid arm M ending in a fixed screw clamp holds sleeve E in a vertical position while allowing rod C and container A to move freely. The weight of the moving assembly is calibrated at exactly 20g; it will drop if it is not held up. At the beginning of the test, container A, filled with solidified fluid, remains fixed to component F, which was immersed in the fluid before freezing. When softening occurs, the moving assembly slides down and finally reaches the end of its travel, limited by pin J, the latter having been withdrawn after solidification from its initial position and inserted in the end hole of rod C.

The whole apparatus is immersed in a cooling system of two concentric containers consisting of two tubes of diameter 35 and 75 mm, both closed at one end.

The acetone-solid CO_2 cooling mixture (250 cm^3) is placed between the two vessels. The inner tube holds the metal test apparatus and a pentane thermometer for measuring the softening point.

A highly sensitive detector may be placed on the end of C; it makes an electrical circuit as soon as the rod begins to slide and can thus actuate a warning signal.

3.3.4. PROCEDURE

a) The test sample should be at the ambient laboratory temperature.

b) The test fluid, at laboratory temperature, is poured into the ring-shaped container A of the apparatus, and rod C is inserted in sleeve E (the white marker on C facilitates this operation). The lower end of the sleeve is now immersed in the annular container until it ouches the bottom.

Any excess fluid which has spilled over the top of the annular container must be wiped off. The moving assembly is locked in position by inserting pin J in the hole in rod C level with head I.

c) In a 250 cm^3 Dewar flask (internal diameter 50 mm, depth 150 mm) a cooling bath at - 70 °C is preparated by mixing excess solid carbon dioxide with acetone. A 25 x 200 mm Pyrex test tube is immersed in the mixture thus prepared. The apparatus is inserted in this tube so that the test fluid is cooled until it solidifies.

The apparatus is left in the cooling bath for 30 minutes.

d) In the cooling system described above, a cooling bath with a temperature of less than - 70 °C is prepared.

e) After 30 minutes of immersion in the Dewar flask, the apparatus is removed from the test tube and returned to the central tube of the cooling system; it is fastened to the rigid arm M. Pin J is removed and inserted in the topmost hole of C. The bath is allowed to warm up spontaneously. It is not necessary to stir the acetone bath, since the release of carbon dioxide bubbles is sufficient to ensure a constantly uniform temperature distribution.

f) Eventually the annular container begins under its own weight to move down from the fixed sleeve and, as soon as this downward movement begins, the electrical circuit is made and the acoustic signal given. The downward movement of the moving assembly is stopped by the pin which impinges on the head I of the sleeve.

3.3.5. EXPRESSION OF RESULTS

The temperature at which the ring-shaped container separates completely from the sleeve is taken as the normal softening point. However, the reheating time must be at least 10 minutes. Three softening tests are carried out and the mean of the three is taken as the softening point.

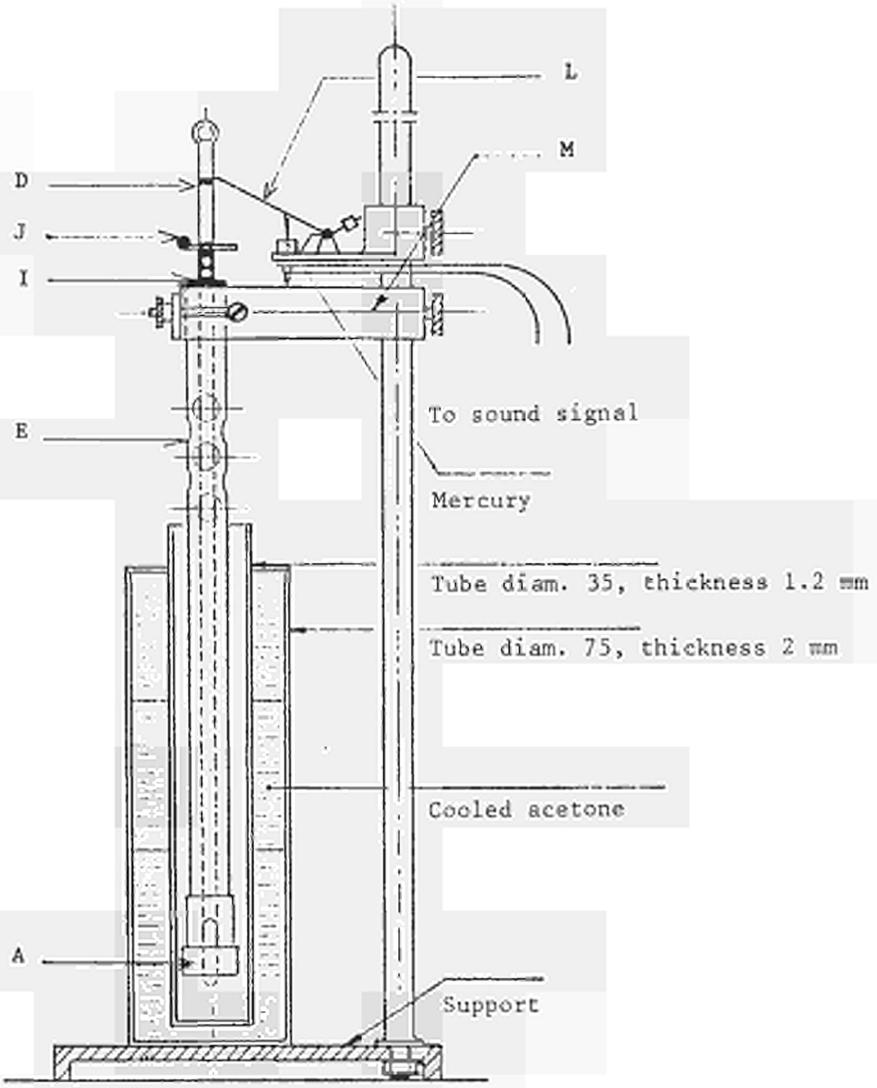
3.3.6. SPECIAL CASE OF PRODUCTS WITH SOFTENING POINTS ABOVE 0 °C

If the softening point of the product tested is above 0 °C, the acetone bath will warm up only slowly from this temperature. In order to counteract this, and also to permit the determination of softening points above ambient temperature, a heating plate is inserted between the support of the apparatus and the outer vessel. Resistors are used to control the current in the heating coil placed under the plate in such a way that the temperatur of the acetone rises evenly at about 0.8 °C per minute.

The procedure is similar to that described above. However, when the temperature of the acetone bath reaches 0 °C, the current in the heating plate is switched on; the test is then continued and, as before, the temperature at which the annular container separates completely from the sleeve under its own weight is recorded.

-(a)-

DETERMINATION OF THE SOFTENING POINT

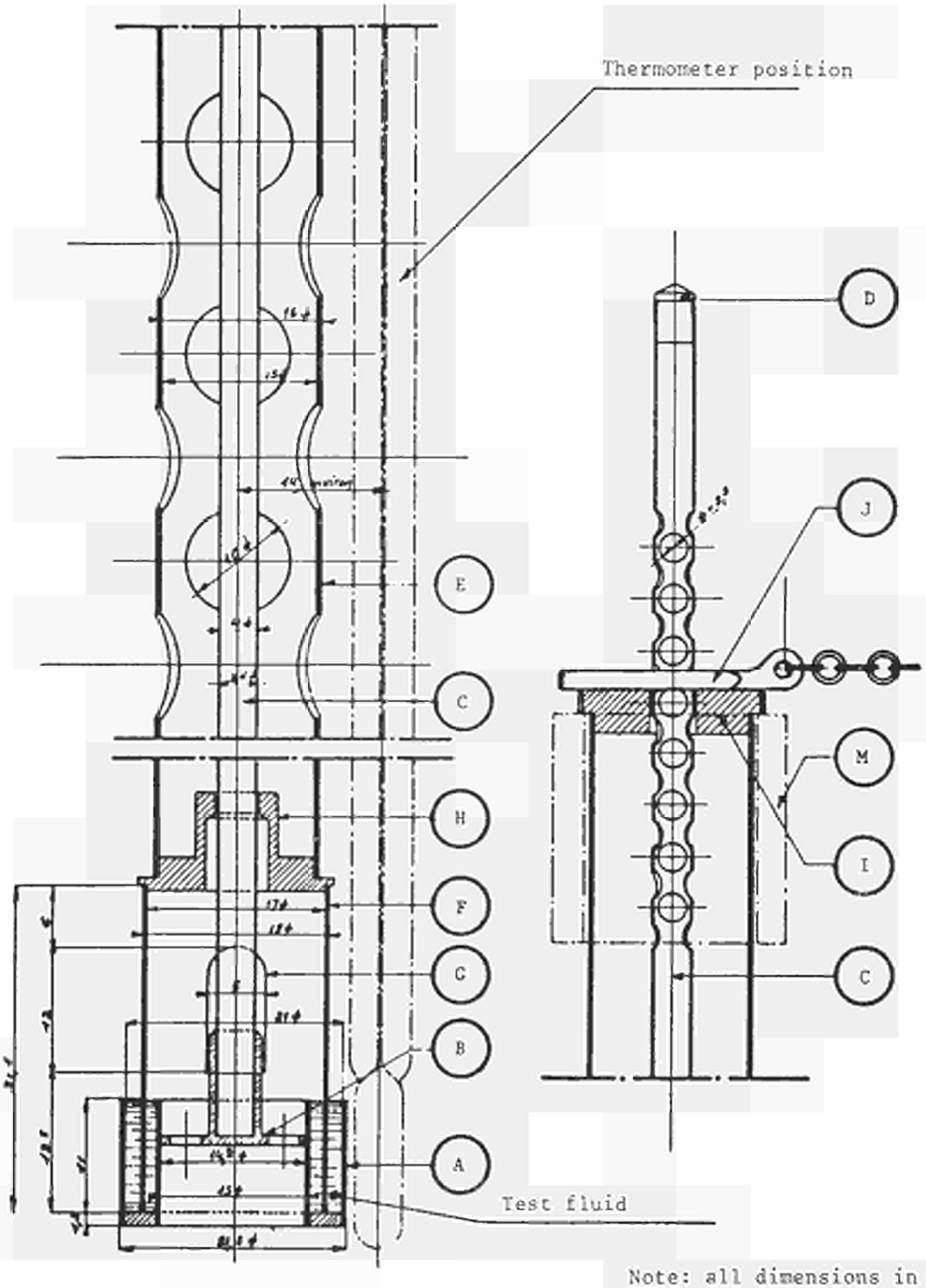


GENERAL DIAGRAM

Note: all dimension in mm.

-(b)-

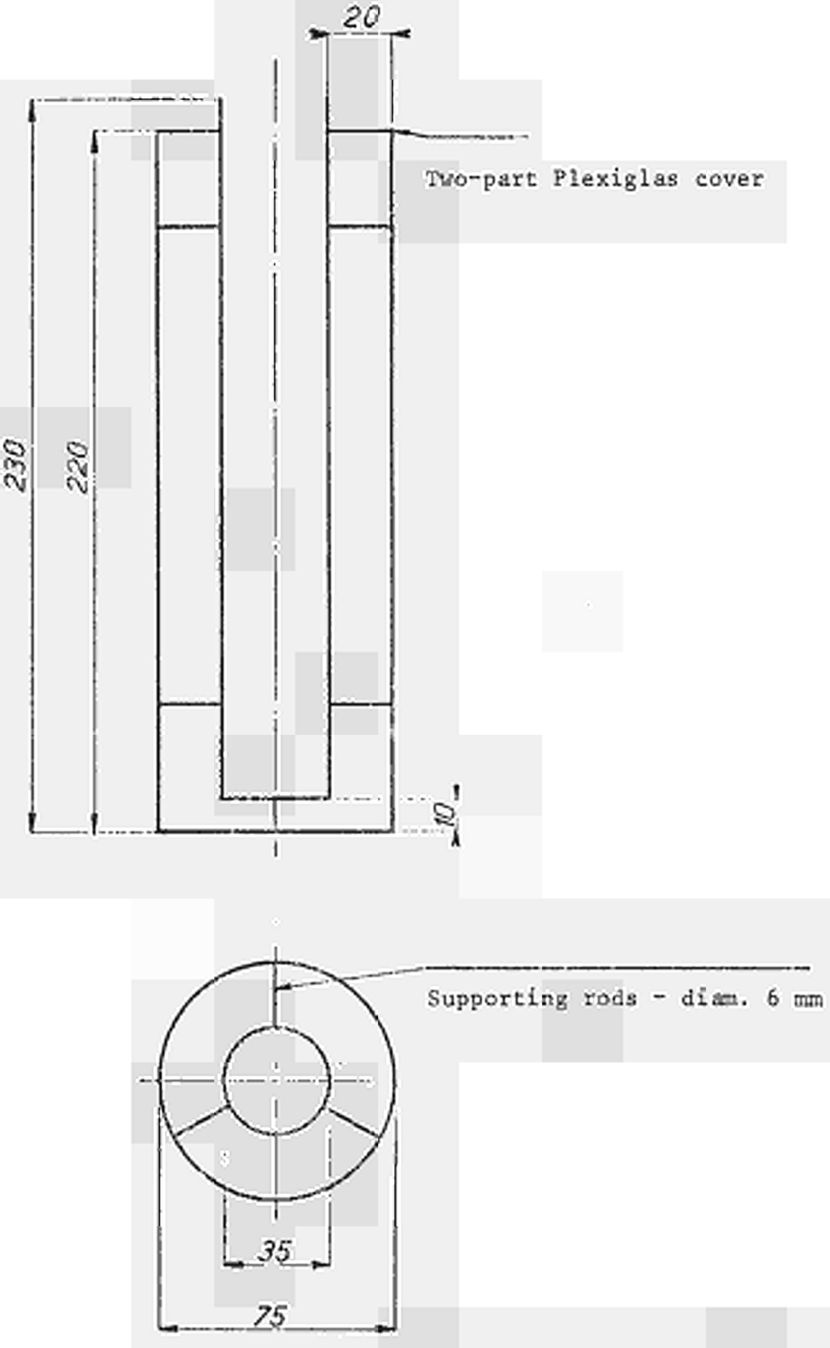
DETERMINATION OF THE SOFTENING POINT



Note: all dimensions in mm.

-(c)-

DETERMINATION OF SOFTENING POINT



1 glass tube diam. 35; thickness 1.2 mm
1 glass tube diam. 75; thickness 2 mm
glass rods diam. 6 mm

CONTAINER FOR ACETONE-SOLID CO_2 BATH

Note: all dimensions in mm.

3.4. DETERMINATION OF KINEMATIC VISCOSITIES

3.4.1. PRINCIPLE

A calibrated tube with two reference marks is filled with the test fluid. A calibrated ball is then introduced into the tube and the falling time of the ball between the two reference marks recorded. This time is used to derive the viscosity by the following simple calculation:

ηa = Absolute viscosity of the fluid in centipoises

ηc = 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

$$\eta a = t \times (D - d) \times K$$

$$\eta c = \frac{\eta a}{d} = t \times \frac{D - d}{d} \times K$$

3.4.2. 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 densitiy 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.

3.4.3. APPARATUS

a) Viscosimeters

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

aa) The Höppler viscosimeter, type BH, with which measurement is carried out in a tube inclined at an angle of 20° to the vertical;

bb) The IFC viscosimeter, with which the measurement is carried out in a tube inclined at 30° to the vertical.

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 typ 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:

aa) ± 0.05 °C at measurement temperatures below + 20 °C;

bb) ± 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:

i) purified kerosene (or aviation spirit) for temperatures below + 20 °C;

ii) pure glycerine or white vaseline oil for temperatures 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 Franch Standard (AFNOR - T 42-011) and in the corresponding German Standard (DIN 53015).

3.4.4. MEASUREMENT 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 previously 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.

3.4.5. NOTES

a) Constants 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.

b) 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 figure 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.

For forms A and B, 5 consecutive measurements must be made, each value being indicated.

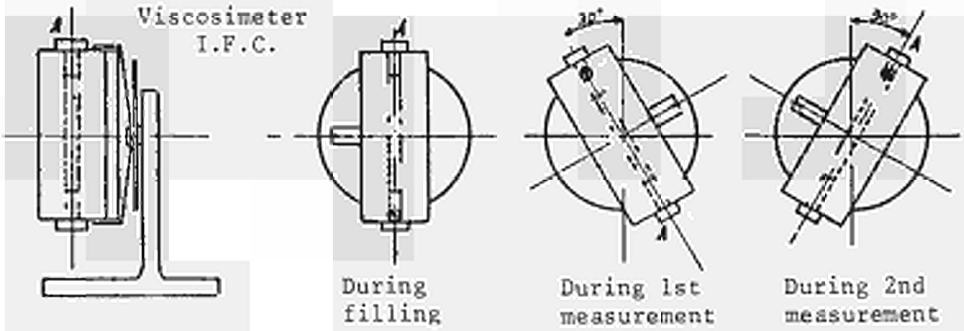
c) Cleaning the calibrated tube and the balls

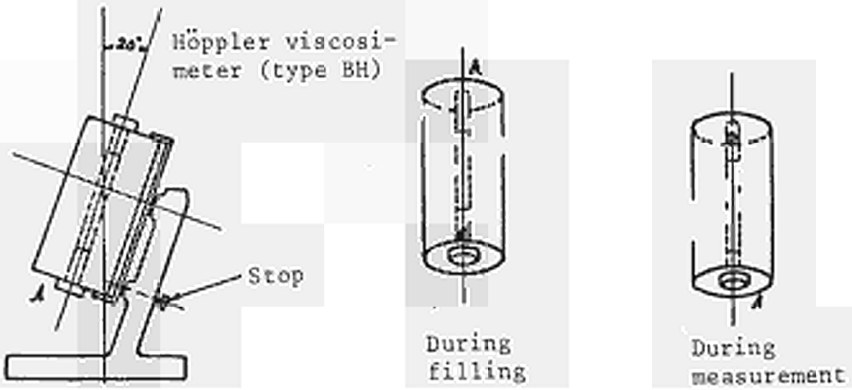
The tube is cleaned with solvents and rinsed with pure ether. In the case of aqueous solutions, clean with a hot solution of soda containing 5% concentrated ammonia to remove any grease, and then rinse with distilled water.

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

-(a)-

MEASUREMENT OF KINEMATIC VISCOSITY





DETERMINATION OF VAPOUR PRESSURE

3.5.1. PRINCIPLE

The vapour pressure of form D fluids is to be determined under given conditions at temperatures ranging from room temperature to the maximum temperature of utilization. The highest testing temperature is 200 °C. The values of vapour pressure are expressed in bars.

3.5.2. APPARATUS

a) A bomb capable of being dismantled,

b) a mercury pressure gauge,

c) a thermostat-controlled bath,

d) a temperature-measuring device.

a) Bomb capable of being dismantled

The bomb used is derived from the Reid bomb, but constructed in such a way that it can be cleaned easily. It consists of two connecting chambers, as follows:

aa) Chamber for test fluid:

internal diameter 54 mm
height of the inner cylindrical portion 60 mm;

bb) Air chamber:

internal diameter 54 mm
height of the inner cylindrical portion 250 mm.

The two chambers are connected by an intermediate piece with a lateral orifice serving as a vent during filling, and as a level indicator for the test fluid. Above the air chamber is the connecting tube to the mercury pressure gauge. The temperature measuring device is also attached to this tube, a seal being fitted. For isolation of the bomb, a leakproof cock is fitted in the connexion between the bomb and the mercury gauge.

The volumetric ratio between the air chamber and the fluid chamber is about 4. The accompanying diagram shows the bomb as a whole.

b) Mercury pressure gauge

This pressure gauge corresponds to the accompanying sketch. At the lower end is a leakproof cock by means of which the level of the mercury can be adjusted to the zero point of the gauge. The mercury gauge is connected to the air chamber by a reinforced rubber tube proof against chemical attack, having an internal diameter of roughly 3 mm. The gauge is graduated in millimeters.

c) Thermostat-controlled bath

The thermostat-controlled bath must be big enough to allow the completely assembled bomb to be immersed in it sufficiently deeply for the top of the air chamber to be at least 30 mm below the level of the fluid in the bath.

It is preferable to use oil as the bath fluid, to enable a temperature of 200 °C to be reached.

The heater must be able to stabilize the temperature at each measurement level.

d) Temperature-measuring devices

Bath

The temperature of the bath in the thermostat is measured by means of a thermometer with a centigrade scale.

Test fluid

The temperature-measuring device should preferably be a thermocouple housed in a cylindrical sleeve with a maximum external diameter of 8 mm.

e) Preliminary precaution

The tightness of each new bomb must be tested under air pressure of 7 bars. No air must escape from the bomb when it is immersed in water.

3.5.3. PROCEDURE

a) Preparation of test

The individual components of the bomb are thoroughly cleaned, rinsed and dried and then kept at a temperature of 25 ± 1 °C.

The volume of fluid required (approximately 160 ml) for the test must be kept for at least one hour at a temperature of 25 ± 1 °C.

b) Filling the fluid chamber

This chamber is to be filled in the dismantled state. The connexion to the air-chamber is then attached; during the operation, it is essential not to forget to remove the screw which closes the air vent. The lower chamber should then be further filled until the fluid just reaches the level of the air vent.

At this point the air vent should be closed by means of its screw; the air-chamber and upper closure are then set in place, the pressure offtake cock being left open, to avoid excess pressure in the bomb.

The temperature measuring device is then attached; the measuring bulb must lie 30 mm from the bottom of the lower chamber, within the test fluid.

c) Test procedure

The bomb is placed in the thermostat-controlled bath which is maintained at a temperature of 25 ± 1 °C and the mercury gauge connected. With the pressure offtake cock open, the mercury gauge must indicate precisely zero. Heating proceeds in steps of 25 °C; measurements are taken at 50, 75, 100, 125, 150, 175 and 200 °C; the rate of heating is 1 °C per minute within each step of 25 °C. At the final heating level, 200 °C, the thermostat must be capable of keeping the temperature constant within ± 1 °C for at least 5 minutes; the pressure should be read at the beginning and end of this period of 5 minutes on the mercury gauge.

d) Expression of results

Corrections must be made to the differences in level read off the mercury pressure gauge. The attached table (Annex VI-b-) gives the correction factors, expressed in millimetres of mercury, for different values of atmospheric pressure in the room where the test is carried out; these values have been prepared for the range of temperature from 20° to 200 °C. Corrections for atmospheric pressures lying between the values given in the tables are obtained in interpolation.

The calculations are based on air at + 25 °C, with a humidity index of 0.6. The figures hold good for a humidity index between 0.5 and 0.7, the maximum difference from the figures in the table being less than 0.8 mm of mercury at + 200 °C.

The corrected value must be converted into bars.

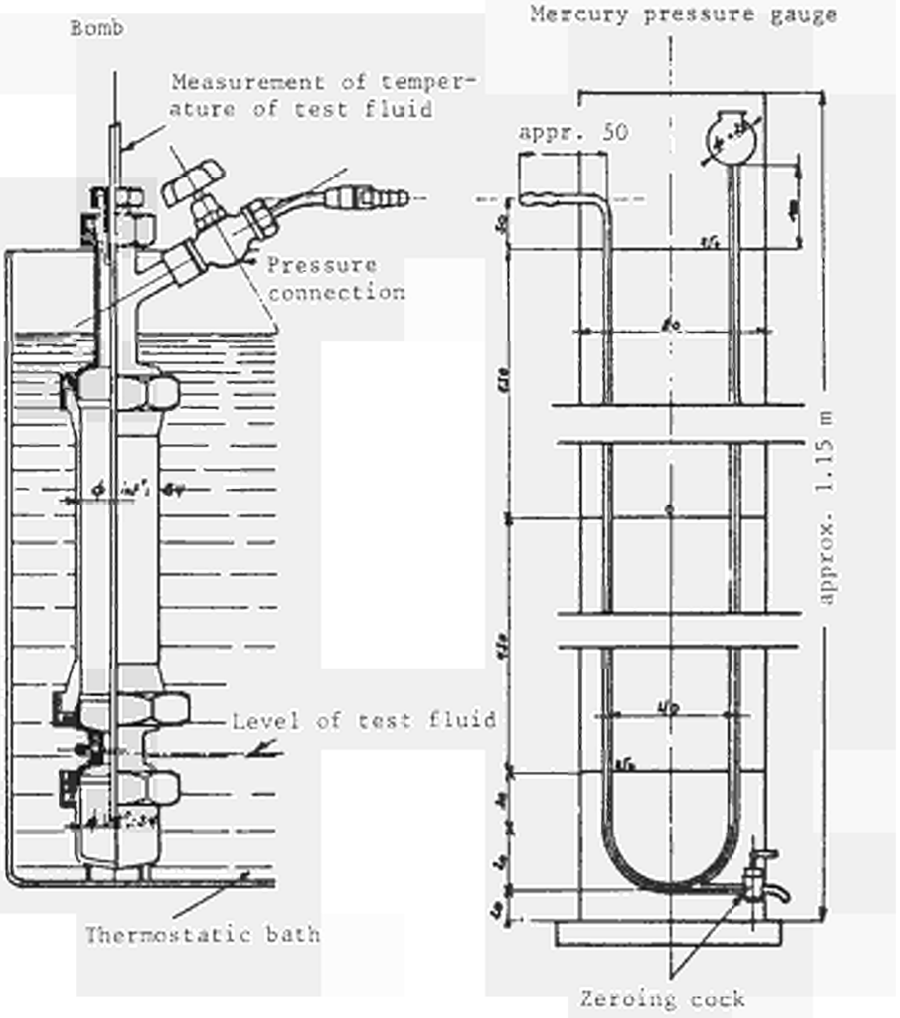
3.5.3. (d)

Table of correction in mm of mercury

Temperatures in °C	Atmospheric pressures, in mm of mercury				
	740	750	760	770	780
+ 20	+ 12.7	+ 12.9	+ 13	+ 13.2	+ 13.3
+ 25	0	0	0	0	0
+ 30	- 12.6	- 12.6	- 12.8	- 13.1	- 13.3
+ 35	- 24.5	- 24.7	- 25	- 25.3	- 25.7
+ 40	- 37.1	- 37.6	- 38.1	- 38.6	- 39.1
+ 45	- 49.8	- 50.4	- 51	- 51.8	- 52.5
+ 50	- 62.5	- 63.3	- 64.1	- 64.9	- 65.8
+ 55	- 75.1	- 75.9	- 77.2	- 78.1	- 79.2
+ 60	- 86.9	- 88	- 89.2	- 90.4	- 91.6
+ 65	- 99.9	- 101.2	- 102.5	- 103.8	- 105.2
+ 70	- 112.2	- 113.7	- 115.2	- 116.8	- 118.4
+ 75	- 124.8	- 126.5	- 128.2	- 129.9	- 131.6
+ 80	- 136.5	- 138.4	- 140.3	- 142.2	- 144.1
+ 85	- 149.3	- 151.3	- 153.3	- 155.3	- 157.3
+ 90	- 162.2	- 165.3	- 167.5	- 168.7	- 170.9
+ 95	- 174.9	- 177.2	- 179.6	- 182	- 184.4
+ 100	- 187.4	- 189.9	- 192.5	- 195	- 197.6
+ 110	- 211.2	- 214	- 216.8	- 219.6	- 222.5
+ 120	- 236.1	- 239.3	- 242.5	- 245.7	- 248.9
+ 130	- 260.4	- 263.9	- 267.5	- 271	- 274.5
+ 140	- 285.3	- 289.1	- 293	- 297	- 300.9
+ 150	- 309.8	- 314	- 318.2	- 322.4	- 326.6
+ 160	- 334.6	- 339.1	- 343.6	- 348.2	- 352.8
+ 170	- 359.4	- 364.2	- 369	- 373.8	- 378.7
+ 180	- 383.9	- 388.9	- 393.9	- 399	- 404.1
+ 190	- 408.8	- 414.3	- 419.8	- 425.3	- 430.9
+ 200	- 432.9	- 438.7	- 444.5	- 450.4	- 456.3

-(a)-

DETERMINATION OF VAPOUR PRESSURE



Note: all dimensions in mm.

DETERMINATION OF pH

3.6.1. FIELD OF APPLICATION

The method serves to determine the degree of acidity of alkalinity of aqueous fluids of forms A and C used in hydraulic systems.

3.6.2. PRINCIPLE

The pH is obtained by means of an electrometric determination of the potential difference between two electrodes immersed in the test fluid. Glass and calomel electrodes are used. The measurement is carried out at 18 °C.

3.6.3. APPARATUS AND MATERIALS

a) pH-meter
b) Glass electrode (measurement electrode, alkali-resistant)
c) Saturated calomel electrode (reference electrode)
d) Buffer solution
   I. Buffer solution pH = 7
      1. 1.1876 g $Na_2HPO_4 + 2H_2O$ in 100 ml dist. H_2O
      2. 2.1008 g $C_6H_8O_7 + H_2O$ (citric acid) in 20 ml 1N-NaOH + 80 ml dist. H_2O
         Mixture: 32.94 ml solution 1) + 7.06 ml solution 2).
   II. Buffer solution pH = 10
      1. 1.91 g $Na_2B_4O_7 + 10\ H_2O$ (borax) in 100 ml dist. H_2O
      2. 0.1 N - NaOH
         Mixture: 59.5 ml solution 1) + 40.5 ml solution 2)

3.6.4. PREPARATION

The two electrodes are connected to the measuring instrument and inserted successively in buffer solutions I and II, at a constant temperature of 18 °C. The actual readings are compared with the theoretical values. If the theoretical values (pH 7 and pH 10) are not those indicated, the readings are adjusted in accordance with the instructions for use for the instrument.

3.6.5. TEST PROCEDURE

The test fluid is warmed up to 18 °C in a glass beaker. The electrodes (washed clean with distilled water) are then immersed in the fluid and the pH read of the pH-meter.

3.6.6. EXPRESSION OF RESULTS

The results are given to the nearest 0.1 pH unit.

DETERMINATION OF SHEAR STRENGTH

3.7.1. FIELD OF APPLICATION

The method serves to determine the mechanical shear strength of fluids used in hydraulic systems. It may be applied to all types of hydraulic fluid (synthetic) except for form A fluids with a viscosity of less than 10 centistokes at 20 °C.

3.7.2. PRINCIPLE

A given volume of fluid is put through an injector a given number of times. The following characteristics are recorded before and after the test, to establish any variations: viscosities, softening point, pH, neutralization number for fluids of forms B and D, water content for forms A, B and C, but not D.

3.7.3. APPARATUS AND SOLVENTS

a) The apparatus conforms to the accompanying diagram. The injector is a Bosch type KD 43 SA/53/13, with a DN 0 SD 211 type spray nozzle. This nozzle must be set to 100 bars *(1)*.

b) Glass containers (3 and 3a) with non-tight lids.

c) Three-way cocks and tubing to connect container (3a) with the injection pump.

d) Timer.

e) 400 ml beakers.

f) Solvents to clean the glass parts: crystallizable benzene or motor spirit for petroleum oils and form B fluids, water for forms A and C fluids, trichlor-ethylene or monochlorobenzene for form D fluids, and pure ethyl alcohol for drying after washing with water.

3.7.4. PREPARATIONS FOR THE TEST

a) 250 ml of the fluid to be tested are poured into container 3, the three-way cock being in position 6.

b) Screw 13 is unscrewed to allow the air to escape from pump 14; as soon as the fluid starts to flow steadily, screw 13 is tightened.

---

(1) Tests with jet nozzles set to 175 bars are being conducted by way of research.

3.7.5. TEST PROCEDURE

a) Start the motor.

b) Start the timer as soon as the fluid begins to flow through tube 2.

c) Ensure that pressure in the delivery circuit is between 100 and 110 bars, using pressure gauge 11, by opening cock 12. This cock must be shut after the check.

d) After 30 minutes' running time, corresponding to at least 50 cycles, stop the motor. Turn the three-way cock to position 5 and collect the fluid in a clean Pyrex beaker (normally the temperature of the fluid at this stage is above 55 °C). Drain the circuit completely by re-starting the motor for a few moments until the flow from tube 2 has stopped.

Note

There should not be much flow from tube 8; an appreciable return of fluid via tube 8 may occur when testing viscous fluids (viscosity 120 centistokes at + 50 °C).

3.7.6. CLEANING

a) Cleaning the circuit: since even partial dismantling is out of the question, cleaning is carried out before each test by circulating 100 to 150 ml of the test fluid for about 10 minutes. If the subsequent tests relate to fluids of the same form, one circuit is enough. If not, two cycles at least will be necessary, the flushing fluid being disposed of each time.

b) Cleaning of glassware and tubing: this is done with solvents and products appropriate to the fluids tested (see para. 3.f).

3.7.7. EXPRESSION OF RESULTS

a) The following tests must be performed on the fluid collected after the shear test:

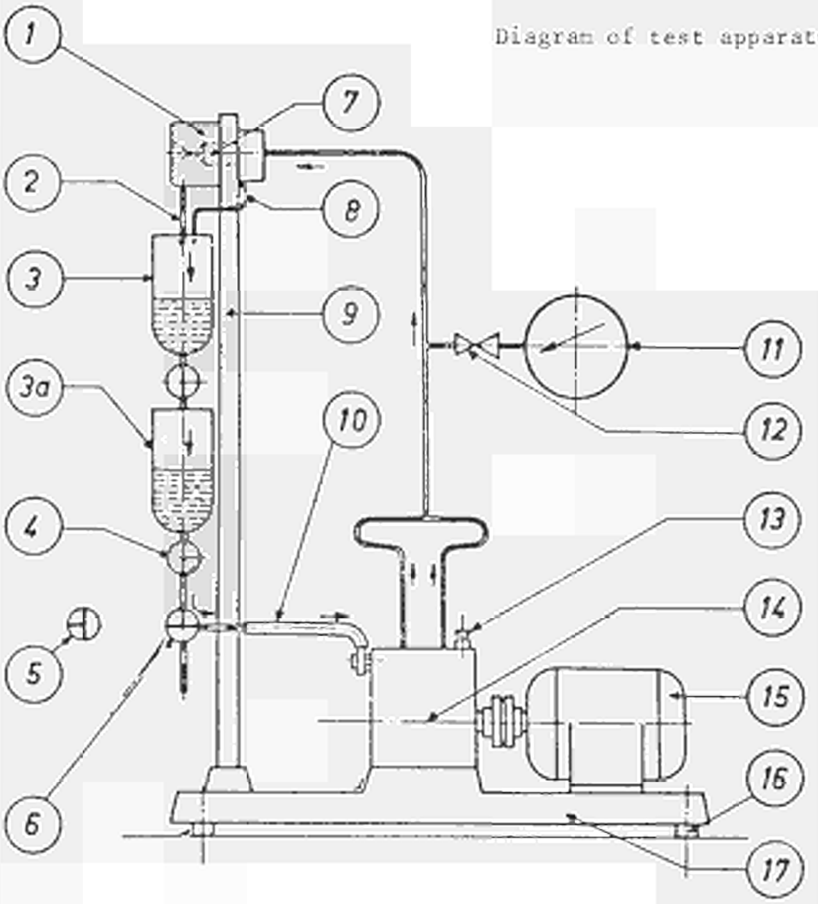
Viscosities : at - 20, 0, 20 and 50 °C for forms C and D; at 20 and 50 °C for forms A and B.
Softening points: for all groups
pH : for forms A and C; and neutralization number for forms B and D.
Water content : for forms A, B and C.

b) The results of these tests are compared with the same determinations on the fluids before shear.

-(a)-

SHEAR STRENGTH TEST

Diagram of test apparatus



Legend:

1	Atomizing chamber
2	Outlet for atomized fluid
3	600 ml glass container, with cover
3a	-ditto-
4	Stop cock
5	3-way cock at the end of test
6	3-way cock during test
7	Injector, set to 100 bars
8	Excess fluid return
9	Support
10	Connection to pump intake
11	Pressure gauge, 0/250 bars
12	Pressure gauge tap
13	Valve to evacuate air from pump
14	Two-cylinder injection pump e.g. Bosch PE 2 B/100 or PE 2 A900 300/3 S 226
15	Electric motor, 1.1 kW, 920 r.p.m.
16	Rubber buffers
17	Base-plate
⬅	Direction of flow of test fluid

DETERMINATION OF ANTI-CORROSIVE POWER

3.8.1. FIELD OF APPLICATION

The method serves to determine the anti-corrosive power of fluids used in hydraulic systems. The method can be applied to all forms of hydraulic fluid (mineral oils, synthetic fluids and emulsions).

3.8.2. PRINCIPLE

Samples of various materials are partly immersed in the hydraulic fluid under test for a fixed period of time. The change in weight of the samples and changes in the surface of the material, as well as changes in the colour of the test solution, are noted.

3.8.3. APPARATUS AND CHEMICALS

a) Glass beakers, 500 ml capacity (tall type);

b) Watch-glasses, to cover the beakers, with an opening in the middle for a lifting look.

c) Glass hooks, from which the sample sheets can be freely suspended in the beaker;

d) Thermostatically controlled heating bath which maintains the test fluid in the beaker at a temperature of 35 ± 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 can be substituted for the thermostatically-controlled bath.

e) Emery paper No. 0;

f) Cotton wool;

g) Regular-grade motor spirit, boiling range 65 to 95 °C;

h) Pure benzene;

i) 96% ethyl alcohol;

j) Sample sheets 100 mm x 20 mm wide x 1 mm thick, with a 4 mm diameter hole at the upper edge of one short side, so that the plates can be hung on a glass hook;

The following materials are used for the test:

Steel with the following composition:

C 0.40 to 0.50 %
Mn 0.5 to 0.8 %
Si 0.35 % max.
S and P = 0.035 % max.

Electrolyt copper,
Pure zinc,
Pure aluminium (at least 99.5 %),
Pure cadmium
Brass (70 % Cu and 30 % Zn)

Note: The same tests can be applied to all other metals and alloys used in the manufacture of mining equipment.

k) Analytical balance with a sensitivity of 0.0002 g;

l) Water for preparing emulsions for THIA fluids of hardness value 40 ± 5. It is obtained by adding calcium chloride to distilled water.

3.8.4. PREPARATION

The sample sheets should be prepared with suitable emery paper, ending up with emery paper No. 0 to give the best possible surface finish. The sheets are then held with forceps and cleaned with dry cotton wool, followed by cotton wool soaked in regular-grade motor spirit. The traces of cotton wool are washed off with pure motor spirit and then with benzene. As soon as the last traces of solvent adhering to the sample sheets have evaporated, the sheets are weighed on the analytical balance. They must then be used immediately for the corrosion tests.

At least 11 beakers are filled for the entire test series each with 250 ml of fluid. The glass beakers are then placed in the thermostatically-controlled heating bath, whose temperature is set to give a constant temperature of 35 ± 1 °C in the test fluid.

Six of the beakers receive one each of the following sample sheets, prepared in advance: steel, copper, zinc, aluminium, cadmium and brass, suspended in such a way that approximately 60 mm of the sheet is immersed in the test fluid. To test the behaviour of fluid in the presence of two metals, the following metals are immersed in four beakers:

Steel - pure cadmium;
Copper - zinc;
Aluminium - zinc;
Steel - aluminium.

The two sample sheets should hang approximately 1 mm apart;

The eleventh beaker contains 250 ml of the test fluid, in which no sample has been immersed, and serves to check the precipitation of the fluid itself during the period of observation.

In every case, the beakers must be covered with watch-glasses to limit evaporation.

3.8.5. PROCEDURE

Care must be taken during the test to maintain the temperature in the heating bath constant. The complete test must run for a minimum of 28 days.

3.8.6. ASSESSMENT

At the end of the test, the specialists must describe the changes in the surface of the test sheets: 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 solution, and of deposits in the fluid. After visual examination, the testers must wash the test pieces in ethyl alcohol in a wash bottle in the case of fluids of forms A, B and C, or in benzene for fluids of form D (do not rub), and dry them by means of oil-free compressed air. The test pieces thus prepared are weighed on the analytical balance.

The difference in weight is expressed in mg per test piece, and the specialists must record both increases (+) and losses (-) in weight.

3.8.7. PROBABLE FUTURE CHANGES TO THE TEST

It is likely that in the future that this method will be replaced by Cotop RP. 484 where the working temperature will be increased from 35 to 50 °C ± 1, and the duration of the test reduced from 28 to 15 days. For the investigation of fluids of forms A, B and C, the appartus will consequently have to be modified to maintain saturation in water vapour above the fluids.

The tendency with pairs of metals is to connect them electrically.

DETERMINATION OF THE RESISTANCE TO AGEING OF FLUIDS CONTAINING NO WATER

NOTE

This method uses the apparatus and the ageing conditions described in standards ASTM D 943-54 and DIN 51587. The method used to study the aged sample has been modified to cover the field of application of non-aqueous fluids.

3.9.1. FIELD OF APPLICATION

This method serves to determine the ageing characteristics of water-free fire-resistant fluids used in hydraulic systems.

3.9.2. PRINCIPLE

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

3.9.3. APPARATUS AND CHEMICALS

a) Oxidizing call (fig. 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 oxidizing cells, 350 mm high, are completedly surrounded by the fluid in the bath.

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

d) A device for winding the catalyzing coils (fig. 2).

e) Thermometer, range 75 to 125 °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 ($\gamma = 1.18$).

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

i) Regular-grade motor spirit, boiling range 65 to 95 °C.

j) Nitric acid, concentrated industrial grade ($\gamma = 1.42$).

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.

3.9.4. 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 regular-grade motor spirit; the surface of the wire is then 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 cloves 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 (fig. 2). The free ends of the steel wire and copper wire are then also attached by six turns; the coils are removed from the spindle and then adjusted to a length of 230 mm ± 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 (fig. 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 motor spirit, 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).

3.9.5. ANALYSIS 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, reaches the prescribed temperature of 95 ± 0.2 °C.

b) The catalyzing coils are then slid over the admission opening of the oxygen inlet pipe, and coil and pipe are centred. A quantity of 300 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 litres/hour, 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.

3.9.6. ANALYTICAL DETERMINATION 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

Some 5 g of the sample are dissolved in a flask in ten times this quantity of pure benzene. This solution is filtered under slight vacuum on a membrane filter which has previously been weighed (type: average pore diameter 0.4 microns, 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 until the filtrate is completely clear. Ater it has been allowed to stand for an hour, the benzene is completely evaporated from the filter. The membrane filter is then placed to dry for half an hour in a desiccator and weighed weight "B". It is advisable to carry out a blank filtering test with pure benzene, since the filter itself may undergo a weight loss of 1 to 2 mg when treated with benzene. The percentage insoluble in benzene 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.

-(a)-

METHOD FOR DETERMINING THE RESISTANCE TO AGEING OF FLUIDS CONTAINING NO WATER

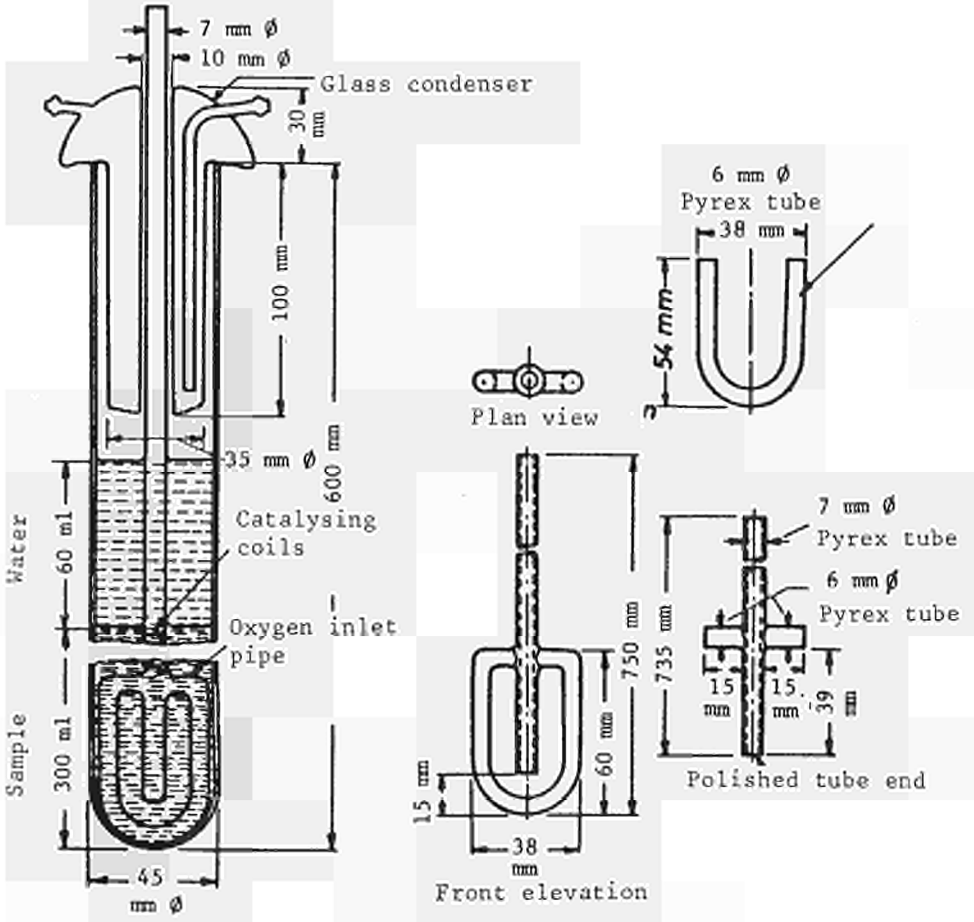


Fig. 1: OXIDIZING CELL

-(b)-

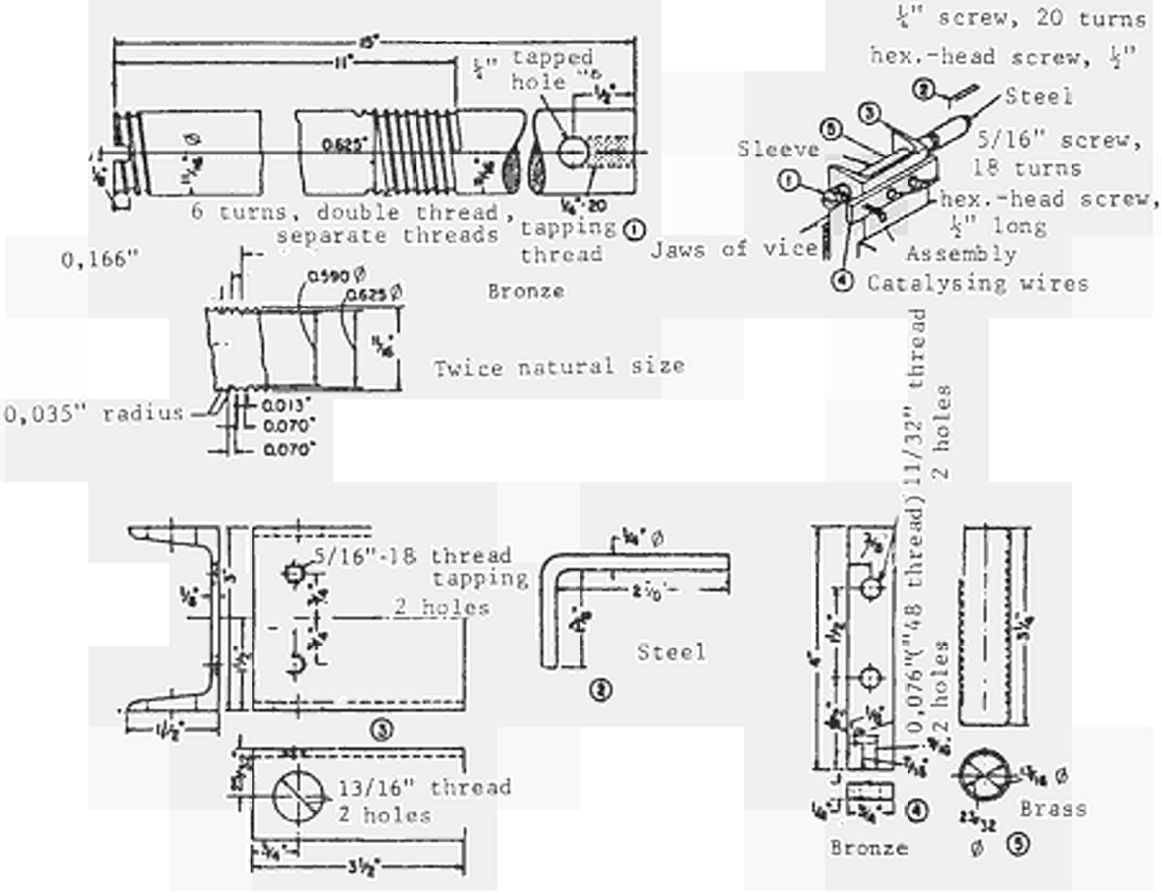


Fig. 2: SPINDLE FOR WINDING CATALYSING COILS

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

DETERMINATION OF THE RESISTANCE TO AGEING OF WATER-CONTAINING FLUIDS

NOTES

The method uses the apparatus and applies the ageing conditions laid down in standards ASTM D 943-54 and DIN 51587. The apparatus, the determination of ageing and the examination of the aged samples have been modified to suit the application to water-containing fluids.

3.10.1. FIELD OF APPLICATION

This method serves to determine the ageing characteristics of water-containing fluids used in hydraulic systems.

3.10.2. PRINCIPLE

The test fluid is subjected to a temperature of 95 °C in the presence of oxygen and after the addition of iron and copper as catalysts.

3.10.3. APPARATUS AND CHEMICALS

a) Oxidizing cell (fig. 1)
The cooling jacket of the oxidizing cell differs from that described in PART III 3.9. in that its envelope is not 100 mm but 200 mm long (fig. 1).

b) A thermostatically-controlled heating bath which maintains the fluid in the oxidizing cell at a temperature of 95 ± 0.2 °C, provided with a stirring device to ensure that the temperature of the bath is uniform throughout. The dimensions of the bath must be such that the required number of oxidizing cells can be completely surrounded by the fluid in the bath up to a height to 350 mm.

c) Flow-meter with minimum capacity of 3 litres/hour and an accuracy of ± 0.1 litre/hour.

d) A device for winding the catalyzing coils (fig. 2).

e) Thermometer graduated from 75 to 125 °C.

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

g) Hydrochloric acid, concentrated technical grade (γ = 1.18).

h) Hydrofluoric acid, concentrated technical grade (approx. 50 %).

i) Regular-grade motor spirit, boiling range 65 to 95 °C.

j) Sulphuric acid, concentrated technical grade (γ = 1.42).

k) Acetone.

l) Sulphochromic acid.

m) Oxygen in a container with a pressure regulator, giving a constant gas flow. It is best to use an oxygen bottle with a two-step regulator.

3.10.4. PREPARATION OF THE APPARATUS

a) Cleaning the catalyst

On the day fixed for the start of the test, 3 m lenghts of steel wire and copper wire are cleaned with cotton wool soaked in regular-grade motor spirit; the surface of the wire is then 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 wool or cotton gloves to avoid contact with the skin.

b) Preparation of the coils of wire

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

c) Cleaning the oxidizing cell

The piping and reaction vessels are cleaned by rinsing with acetone, running water, sulphochromic acid and again running 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 pipe is fitted, together with the cooling jacket, and the whole assembly is left in this condition for at least 24 hours before beginning the test. Shortly before the test begins, the vessels are emptied and dried, and the outer walls of the 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. This cleaning process is repeated with acetone replacing the motor spirit, after which the tubes are filled with an oxidizing mixture composed of three parts of HCl and one part HNO_3, which is left in the apparatus for at least 24 hours at room temperature. The apparatus is then rinsed in running water to remove all traces of the acids, and the organic reaction products are removed by means of acetone.

If deposits remain inside the vessel, it should be rinsed out with a mixture of equal parts of hydrofluoric acid and hydrochloric acid. This mixture of acids should be left in the vessel until the deposits have disappeared or dissolved, and the acid is then rinsed away with large quantities of tap water. The final cleaning is then carried out as in paragraph c).

3.10.5. DETERMINATION OF AGEING

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

b) The catalyzing coils are then slid over the inlet of the oxygen pipe and the coils and pipe are centred. A quantity of 360 ml of test fluid is poured on to the coil until it is completely wetted. 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 pipe and connected to the cooling water supply (the temperature of the cooling water flowing off must not exceed 35 °C during the test).

c) The oxygen pipe is connected to the oxygen bottle via a flow-meter, the quantity of gas adjusted to 3 ± 0.5 litres/hour. The time is then recorded. It is necessary to check the oxygen flow at least twice a day, to respect the prescribed tolerance.

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

e) By constantly topping up with distilled water the level of fluid in the oxidizing cell is maintained in spite of evaporation losses.

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 test samples are removed from the cell, the total volume reduction is noted and the volume of test liquid may be maintained to this mark, if required, by adding fresh material.

3.10.6. ANALYTICAL DETERMINATION OF AGEING

a) During the ageing process, a sample of about 10 ml is taken roughly every 2 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 part (approx. 5 g) being used to determine the neutralization number (form B fluids) and the pH (forms A and C), the remainder to determine the insoluble substances eliminated (deposition of sludge). The determination of these waste substances is restricted to form C fluids and is not applicable to emulsions.

c) Determination of the neutralization number and pH

If it is not possible to determine the neutralization number by the normal commercial method using alkali blue as a colour indicator, it must be determined potentiometrically. In this case, either the total acid number (TAN) or the total base number (TBN) must be determined, by the method ASTM D 664-54. The pH is measured by means of a compound alkaline-resistant glass electrode.

d) Determination of the content of insoluble matter

Approximately 5 g of the fluid are diluted in 5 ml distilled water in a beaker, then filtered under low vacuum using a previously-weighed membrane filter (type: average pore diameter 0.4 microns, 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 distilled water until the filtrate is completely clear.

The membrane filter is then placed in a desiccator for one hour, after which it is weighed weight "B". It is advisable to carry out a blank filtration test with distilled water, and to determine the variation in weight. The percentage insoluble in water is calculated by the formula $\rho = (B-A) \times 20$.

e) The duration of the test must be at least 200 hours of ageing. Once the ageing is finished other tests of many different kinds can be carried out if desired.

-(a)-

METHOD FOR DETERMINING THE RESISTANCE TO AGEING OF WATER-CONTAINING FLUIDS

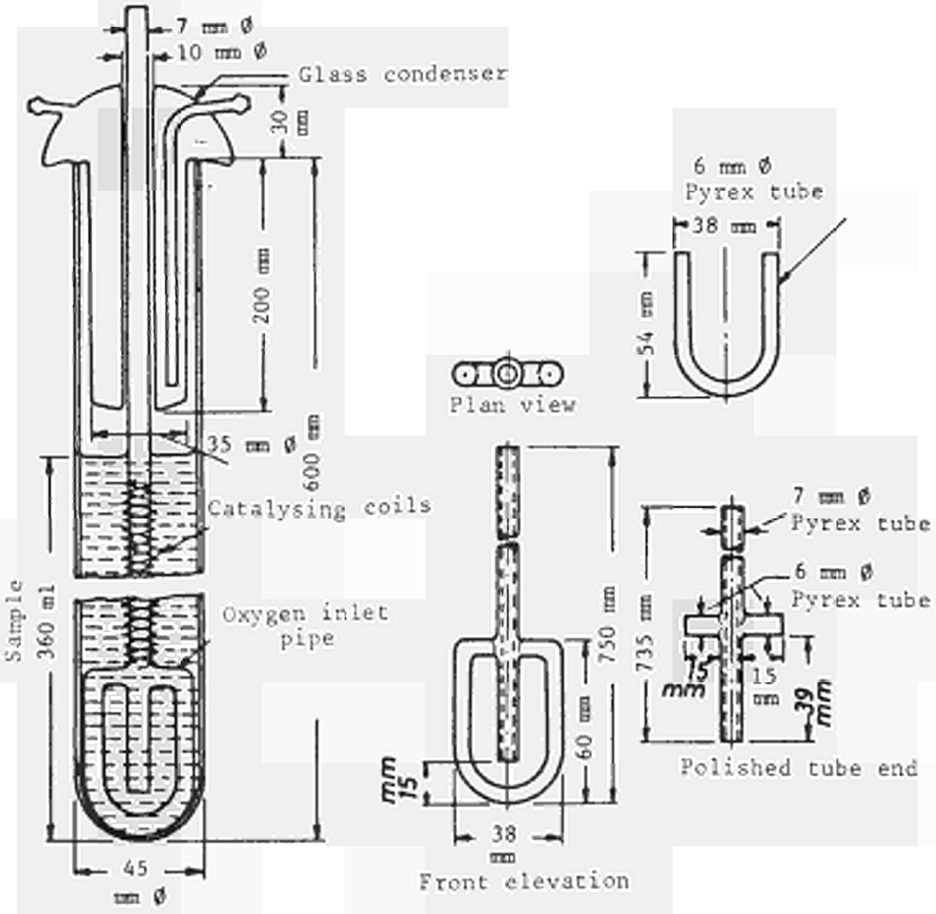


Fig. 1: OXIDIZING CELL

-(b)-

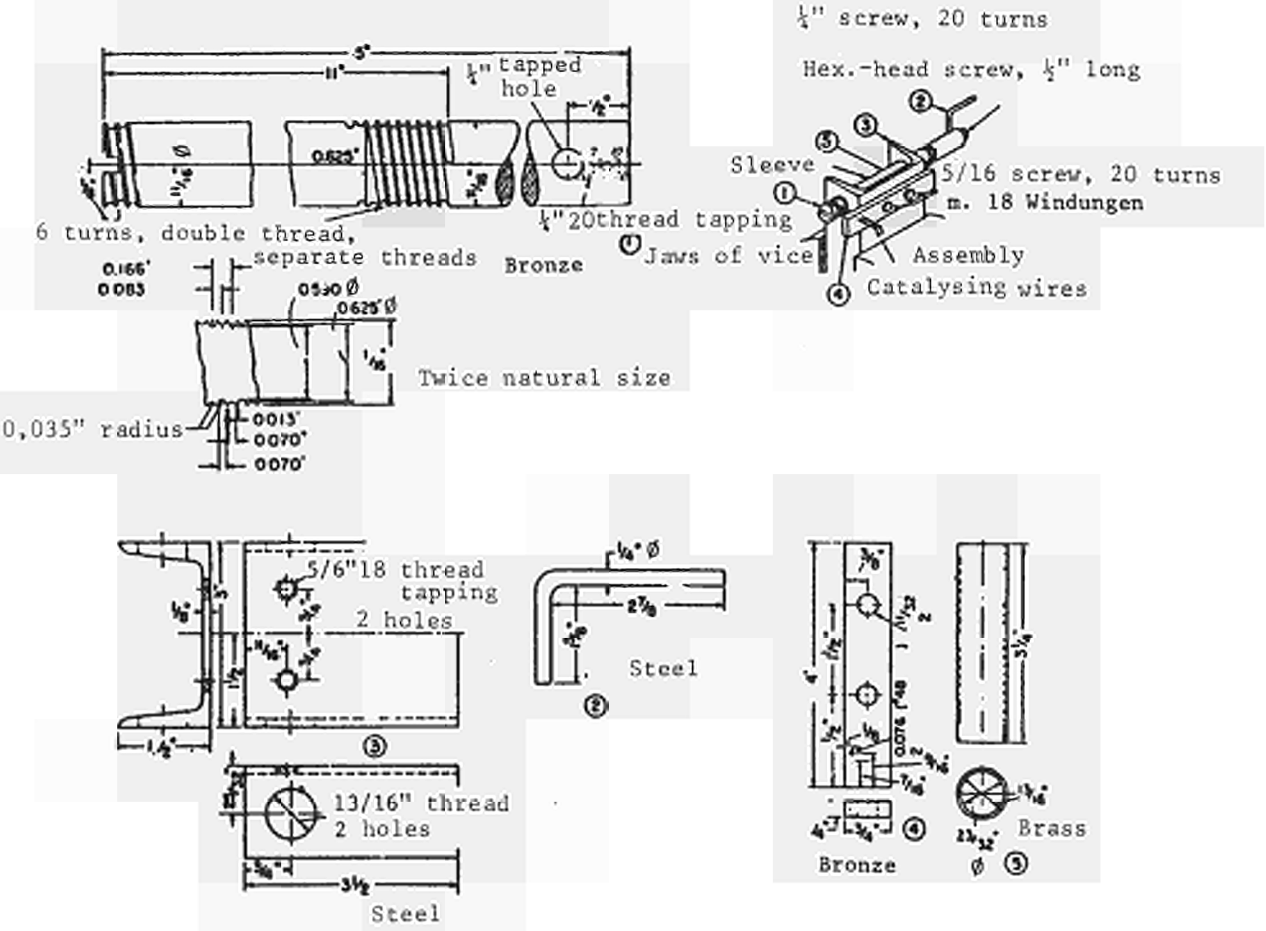


Fig. 2: SPINDLE FOR WINDING CATALYSING COILS

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

METHOD FOR DETERMINING THE BEHAVIOUR OF PACKINGS AND SEALS

3.11.1. APPLICATION

This method is used to determine the behaviour of sealing materials in contact with fire-resistant fluids of forms A, B, C and D at a test temperature of 60 °C. Form D fluids are also tested at 150 °C.

3.11.2. PRINCIPLE

Sample pieces of the sealing material are suspended in the test fluid under clearly-defined conditions. The volume and Shore hardness are measured before and after the test.

3.11.3. APPARATUS

a) Deep glass beakers, 250 ml capacity;

b) Erlenmeyer flasks, 500 ml capacity;

c) Bowl-type cooler (length 250 mm) or mushroom-type;

d) A thermostatically-controlled bath for a bath temperature of 60 ± 1 °C and if appropriate 150 °C. The dimensions of the bath must be such that at least 2 beakers or Erlenmeyer flasks can be surrounded to a heigth of 70 mm by the bath fluid;

e) Balance;

f) Shore-hardness apparatus with scale divisions of 5 hardness units, calibrated from 0 to 100 (0 being the lowest hardness and 100 the highest). The indentor body consists of a truncated cone. The apparatus must conform to the requirements of ASTM D 676-55 T.

3.11.4. TEST PROCEDURE

Circular sample pieces of the sealing material, 40 mm in diameter and 6 mm thick, are cut out. Two samples for each material quality are made for each test.

The samples are cleaned by dabbing with a filter paper dipped in 96 % ethyl alcohol. The sample volume is determined by weighing them first in free air and then in water. The samples are then dried and heated to 20 ± 2 °C before measurement of the Shore hardness.

This is measured at three different points on each sample. The measuring points must be 5 mm apart and 13 mm away from the sample edges. The Shore hardness apparatus is brought down without jerking on to the indentor with the bearing surface surrounding the tip of the indentor until it impinges firmly on the sample. The hardness value is read off the scale after three seconds contact between the bearing surface of the Shore hardness apparatus and the sample.

For form D fire-resistant fluids, a sufficient quantity of fluid is poured into the beaker to give a ratio of about 1:15 between the sample volume and the fluid volume (5 g of sample require some 100 ml of fluid).

For forms A, B and C fire-resistant fluids, the beaker is replaced by an Erlenmeyer flask, as otherwise too much fluid would evaporate during the period of chemical attack. Once the sample piece has been placed in the fluid, a water-cooled bowl-type cooler is placed over the Erlenmeyer flask.

Two or three glass rods arranged at the bottom of the receptacle prevent the seals from touching it.

If required the Erlenmeyer flask and its cooler can be used instead of the glass beaker for form D fluids.

The beaker or the Erlenmeyer flask is kept for 21 days at a temperature of 60 ± 1 °C or at 150 °C as the case may be. The sample pieces are then cleaned by dabbing with a filter paper dipped in 96° ethyl alcohol and dried between two layers of filter paper. Five minutes after drying, the volume and Shore hardness of each sample are determined in the manner described above.

3.11.5. EXPRESSION OF RESULTS

The proportional increase in volume, which is taken as the swelling index, can be established by the following formula:

$$V = \frac{V_2 - V_1}{V_1} \times 100$$

where:

V = proportional increase in volume of the sample;

V_1 = volume of the sample before immersion;

V_2 = volume of the sample after immersion.

If the different values deviate by more that 1 % (in absolute figures) from the mean, the test must be repeated.

The Shore hardness measurements are expressed in hardness units. The change in Shore hardness is calculated by the following formula:

$$H = H_2 - H_1$$

where:

H = difference in Shore hardness before and after immersion

H_1 = Shore hardness before immersion

H_2 = Shore hardness after immersion.

The different values must not deviate from the mean by more than two Shore hardness units.

3.11.6. CHOICE OF PACKINGS AND SEALS

In order to determine the behaviour of fluids of form D, a fluorinated elastomer seal should be used; for forms A, B and C fluids, only the grades of seals and packings specified by the hydraulic equipment manufacturers may be used. However, the expressed wish is to limit the number of grades to the minimum possible.

A. ASSESSMENT OF EXTREME PRESSURE CHARACTERISTICS

(4-ball machine)

3.12.1. AIM

With the 4-ball machine the extreme pressure characteristics of fluids of forms A, B and C, and especially of form D may be assessed.

3.12.2. PRINCIPLE

A sample of fluid is subjected to a series of tests of fixed duration, under increasing loads, in the 4-ball machine, until binding point is reached; the mean Hertz load, the limit binding load and the wear after a one-hour test under constant load are determined.

3.12.3. APPARATUS

a) A 1460 rev/min 4-ball machine, Royal Dutch-Shell model. The attached figure shows a section through this machine.

b) Binocular magnifier for measuring the test indentations on the samples, with a minimum magnification of 15 to 20 X, fitted with an ocular micrometer. The scale of the ocular micrometer is calibrated by means of a suitably-set micrometer scale. It is recommended to use a special object-slide with a hemispherical bowl 13 mm in diameter.

c) Test piece: steel bearing balls, 12.7 mm diameter, made of 100 Cr6 (W3), hardness 64 ± 2 HRC, made by SKF, Schweinfurt. (see footnote)

d) Chronometer reading in tenths of a second.

3.12.4. CLEANING AGENTS

a) The solvents used to clean the sample pieces must not possess extreme pressure characteristics, which excludes the use of such substances as carbon tetrachloride, for example. Either a completely-evaporating light petroleum or heptane distillate (petroleum ether) for else crystallizable benzene (C_6H_6) is to be used.

b) The solvents for cleaning the bowl holding the balls, the centring bicone and the mandrel vary according to the nature of the fluid under test.

There are no restrictions for these solvents as to extreme pressure characteristics.

With emulsions of forms A and B, crystallizable benzene or motor spirit should be used first, followed by 95 % ethyl or methyl alcohol.

With form C products careful rinsing with pure water is followed by drying with 95% pure ethyl or methyl alcohol.

With form D products, a suitable solvent must be found, e.g. monochlorobenzene, trichloroethylene, carbon disulphide, 95 % ethyl alcohol, or crystallizable benzene.

Note : The test apparatus uses a bearing ball of diametre 12.7 mm ± 0.63 mm; hardness 64 ± 2 HRC in chrome steel of the following composition: carbon 0.90 to 1.10 %; chrome 1.30 to 1.60 %; manganese 0.25 to 0.45 %; silicon 0.20 to 0.35 %; phosphorus less than 0.025 % and sulphur less than 0.025 %. This complies the French Specification 100 C.6 and the German Specification 100 Cr6 (W3).

3.12.5. PREPARATION OF TEST

a) Cleaning the balls

A new set of four balls is needed for each test. They are cleaned with one of the solvents listed in paragraph 4a) above.

b) Cleaning the apparatus

The ball holder bowl, the centring bicone and the mandrel which receives the rotating ball must be cleaned with one of the solvents listed in 4b) above, according to the type of fluid under test, and then dried before assembly of the apparatus.

During successive tests on one fluid, it is sufficient to empty the ball race without cleaning it with solvents; nevertheless it is recommended to wipe the various components with a clean, dry rag before the next test. Complete cleaning is essential on completion of each series of tests on a given fluid.

c) Assembly

A new ball is pressed by hand into the mandrel, which is then fitted in the mandrel holder and locked.

Three similar balls are placed in the ball holder bowl, and centred by fitting the bicone. The assembly is locked by tightening the bolt screwed on the outer thread of the ball holder bowl.

The test fluid is poured into the bowl to cover the top surfaces of the balls to a depth of some 3 mm.

The assembly is then positioned beneath the rotating ball and centred there by means of the support plate. The lever carrying the weights is released, and the stationary balls are pressed against the rotating ball from below, under the effect of the test load.

d) Changing the test fluid

With low loads (up to 40 kg) it is not absolutely necessary to change the test fluid for each successive loading in the case of form D fluids.

In the case of forms A and B emulsions, and with fluids of form C, it is, however, preferable to make up the total volume of test fluid for each new loading, because appreciable evaporation may occur.

Above 40 kg, all the test fluid must be changed after each test, care being taken to cool the ball holder bowl to laboratory temperature.

3.12.6. TEST PROCEDURE

a) Progressive load test

For each load selected, a 10 second test is carried out.

The test begins at a load of 40 kg. The load is then increased according to the progression set out in the attached table.

For the "binding" load, binding is obtained within about two seconds. A control test is carried out under the same load which will be taken as the limit binding load.

If binding occurs at 320 kg or less, new tests at loads just below 40 kg must be carried out so that 20 in all are performed, not counting the tests corresponding to the binding load.

b) Constant load test

A one hour test is carried out with a 40 kg load.

c) Measuring the indentations

The wear indentations formed on each ball are measured in two directions perpendicular to each other, one being parallel to the scratches on the indentation surface. The wear diameter is taken to be the arithmetic mean of six measurements of the indentations on three balls.

3.12.7. EXPRESSION OF RESULTS

a) Corrected load

For each of the test loads - excluding the binding load - the corrected load is obtained by dividing the factor K - the product of the test load and the Hartz diameter at this load - by the mean indentation diameter. The attached table gives the values of K for the various possible test loads.

b) Corrected mean load Pc

The corrected loads for the 19 to 20 loads of 320 kg or less are added together (total A). The mean corrected load is then calculated for loads higher than 320 kg (mean B).

Total A is added to mean B and the sum of the two is divided by 20

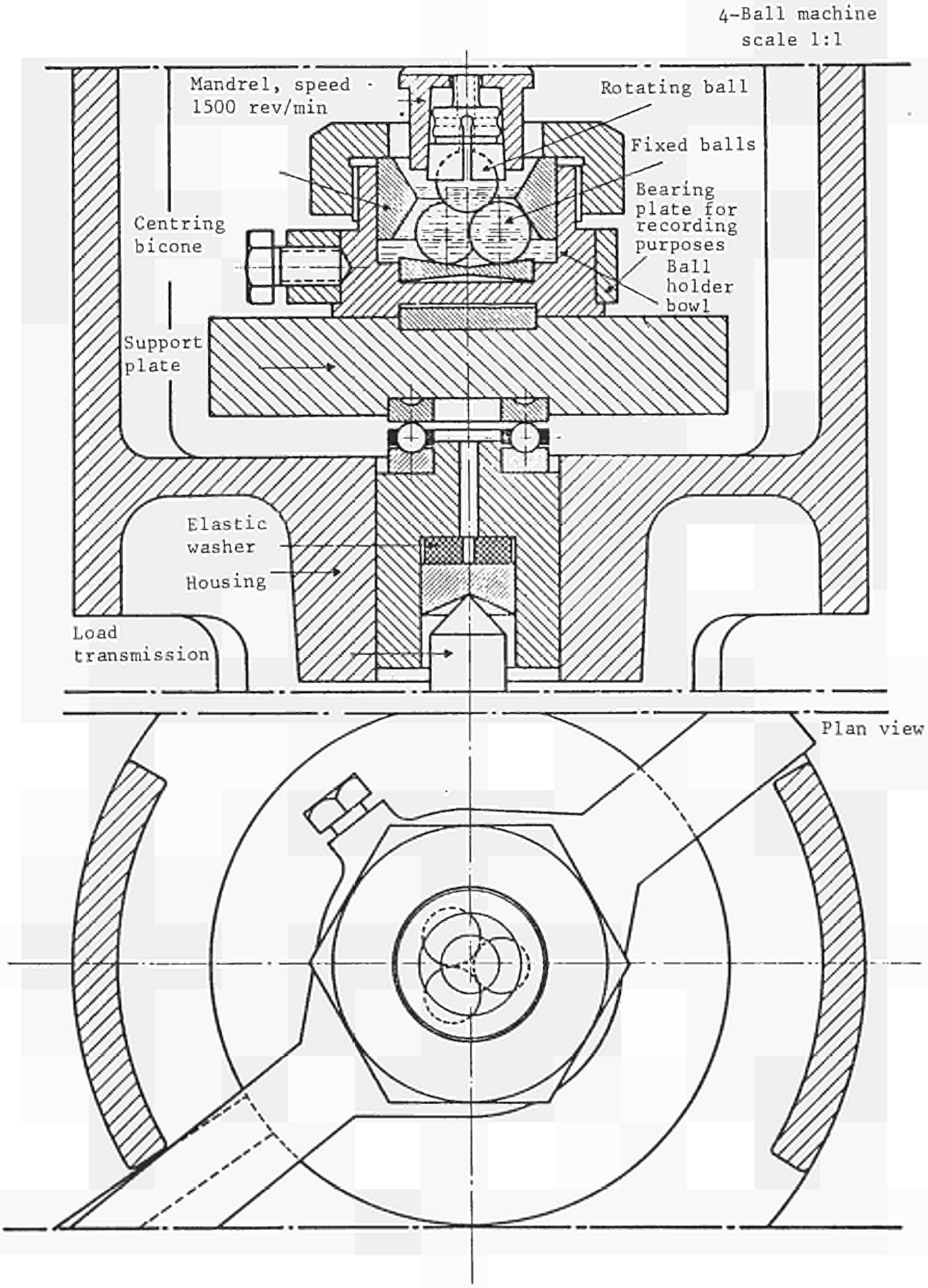
$$Pc = \frac{A + B}{20}$$

c) Expression of results

(1) Corrected mean load Pc
(2) Limit binding load
(3) Mean indentation diameter for the 40 kg load for one hour.

-(a)-

ASSESSMENT OF EXTREME PRESSURE CHARACTERISTICS



4 BALL MACHINE

CORRECTED MEAN LOADS

Applied load P (kg)	Diam. of indentation						Mean diam d (mm)	Factor K = P.dH	Corrected load Pc = $\frac{K}{d}$
	Ball No 1		Ball No 2		Ball No 3				
	1	2	3	4	5	6			
6								0.952	
7								1.169	
8								1.397	
9								1.634	
10								1.881	
12								2.398	
14								2.946	
16								3.520	
18								4.118	
20								4.739	
22								5.381	
25								6.382	
28								7.423	
32								8.869	
36								10.38	
Base - 40								11.94	
45								13.97	
50								16.08	
56								18.70	
63								21.88	
70								25.19	
80								30.09	
90								35.21	
100								40.52	
110								46.01	
125								54.56	
140								63.46	
160								75.83	
180								88.72	
200								102.11	
225								119.47	
250								137.49	
280								159.92	
320								191.08	
Total A									
360								223.57	
400								257.29	
450								301.04	
500								346.45	
560								402.96	
620								461.53	
700								542.60	
800								648.34	
Corrected mean load = $\frac{A + B}{20}$ = ____________							Total		________
							Mean B		________

B. ASSESSMENT OF ANTI-WEAR CHARACTERISTICS

(VICKERS pump test bench)

3.13.1. AIM

The purpose of this method is to assess the anti-wear proporties of fire-resistant hydraulic fluids.

3.13.2. PRINCIPLE

Under the mechanical and dynamic conditions encountered in, for example, the operation of a hydraulic pump, the difference in weight due to wear of pump components which slide against each other is determined. The weight difference as a function of operating time is taken as a measure of the wear.

3.13.3. DESCRIPTION OF THE TEST BENCH AND APPARATUS

The following items are required for assembling the test bench:

a) Vickers hydraulic vane-type pump, type 150 C - 10 (or V 104 C - 10) driven by an electric motor of over 11 kW power, nominal rotational speed 1460 rev/min.

b) A pressure regulating valve.

c) A cooler connected to a supply of cooling water.

d) A filter on the return circuit with 25 µ diameter pores.

e) A 0 to 45 litre/min flowmeter

f) Tank of minimum capacity 60 litres, made of material resistant to all the types of fluid tested (e.g. stainless steel).

g) Piping connecting the tank to the pump (1" diameter).

h) Piping connecting the pump to the tank (3/4" diameter).

i) A pressure gauge, a thermometer and an isolating cock.

These various components, all of standard commercial design, are assembled as shown in figure 1. Before assembly all the internal coatings should be completely removed and the joints replaced by Viton joints. The filters should be examined in advance to check whether the substances they are made of are resistant to the fluids tested. In assembly, avoid small-radius bends in the piping connecting the outlet of the tank and the inlet of the pump. The pump intake must be between 500 and 700 mm below the surface of the fluid. The isolating cock should preferably be a ball cock.

3.13.4. INSTRUMENTATION

The duration of the test is measured by means of a pulse-type timer with a pulse interval of less than 1 min. A pre-selection system to stop the motor at the end of a predetermined period should preferably be used. Other safety devices can be used to permit working without supervision of the bench, e.g.:

Pressurestat which stops the motor in the event of a presssure failure (as in the case of a broken shaft or abnormal wear);

Electrical device for automatically reconnecting the motor should the voltage drop momentarily or should the current fail for less than 1 min.

For surveillance of all factors during the tests and to allow continuous use of the test bench without constant supervision, it is necessary to use thermostats and pressure gauges with minimum/maximum controllers and fluid level control. The use of a multi-colour recorder compensator in addition to indicating instruments is recommended.

The following measuring points are specified :

a) vacuum at suction side of pump
b) pump operating pressure
c) temperature inside the tank.

The effectiveness of the temperature control can be verified by measuring the temperature of the fluid before and after the cooler.

3.13.5. BALANCE

The precision balance required for determining the variation in weight of the pump components subject to wear must have a range of at least 200 g and an accuracy of o.1 mg.

3.13.6. CLEANING AGENTS

The solvents for cleaning the test bench and the components subjected to wear must not possess extreme pressure characteristics, so that products such as carbon tetrachloride cannot be used. For fluids of form D a light petroleum distillate which evaporates completely is used. For the first cleaning operation, butyl glycol may be used to examine aqueous fluids. For the final cleaning operation, ethyl or methyl alcohol (minimum 95 %) is used.

3.13.7. PREPARATION OF TEST

a) Cleaning the parts subject to wear

An essential prerequisite for the precise determination of the weight of the parts subject to wear is that they must be cleaned before weighing. Various tests have shown that mere washing with solvents does not give a reproducible degree of cleanliness in every case. Ultrasonic cleaning has proved suitable. Ultrasonic exposure of 30 to 60 sec is adequate.

b) Regulating the temperature (viscosity)

Since the viscosity during operation is the determining factor for the hydrodynamic part, the viscosity of the fluid under compression must remain constant before entering the pump. For viscosity to be automatically controlled, it must be measured continuously. For this purpose, only rotary-type viscometers can normally be used (in these the shear gradient on the rotating assembly affects the measurement of viscosity). According to the composition of the hydraulic fluid, particularly in the case of fluids containing polymerization products, an additional factor is thus involved. For this reason, instead of direct control through viscosity the temperature must be controlled. A sensing thermometer at the outlet of the cooler controls the cooling water flow.

c) Fixing the test temperature

In order to compare the performance of products of different rypes and viscosities, the fluid temperature is kept such that viscosities of 13 cSt are obtained for fluids of form D. For fluids of forms A, B and C, the temperature must give the following viscosities:

Fluids THI 2	13 cSt
THI 4	30 cSt
THI 8	60 cSt

d) Fixing the working pressure

Generally speaking, the working pressure is twice the rated pressure for this type of pump, viz. 140 bars for fluids of form D. For fluids of forms A, B and C, the test is carried out at 105 bars.

e) Duration

The pump test must not exceed 250 hours.

f) Requirements for parts subject to wear

The parts subject to wear must be checked very carefully before assembly for compliance with tolerances. To detect cracks, an ultrasonic examination of each rotor before assembly is recommended. Using a 12 MHz miniaturized test head, all points on the front and side surfaces between the grooves of the rotor, both radially and axially, should be examined.

3.13.8. PUMP TEST PROCEDURE

Before the test itself begins, all components of the test bench containing fluids must be carefully cleaned. Butyl glycol is used as the cleaning fluid for checking aqueous fluids (forms A, B and C) and motor spirit for non-aqueous fluids. The cleaning fluid is drained off completely by dismantling all the piping. A new filter element is placed in the filter. The apparatus is then rinsed with the fluid to be tested. The rinsing fluid is discarded.

For the test, 55 litres of fluid are placed in the apparatus.

A new Vickers No 711628* cartridge is used for each test. The components subject to wear are first of all roughly cleaned with a rag dipped in motor spirit, and then with a rag dipped in acetone. Final cleaning takes place in the ultrasonic bath. After weighing, the components are left overnight in the test fluid.

In the case of aqueous fluids the shaft and bearings, which are subjected to intensive loads, are replaced as often as possible.

For the pump test, the components subject to wear are assembled - the vanes being mounted the right-way round for the direction of rotation - and the test bench is run on no-load for at least one hour, without pressure and with the cooling water supply cut off (the contact period). The difference between the level of the fluid in the bench and the pump intake must be at least 500 mm, extra fluid being added if required. At the end of the contact period, the pressure is raised to 20 bars within 60 sec. The pump must be operated at this pressure for 10 min. The pressure is then stepped up by about 20 bars every 10 min (the running-in period). The cooling water is set in circulation only when the fluid reaches the rest temperature. For forms A, B and C fluids the maximum working pressure of 105 bars is reached after 50 min running-in time, and for form D fluids the maximum pressure of 140 bars is reached after 70 min. The flow is set at 28 litres/min ± 2 litres/min by means of the cover securing screws. These are tightened with a torque wrench, (range 0-400 cm kf) making sure that the lid stays perpendicular to the shaft.

(*Certificate No 71162)

The operating temperature may not vary from ± 20 °C. After 30 min the pressure in the inlet line is measured. It must not exceed - 0.05 bar.

The test can be interrupted at suitable intervals (e.g. 50, 100, 250 hours) to calculate the variation in weight of the components subject to wear.

3.13.9. INTERPRETATION OF RESULTS

To evaluate the anti-wear properties the variation in weight of the components subject to wear (ring and vanes) is determined in each case as a function of the operating time, inclusive of running-in.

3.13.10. REPRODUCIBILITY OF RESULTS

Differences on repetition of the test arise not only from the fluid, but also due to the manufacturing tolerances of the components subject to wear and to the accuracy of observance of the test conditions. Faulty assembly of the components subject to wear or incomplete bleeding of air from the system may produce discrepancies in the results. High weight losses - over 1,000 mg - are bound to be accomponaied by big absolute differences between individual measurements. With high weight losses, fluids cannot be evaluated solely in accordance with the absolute difference in weight sustained by the wearing components. Here, the operating time up to the occurence of seizing noises, pressure loss reduced flow, or breakage of the shaft must be taken into account.

-(a)-

ASSESSMENT OF ANTI-WEAR PROPERTIES

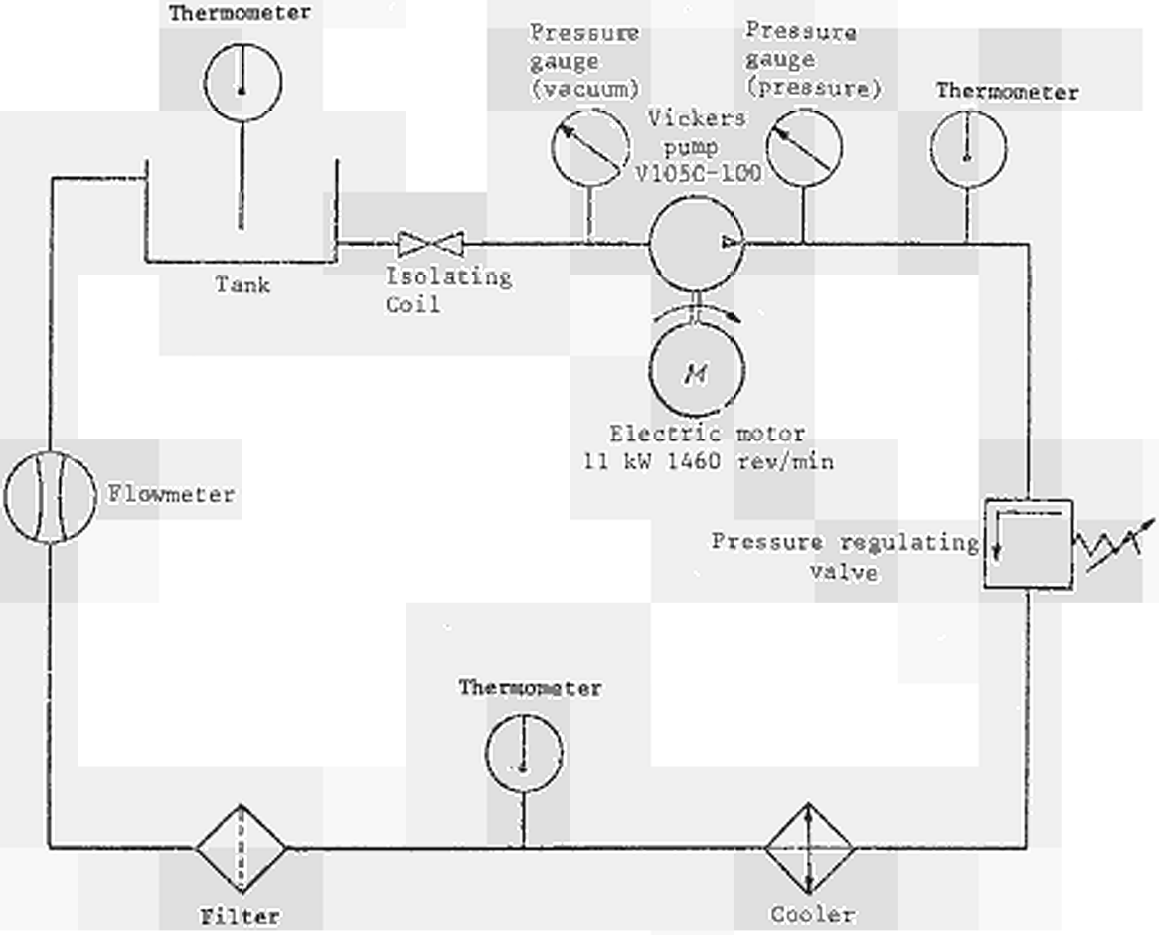


Diagram of test assembly

DETERMINATION OF FOAMING TENDENCY

(Taken from ASTM 892 - 63)

3.14.1. APPLICATION

Hydraulic fluids with or without addition of water (forms A, B, C and D).

3.14.2. PRINCIPLE

A well-diffused current of air is introduced into the fluid at different temperatures and the resulting volume of foam is measured.

3.14.3. APPARATUS

a) 1000 ml measuring cylinder.
For this volume the graduated scale for 1000 ml must extend over 365 mm ± 15 mm.

b) Air inlet tube with alundum diffusion stone (Norton Company, Refract. Division, Worcester, 6, Mass., U.S.A., designation No. ME 46239, fine grade). The air inlet tube is made of brass and is at least 450 mm long. The diffusion stone, which is spherical, porous, made of sintered aluminium oxide, and 25 mm in diameter, is attached to the lower end of the inlet tube.

c) Thermostat consisting of a glass vessel filled with water, fitted with a heating system, an automatic temperature control device and a stirrer. The glass vessel must be big enough for the measuring cylinder to be immersed up to the 900 ml mark. The temperature of the heating bath must be adjustable either to 20 °C ± 0.5°, or to 50 ± 0.5°, or to 95 ± 0.5 °C.

d) Flowmeter to set the specified air flow: the types used are either the normal rotameters or the fluid-filled U-tube type of flowmeter which indicates the pressure difference upstream and downstream of a capillary tube inserted in the air current. (It is advisable to have a constriction at the bottom of the U-tube to damp out deviation waves in the fluid due to pressure variations.)

e) Timer.

f) Thermometer: microthermometer graduated 0 to 100 °C, with scale divisions of 0.2 °C.

3.14.4. CHEMICALS

a) Petroleum ether or heptane.

b) Chemically pure acetone.

c) pure carbon tetrachloride.

3.14.5. PREPARATION OF TEST

a) To eliminate all the fluid residues from previous tests, which might in certain circumstances falsify the results, the apparatus used for the test must be thoroughly cleaned.

This is essential in particular for:

aa) The measuring cylinder: this must be thoroughly cleaned with petroleum ether or heptane, then with acetone and finally with distilled water, after which it is dried in a current of pure (oil-free) air;

bb) the diffusion stone in the air inlet tube: the stone is cleaned successively with petroleum ether or heptane, pure $C\ Cl_4$ and finally with a fresh quantity of petroleum ether or heptane, by immersion in about 300 ml of each of these solvents, which are aspirated under vacuum and then expelled by air pressure (five times for each solvent), then dried in air. After this, the diffusion stone is place in boiling carbon tetrachloride; it is rinsed in acetone and dried in oil free air. The pipe is finally wiped with a clear dry cloth.

b) The air inlet tube is then inserted in the orifice of a rubber bung pierced in two places, which seals the measuring cylinder, so that the diffusion stone just touches the bottom of the measuring cylinder.

c) Then some 200 ml of the sample are heated in a clean glass vessel to 50 ± 2 °C and then cooled to 25 °C ± 2°. In addition, the heating bath is brought to 25 ± 0.5 °C. The measuring cylinder is filled up to the 190 ml mark with the prepared sample, and then placed in the heating bath in such a way that the water reaches at least the 900 ml mark.

3.14.6. TEST PROCEDURE

a) As soon as the fluid in the measuring cylinder has reached the temperature of the bath, the rubber bung carrying the air-inlet tube is placed on the measuring cylinder (with the diffusion stone just touching the bottom of the cylinder) and a period of 5 minutes is allowed to elapse for the stone to become saturated with fluid. After this operation, the tube is connected to the aire supply and a flow of air of 94 ml ± 5 ml per minute is established. (The air must be dry and oil-free). If necessary, one or more filters containing calcium chloride, activated carbon or cottonwool can be placed in the air line.) When 5 minutes ± 10 seconds has elapsed after the appearance of the first air bubbles on the surface of the diffusion stone, the flow of air is cut off. The volume of foam which has formed between the upper edge of the foam layer (average value) and the underlying layer of fluid is immediately measured in ml.

Without removing the air inlet, the measuring cylinder is left for a further 10 minutes ± 10 seconds in the water bath, after which the foam volume is again measured.

b) For forms A, B and C fluids at 50 °C, and for form D fluids at 95 °C, the preparations are identical to those described in section 5, but the bath is heated to 50 ± 0.5°, or 95 ± 0.5 °C respectively.

For introducing the air, a freshly-cleaned air-inlet tube with a diffusion stone should be used.

The measurement is taken as in section 6a) after the necessary lapse of time for bubble formation or settlement time, as appropiate.

c) The foam remaining after the test described in section 6b) is then broken down by gentle stirring. The fluid is cooled to below 40 °C, by exposing the measuring cylinder to the ambient air, then a previously cleaned air inlet tube fitted with a diffusion stone is inserted. When the fluid has reached the temperature of the heating bath (25° ± 0.5 °C), the process described in section 6a) is repeated and the volume of foam measured after the lapse of time necessary for bubble formation and settlement.

3.14.7. EXPRESSION OF RESULTS

Table

Temperature of fluid samples	Volume of foam measured immediately after interruption of air supply	Foam volume after 10 min
25 °C	.... ml	.... ml
50° or 95 °C	.... ml	.... ml
25 °C (after test at 50° or 95 °C)	.... ml	.... ml

DETERMINATION OF EMULSION STABILITY

3.15.1. PRINCIPLE

To check that the emulsion prepared ready for use remains stable when stored, at two temperatures: + 20 °C, + 50 °C.

3.15.2. APPARATUS

a) Two cylindrical test tubes of capacity about 250 ml each, with ground stoppers; these tubes should be approximately 40 mm in external diameter and about 240 mm high for 250 ml.

b) Thermostats for temperatures of + 20 °C and + 50 °C, accurate to within ± 1 °C.

c) Mechanical stirrer.

3.15.3. TEST PROCEDURE

a) Sample preparation

The volume of the sample is approximately 600 ml. Two cases may occur:

aa) the emulsion is ready for use;

bb) only a concentrate is supplied, and the emulsion must be made up before use.

In the second case, the emulsion is made up using water with a hardness index of 40 ± 5, by adding calcium chloride to distilled water, with mechanical stirring for at least five minutes.

b) The test

The sample is divided between the two 250 ml test tubes. These are then sealed and placed in the conditions of temperature mentioned above, one test tube in the thermostatically controlled bath at + 20 °C, and the other in the thermostatically controlled bath at + 50 °C. The samples are examined after a test period of 600 hours.

DETERMINATION OF DE-AERATION ABILITY

3.16.1. APPLICATION

The method serves to determine the air expulsion properties of fluids of forms A, C and D.

3.16.2. PRINCIPLE

In an "impinger" test vessel, compressed air is blown into the fluid from a nozzle. Intensive mixing of fluid with air produces supersaturation with air. The time taken by the bubbles to rise in the fluid is a measure of the de-aeration ability. Since it is not possible to measure the rate of ascent of bubbles by simple means, the time taken for the sample of fluid to rid itself of air bubbles is measured. The time taken by the fluid to eliminate dispersed air (in the form of air bubbles) up to 0.2 % by volume, is the criterion used to determine the de-aeration ability.

3.16.3. APPARATUS AND CHEMICALS

Filter, e.g. with silica gel for eliminating oil and impurities from the compressed air;

Compressed air heater;

Thermometer, scale 0 to 100 °C, graduated in 0.1 °C divisions;

Mercury pressure gauge, scale 0 to 250 torr;

Measurement vessel with air inlet tube (Impinger, as per RW TÜV, Essen), as in fig. 1;

Densimetric balance - reading accuracy 0.001 g/ml - with 5 ml volumetric displacement plunger, length 80 mm ± 1.5;

Thermostat with 50 °C setting;

Carbon tetrachloride;

Sulphochromic acid;

Acetone.

3.16.4. PREPARATION

The apparatus is assembled as shown in figure 2. The compressed air is supplied either from a compressed air line or a compressed air cylinder. Before it enters the impinger, it must be heated to the test temperature by the heater.

The inside of the impinger and the air inlet piping must be carefully cleaned before each determination. Traces of fluid of form D are eliminated by rinsing with carbon tetrachloride and those of forms A and C with water. Cleaning is continued with sulphochromic acid, then by rinsing with distilled water until the final water no longer contains any traces of acid. The various parts of the apparatus are then rinsed with acetone and dried with filtered compressed air.

The test sample is heated in a drying cabinet to 40 to 50 °C, then shaken vigorously. 180 ml of the sample heated to 40 to 50 °C are placed in the impinger. The thermostat is set at 50 °C ± 0.1 and the fluid in the impinger is kept at 50 °C ± 0.1. The temperature must be checked by means of the thermometer.

When a temperature of 50 °C is reached, the plunger of the densimeter is placed in the fluid, care being taken that no air bubble adheres to it and that the inner edge of the plunger is 10 mm ± 2 above the bottom of the impinger. When the plunger has reached the temperature of the fluid, the density of the fluid is measured and the reading noted (initial value of the density of the fluid with no air bubbles = d_o). The plunger is then withdrawn and placed in a thermostatically controlled air bath at the test temperature (e.g. a glass container placed in the thermostat).

3.16.5. DETERMINATION PROCEDURE

The air inlet piping is fitted to the impinger and the pipes are connected as shown in figure 2. The air outlet pipe is connected to an exhaust line. After a 5 minutes waiting period, air is introduced. The pressure gauge is set at 150 torr and this pressure is maintained for 7 minutes, with adjustments if necessary.

After 7 minutes, the air inlet is closed, the unions are disconnected and the air inlet pipe is removed very rapidly from the impinger. Immediately afterwards, the plunger, maintained at the test temperature, is placed in the air containing fluid, the base of the plunger being kept 100 mm ± 2 above the bottom of the impinger. The first density measurement (d_1) must be taken not more than one minute after the air is cut off. Density readings (d_n) are then taken every minute. Measurement ends when the initial density of the fluid without air (d_o) is reached. If the separation time exceeds 15 minutes, the interval between each reading may be increased to 5 minutes after the first 15 minutes.

3.16.6. EXPRESSION OF RESULTS

The air content is calculated as follows from the density readings obtained:

$$L_n = \frac{100\ (d_o - d_n)}{d_o - d_l}$$

L_n = content of air dispersed in the fluid (% volume) after n min (n = 1, 2, 3 etc. minutes);

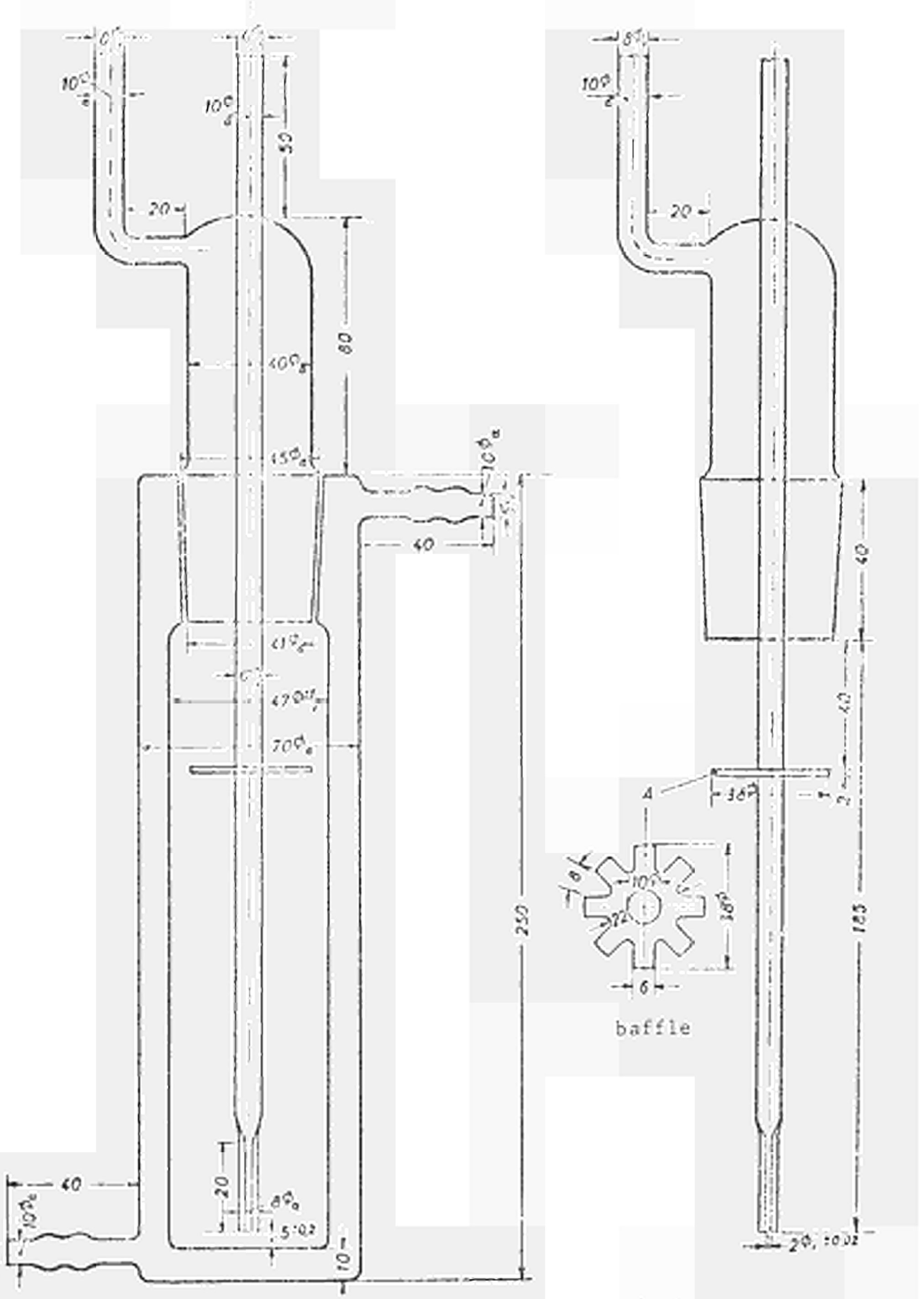
d_o = density of the fluid with no air bubbles, in g/ml;

d_n = density of the fluid containing air, after n = 1, 2, 3, 4 etc. minutes;

d_l = density of air in g/ml at the test temperature.

The air content of the fluid (air bubbles) expressed as % volume is plotted graphically against the number of minutes elapsed after the cut off of the air supply. On the graph, the de-aeration ability of the fluid corresponds to the time after which, after the cut-off of the air, the volume of air dispersed in the fluid (air bubbles) has fallen to 0.2 % by volume. The result is expressed in minutes, to the nearest minute.

-(a)-



Test vessel (impinger)

Air inlet pipe

Fig. 1 - TÜV ESSEN IMPINGER USED FOR DISPERSION OF AIR IN OIL

Note: all dimensions in mm.

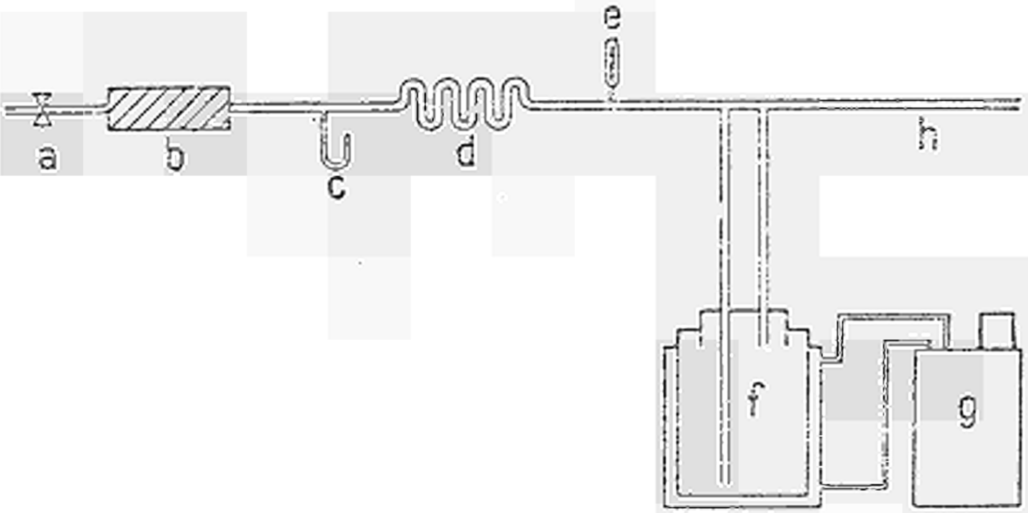


FIG. 2 - TEST ASSEMBLY. a) REGULATING COCK, b) FILTER, c) PRESSURE GAUGE, d) HEATER, e) THERMOMETER, f) IMPINGER WITH AIR INLET PIPE, g) THERMOSTAT, h) AIR OUTLET

PART IV - DESCRIPTION OF TEST PROCEDURES CONCERNING THE HEALTH HAZARD

(for fluids meeting the 'Community of Six' requirements)

DETERMINATION OF THE TOXICITY OF FIRE-RESISTANT HYDRAULIC FLUIDS MISCIBLE WITH WATER, GROUPS A AND C

4.1.1 IDENTIFICATION BY INFRA-RED SPECTROPHOTOMETRY

A weighed sample of fluid is placed in a phosphoric anhydride desiccator, heated to 70°C and left until it reaches constant weight.

The residual fluid is extracted ten times by its own volume of peroxide-free ethyl ether.

The ether fraction collected is evaporated *in vacuo*. An infra-red spectrum of the residue is taken.

With a fluid of form A, it is possible to separate the oily phase, which can be used for infra-red spectrophotometry, from the original fluid by physical means.

The spectra are not analysed, but are used as identity references for the product.

4.1.2 ORAL TOXICITY

The determination of oral toxicity (LD 50, i.e. the lethal dose for 50% of the animals) is performed on male white mice and is expressed in g/kg body weight.

The hydraulic fluid under test (diluted with water where appropriate) is administered to adult male white mice by buccal probe. The observation period is 14 days.

The buccal probe is fitted as a cannula to a tuberculin syringe. The front end of the probe is rounded and thickened, to avoid injury to the animals.

Each mouse is weighed, and the quantity of fluid introduced into its stomach referred to the weight of the animal.

The first approximation of LD 50 is estimated on a small number of animals. Four of them receive a dose P. The next four receive a dose of 2P, the next four 4 P, and so on. If the information dose P proves too high, the subsequent experiments should be performed with fractions of P.

After this first approximation, the range of LD 50 is fairly well defined. The test is then repeated on groups of mice, the numbers of which are determined by the statistical certainty of the values obtained (tolerance limit 20%). Each group receives 0.4, 0.6, 0.8, 1.0, 1.2, etc. times the roughly estimated LD 50 values.

For each dose, the number of dead animals is expressed as a percentage (total number of animals used for each dose = 100%). This value is plotted (ordinates) in a logarithmic probability graph against the dose (g/kg) (abscissa). Through the resulting points, the approximation straight line is drawn; from this the dose (g/kg) proving lethal for 50% of the animals is determined graphically.

The results are expressed on a points basis, as follows:

LD $50 > 5$ g/kg : 0
LD $50 \leq 5$ g/kg and 0.05 g/kg: 5
LD $50 \leq 0.05$g/kg : 10

4.1.3 DETERMINATION OF TOXICITY

(a) Overall toxicity

The test is started with the liquid at normal concentrations (ie. as used) in a shallow vessel (capacity 5 litres) and at room temperature, by diluting this liquid to a final volume of 3 litre at concentrations of:

1.10^{-1}
1.10^{-2}
1.10^{-3}
1.10^{-4}
1.10^{-5}

made up with water; three goldfish (Carassius auratus), weighing 5 to 10 g, are placed in each vessel. Over a total period of 120 hours the survival time of each batch of fish is noted. If any fish die within this period, the symptoms appearing before death are noted: haemorrhages or motor co-ordination disturbances.

The results are scored as follows:

Survival of all fish : 0

The fish in the 1.10^{-1} concentration die in more than one hour: 1
in less than one hour: 2

The fish in the 1.10^{-2} concentration die in more than one hour: 3
in less than one hour: 4

The fish in the 1.10^{-3} concentration die in more than one hour: 5
in less than one hour: 6

The fish in the 1.10^{-4} concentration die in more than one hour: 7
in less than one hour: 8

The fish in the 1.10^{-5} concentration die in more than one hour: 9
in less than one hour: 10

(b) Irritant effect

(1) The irritant effect on the eyes:

Using a standard eye-dropper, a drop of test fluid at normal concentration is placed in the conjunctival sac of the right eye of two male albino rabbits weighing not more than 1 kg.

The state of the right eye and its accessory parts (except the external eyelid) is examined and compared with the left eye, once a day for 5 days.

During the whole test period the animals must be kept isolated.

Disregarding any infections, the results are scored as follows:

No reaction observed	: 0
Inflammatory reactions, according to speed of appearance, extent and duration	: 1 to 5
Permanent lesions without loss of vision	: 6 to 10
Loss of vision	: 10

(2) The irritant effect on the skin:

Using the same animals as were employed for test (1) cutaneous irritation is investigated by the patch test, as follows:

24 hours before the test, a patch of skin on the side measuring 8 x 8 cm is sheared without injuring the epidermis.

A piece of cotton wool measuring 4 x 4 cm covered by a sheet of inert material which also prevents the test fluid from diffusing, is applied to the patch in such a way that direct contact with the skin is possible only over an area of 2 x 2 cm.

After the cotton wool has been soaked in 2 ml of the test solution, the material is placed against the skin and bandaged into position.

After a contact time of 24 hours, it is removed and the condition of the skin is examined immediately and daily for the next 14 days.

Disregarding any infection, the results are classified as follows:

No reaction observed	: 0
Erythema, according to extent and duration	: 1 to 3
Erythema and oedema (cutaneous) without induration	: 4
Complex lesions of the skin according to extent and duration	: 5 to 10
Death of the test animals	: 10

(c) Aerosol toxicity

The apparatus is indicated in Figure 1: details of the different parts are given on the diagram.

The liquid, diluted at normal usage concentrations, for A type fluids, and at the rate of 1 to 2 for C type fluids, with distilled water, is placed in an aerosol generator. The temperature of the generator is maintained at 20°C for A type liquids and 70°C for C type liquids. The temperature is controlled by means of a thermocouple where the hot joint is put on the aspiration orifice of the atomiser. The air pressure applied to the aerosol generator is measured by means of a manometer. The generator must dispense 20 to 30 ml of liquid per hour such that the aerosol produced contains 90% (by number) of particles less than 3 microns in diameter. The consumption of air must be 1 litre per hour.

After leaving the aerosol generator, the mist passes through a U tube made of stainless steel, carrying at its bottom a collector, then it is carried to the inhalation chamber by a flexible tube. The inhalation chamber is made of transparent plastic, with a volume of about 140 litres. The recommended dimensions are 800 mm long, 350 mm wide and 500 mm high. The aerosol enters the chamber laterally by an orifice situated at 12 cm from the end and leaves by an exit orifice situated on the opposite wall at the same height. In the front face of the chamber is a door, where the animals are put in. This door has a port designed to allow samples to be taken. The rats are placed in a cage with top and side faces formed from bars or mesh. The cage must be placed centrally and 7 cm above the floor of the inhalation chamber. A depression of 3 mm (water quage) must be maintained in the inhalation chamber whilst a variable means of aspirating is connected in the exit port. The depression is measured by means of an inclined guage. A thermometer controls the temperature of the interior of the inhalation chamber.

Three male Wistar rats each of about 200 grams in weight are put into the cage where they are exposed to the aerosol for one hour. After the end of the test, and for the following 14 days, the animals are weighed.

Three other rats of the same weight and age are kept in similar conditions as a control sample.

Afterwards, all infections/disorders recorded are counted in the following manner:

- no reaction noted.. 0
- symptoms of irritation e.g. disturbance of circulation or nervous system.. 1
- loss or insufficient gain in weight as compared with the control sample
  Maximum difference noted:
  - during first 3 days.................................... 2
  - between 3 and 6 days.................................. 3
  - more than 6 days...................................... 4
- death of one of the animals in 14 days...................... 5

- death of two of the animals in 14 days	6
- death of three of the animals in 14 days	7
- death of one of the animals during the test	8
- death of one of the animals during the test and another within 14 days	9
- death of two of the animals during the test	9
- death of one animal during the test, and two others within 14 days	10
- death of two animals during the test and the other within 14 days	10
- death of three animals during the test	10

(d) Thermal decomposition products:

The test is to be carried out at 200 and 700°C on a liquid diluted at normal usage concentrations sprayed onto a plate heated to 200°C and 700°C.

The apparatus is represented by the diagram. Figure 2. The details of the different parts are indicated in the legend.

The thermic decomposition of the fluid occurs in a closed metallic chamber (A). The liquid is placed in a graduated flask connected to a single cylinder Diesel injection pump (B). The pump, rotated at 300 r.p.m. supplies the fluid to a Diesel Injector (C), placed at the end of the chamber. The cone angle of the injector is 45°, with a pressure of 100 bars, at one milliliter per minute. The atomised liquid is projected onto a metal plate (D), 6 cm in diameter, placed 11.5 cm from the nozzle of the atomiser. The plate is heated electrically (E), to a controlled temperature of 200° and 700°C, checked by thermo-couple (F) with the joint 1 mm from the surface of the plate, and maintained constant throughout the test to ± 10°C of the prescribed temperature (see figure 3).

The resulting aerosol is carried in a primary air current of 10 litres per minute, to enter the chamber by two lateral orifices (G). The vertical wall (H) acts as a baffle between the injector and the plate to prevent direct aspiration of the injected product out of the exit port. The chamber is inclined at the prescribed angle towards the collector I.

At the exit of the chamber, the aerosol passes through a condensation trap, formed from a stainless steel U-tube (J), with a collection point at K.

At the end of the U-tube, the aerosol is diluted by a secondary 20 litre per minute air current. To ensure a good mixture, the secondary air is injected in the counter-current direction, at an angle of 30° by a Y-tube (L). The primary air current and the secondary are supplied from sources M and N, which are monitored by rotameters. The tubes are dipping at 1 in 25 to ensure the flow of condensing fluid towards the collector.

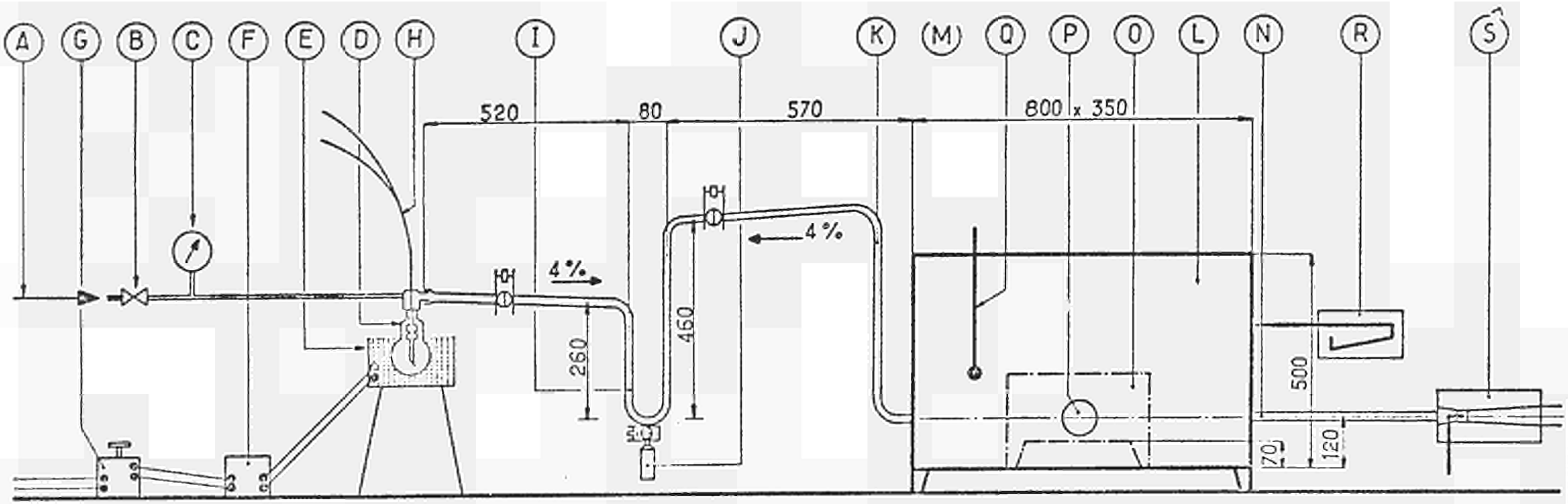
Thus the diluted aerosol is sent through a flexible tube (O) into the inhalation chamber (P), which is formed from transparant plastic, with a volume of approximately 140 litres; the recommended dimensions of which are 800 mm long, 350 mm wide, and 500 mm high. The aerosol enters the chamber by a lateral orifice (Q), situated 12 cm from the end, and leaves by port (R) situated in the opposite wall at the same height. In the front face of the chamber is a door allowing the animals to be introduced. This door has a port designed to allow the concentration of the aerosol in the chamber to be measured. The rats are placed in the cage (T), the top and side faces of which are formed from bars or mesh. The bottom of the cage must be 7 cm above the floor of the inhalation chamber. A depression of 3 mm (Water Gauge) is maintained in the inhalation chamber and a means of aspirating (U) is connected to the outlet. The depression is measured by an inclined guage. The thermometer (V) measures the temperature of the interior of the inhalation chamber. The procedure adopted and the scale of values to be applied to the results is the same as described in Part IV 4.1.3 (c). The scoring is valid for thermal decomposition products at both 200 and 700°C.

(e) Final scoring and conditions of approval

Each product tested is given a rating of 0 to 170, determined by the sum of all the scores obtained in each of the above tests, the latter being weighted by the coefficients given in the following table:

Oral toxicity	LD 50	1
Irritant effect	eyes	5
	skin	5
Aerosol	cold or hot	2
Test on fish		2
Thermal decomposition	200°C	1
	700°C	1

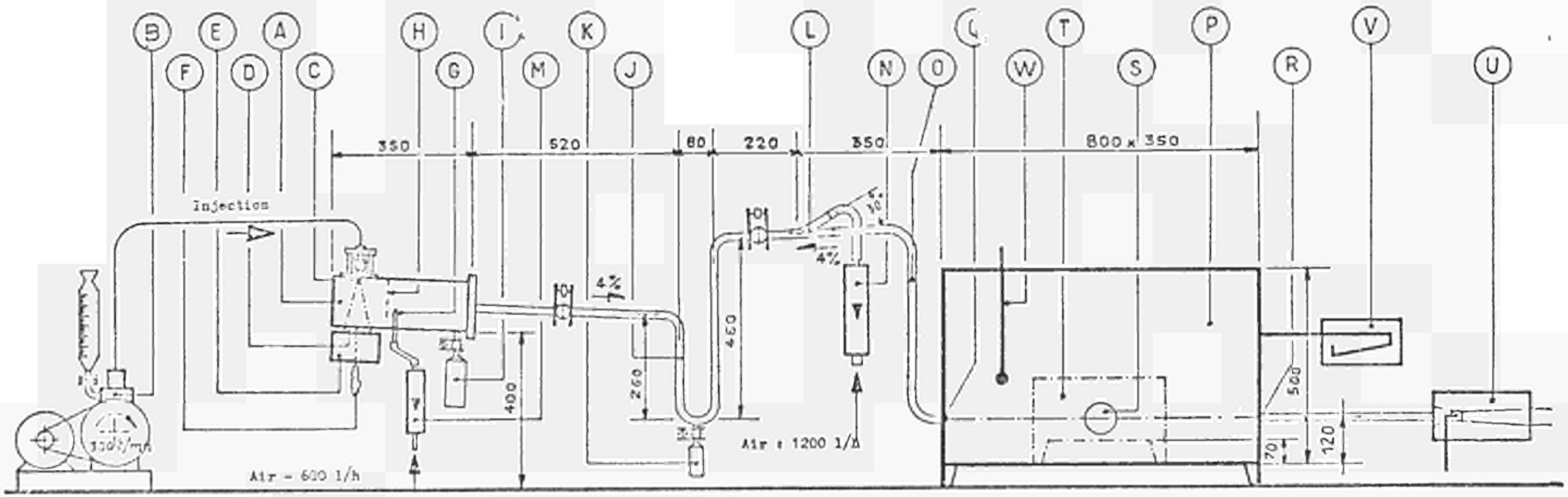
Any product which scores 10 before weighting in any one test, or 50 after weighting, is rejected.



LEGEND :

A Compressed air supply
B Pressure reducer
C Control manometer
D CERCHAR aerosol generator Type A1 with changeable nozzles for Types A-C and D liquids.
E Heating element 220 volt - 65 watt for 100 ml bulb
F Temperature control
G Variable autotransformer 400 VA
H Thermocoax Thermocouple; Cr/Al 0,5 mm diameter
I Condensing U tube of 15 mm. internal diameter fitted with Rotolex 29/15 spherical valve
J Condensate collector through spherical valve - Volume 25 ml.
K Flexible tube internal diameter 15 mm
L Inhalation chamber 140 l. Internal dimensions 800 x 350 x 500 mm.
M Inlet 15 mm. dia. for aerosols
N Outlet 15 mm. dia. for aerosols
O Cage for the animals; Length 320 mm. Width 250 mm. and height over feet 230 mm.
P Sampling orifice
Q Thermometer 0 - 50°C
R Inclined gauge
S Means of aspiration

Figure 1. Schematic diagram of the apparatus for testing the toxicity of aerosols.



LEGEND :

A	Metallic chamber: internal dimensions: Length 350 mm, Width 66 mm, Height 119 mm.
B	BOSCH pump Type 0400.462.024, modified to single cylinder with piston 5 mm dia. (1418.425.000)
C	Injector BOSCH (0434.200.024 - DN 30S3)
D	Heated stainless steel plate
E	Heating element; 220 v. 1800 watt ceramic ring 90 mm dia. 78 mm high, externally armoured in stainless steel. Supplied by Ste VULCAIN 1 Av. Kleber, Nogent sur Marne 94
F	Thermocouple (thermocoax chromel-alumel Ø 0,5 mm)
G	Air entry 10 mm internal diameter
H	Baffle 95 mm high 66 mm wide
I	50 ml. collector for liquid, connected through Rotulex spherical valve 19/9
J	Condensor formed from 15 mm. internal diameter tube with collector connected through Rotulex 29/15 valve
K	Collector for condensate Volume 25 ml connected through spherical valve
L	Y Tube for dilution, angle 30°, 8 mm air entry tube
M-N	Rotammeters
O	Flexible tube 15 mm diameter
P	Inhalation chamber 140 l. Internal dimensions: Length 800 mm, Width 350 mm, Height 500 mm
Q	Aerosol entry 15 mm diameter
R	Aerosol exit 15 mm diameter
S	Sampling orifice
T	Cage containing the animals 320 mm long, 250 mm wide, and 230 mm high
U	Aspirator
V	Inclined gauge
W	Thermometer 0 - 50°C.

Fig. 2 - Schematic diagram for the apparatus for testing the products of thermal decomposition

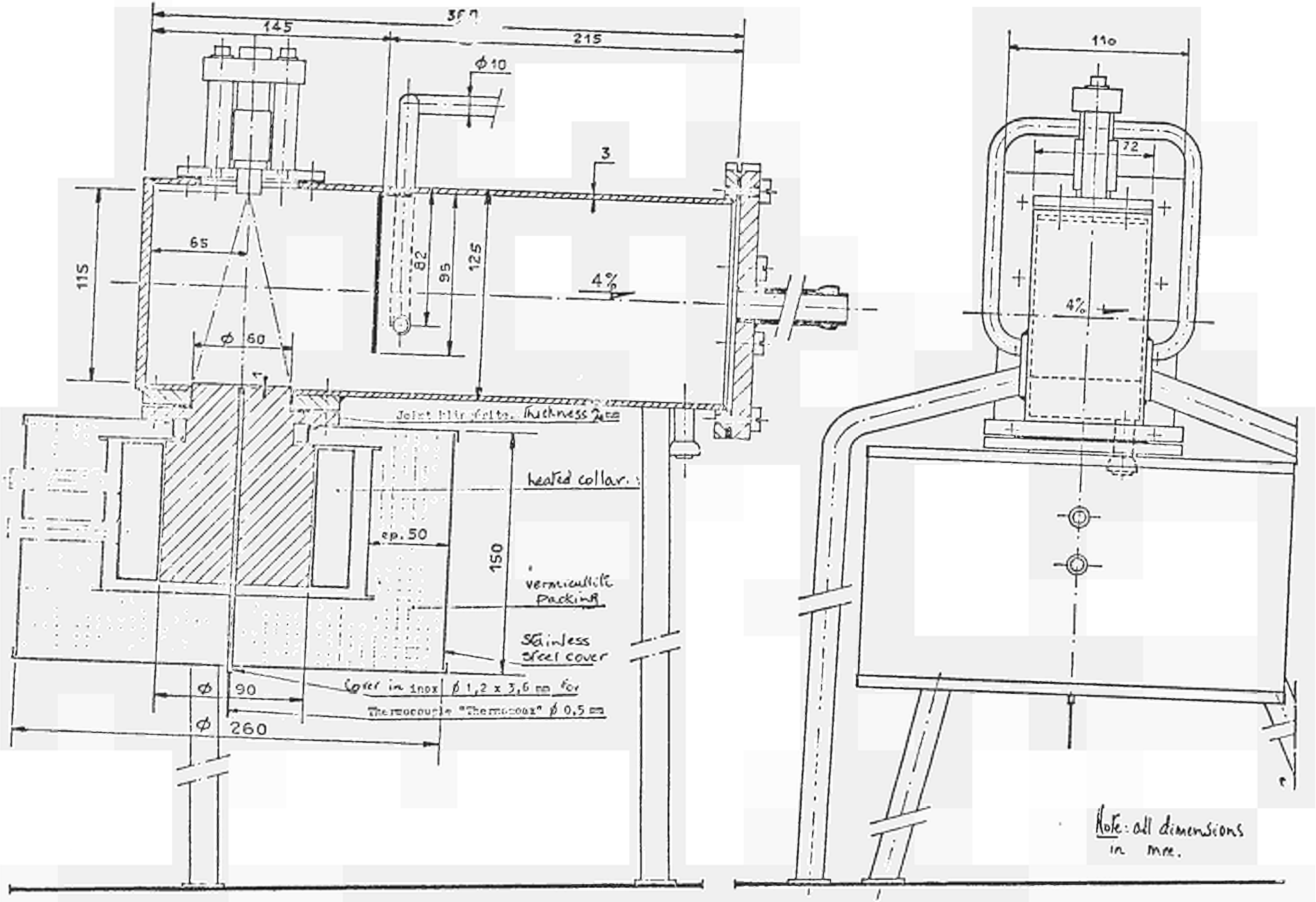


FIG. 3 - TEST CHAMBER FOR PRODUCTS OF THERMAL DECOMPOSITION

DETERMINATION OF THE TOXICITY OF FIRE-RESISTANT HYDRAULIC FLUIDS NOT MISCIBLE WITH WATER, FORM D

4.2.1 IDENTIFICATION BY INFRA-RED SPECTROPHOTOMETRY

An infra-red spectrum is taken of a sample of fluid. The spectra are not analysed but are used as an identity reference for the product.

4.2.2 ORAL TOXICITY

The determination of oral toxicity (LD 50, i.e. the dose which causes the death of 50% of the animals) is performed on male white mice; it is expressed in g/kg of body weight.

The hydraulic fluid under test (diluted where appropriate in water) is administered to adult male white mice by buccal probe. The observation period is 14 days.

The probe is fitted as a cannula to a tuberculin syringe. The front end of the buccal probe is rounded and thickened to avoid injury to the animals.

Each mouse is weighed and the quantity of product introduced into its stomach referred to the weight of the animal.

The first approximation of LD 50 is determined on a small number of animals. Four of them receive a dose P. The next four receive a dose 2 P, the next four 4 P, etc. If the information dose P is too high, subsequent experiments should be performed with fractions of P.

After this first approximation, the range of LD 50 is fairly well defined. The test is then repeated on groups of mice, the numbers of which are determined by the statistical certainty (tolerance limit 20%) of the values obtained. Each group receives 0.4, 0.6, 0.8, 1.0, 1.2, etc., times the roughly assessed LD 50 values.

At each dose the number of dead animals is expressed as a percentage (total number of animals used for each dose = 100%). This value is plotted (ordinates) in a logarithmic probability diagram against the dose (g/kg) (abscissa). The approximation straight line from which the lethal dose (g/kg) for 50% of the animals is determined graphically is drawn through the points obtained.

The results are scored as follows:

LD 50 $>$ 5 g/kg : 0
LD 50 $\leqq$ 5 g/kg : 5
LD 50 $\leqq$ 0.05 g/kg : 10

4.2.3 DETERMINATION OF IRRITANT EFFECT

(a) Irritant effect

1. Test to determine irritant effect on the eye

Using a standard eye-dropper, a drop of test fluid is placed in the conjunctival sac of the right eye of two male albino rabbits of not more than 1 kg weight. The state of the right eye and its accessory parts (excluding the external eyelids) is examined and compared with that of the left eye, once a day for 5 days.

The animals must be kept isolated during the whole test period.

Disregarding any infections, the results are scored as follows:

No reaction observed	: 0
Inflammatory reactions according to speed of appearance, extent and duration	: 1 to 5
Permanent lesions without loss of vision	: 6 to 10
Loss of vision	: 10

2. Irritant effect on the skin

On the same animals used for test A, the cutaneous irritation is investigated by the patch test described below:

24 hours before the test, a patch of skin on the side measuring 8 x 8 cm is shaved, taking care not to injure the epidermis. The material applied to the patch consists of a piece of cotton wool 4 x 4 cm, covered with a sheet of enert material which also prevents the test fluid from diffusion, in such a way that direct contact with the skin is possible only over an area of 2 x 2 cm.

After the cotton wool has been soaked in 2 ml of the test fluid, the material is placed against the skin and bandaged into position.

After a contact time of 24 hours, the material is removed and the condition of the skin is examined immediately and daily for the next 14 days.

Disregarding any infections, the results are classified as follows:

No reaction observed	: 0
Erythema, according to extent and duration	: 1 to 3
Erythema and cutaneous oedema, without induration	: 4
Complex lesions of the skin according to extent and duration	: 5 to 10
Death of the experimental animals	: 10

(b) Aerosol toxicity

1. Cold aerosol

The apparatus is the same as that shown in figure 1, part IV.4.1 The details of the elements are shown in the legend.

The undiluted liquid is placed in the aerosol generator which is maintained at 50° C. The temperature is controlled by a thermocouple the joint of which is placed in contact with the aspiration nozzle. The input air pressure is measured by a manometer. The generator must disperse from 10 to 15 ml per hour of liquid in the form of an aerosol. The 90% of particles from which (when counted) are less than 3 microns in diameter. The air consumption is 1 m^3 per hour.

On leaving the generator, the aerosol passes through a stainless steel U-tube with a trap at its lowest point, and is then sent by flexible tube to the inhalation chamber built of transparant plastic, with a volume of about 140 litres. The dimensions recommended for this are 800 mm long; 350 mm wide, and 500 mm high. The aerosol enters the chamber by a lateral orifice 12 cm from the end, and leaves by an orifice in the opposite wall at the same height. In the front face of the chamber is a door allowing the animals to be introduced. The door has a port through which samples for the determination of the concentration can be taken. The rats are placed in a cage, the top and side faces of which are constructed of bars or mesh. The bottom of the cage is 7 cm above the base of the inhalation chamber, and placed centrally inside it. A depression of 3 mm (Water gauge) is maintained in the chamber, as a means of drawing the aerosol towards the exit. This depression is measured by an inclined guage. A thermometer allows the temperature of the inhalation chamber to be controlled.

Three male Wistar rats each of about 200 grams in weight are put into the cage where they are exposed to the aerosol for one hour. After the end of the test, and for the following 14 days, the animals are weighed.

Three other rats of the same weight and age are kept in similar conditions as a control sample.

Afterwards all infections/disorders are counted in the following manner:

- no reaction noted..	0
- symptoms of irritation: e.g. disturbance of circulation or nervous system..	1
- loss or insufficient gain in weight as compared with the control sample: maximum difference noted	
- during first 3 days..................................	2
- between 3 and 6 days................................	3
- more than 6 days.....................................	4
- death of one of the animals in 14 days....................	5
- death of two of the animals in 14 days....................	6
- death of three of the animals in 14 days..................	7

- death of one of the animals during the test	8
- death of one of the animals during the test and another within 14 days	9
- death of two of the animals during the test	9
- death of one animal during the test, and two others within 14 days	10
- death of two animals during the test and the other within 14 days	10
- death of three animals during the test	10

2. Hot Aerosol

The test described above is repeated on a new group of animals with a liquid temperature in the generator of 150°C.

For all infections, the results are judged on the same scale as for the cold aerosol test.

(c) Thermal decomposition products

The test is to be carried out with undiluted fluid which sprayed onto a heated plate.

The apparatus is represented by the diagram, figure 2, part IV.4.1 The details of the different parts are indicated in the legend.

The thermic decomposition of the fluid occurs in a closed metallic chamber (A). The liquid is placed in a graduated flask connected to a single cylinder diesel injection pump (B). The pump, rotated at 300 r.p.m. supplies the fluid to a diesel injector (C), placed at the end of the chamber. The cone angle of the injector is 45° with a pressure of 100 bars, at one milliliter per minute. The atomised liquid is projected onto a metal plate (D), 6 cm in diameter, placed 11.5 cm from the nozzle of the atomiser. The plate is heated electrically (E), to a controlled temperature of 200° C and 700° C, checked by thermocouple (F) with the joint 1 mm from the surface of the plate, and maintained constant throughout the test to $\pm 10^{\circ}$ C of the prescribed temperature (see figure 3, part IV.4.1.).

The resulting aerosol is carried in a primary air current of 10 litres per minute, to enter the chamber by lateral orifice (G). The vertical wall (H) acts as a baffle between the injector and the plate to prevent direct aspiration of the injected product out of the exit port. The chamber is inclined at the prescribed angle towards the collector I. At the exit of the chamber, the aerosol passes through a condensation trap, formed from a stainless steel U-tube, with a collection point at K.

At the end of the U-tube, the aerosol is diluted by a secondary 20 litre per minute air current. To ensure a good mixture, the secondary air is injected in counter-current direction, at an angle of 30° by a Y-tube (L). The primary air current and the secondary are supplied from sources M and N, which are monitored by rotameters. The tubes are dipping at 1 in 25 to ensure the flow of condensing fluid towards the collector.

Thus the diluted aerosol is sent through a flexible tube (O) into the inhalation chamber (P), which is formed from transparant plastic, with a volume of approximately 140 litres; the recommended dimensions of which are 800 mm long, 350 mm wide, and 500 mm high. The aerosol enters the chamber by a lateral orifice (Q), situated 12 cm from the end, and leaves by port (R) situated in the opposite wall at the same height. In the front face of the chamber is a door allowing the animals to be introduced. This door has a port designed to allow samples to be taken. The rats are placed in the cage (T), the top and side faces of which are formed from bars or mesh. The bottom of the cage must be 7 cm above the floor of the inhalation chamber. A depression of 3 mm (Water Gauge) is maintained in the inhalation chamber and a means of aspirating (U) is connected to the outlet. The depression is measured by an inclined gauge. The thermometer (W) measures the temperature of the interior of the inhalation chamber. The procedure adopted and the scale of values to be applied to the results is the same as described in Part IV.4.2.3 (b) (1). The scoring is valid for thermal decomposition products at both 200 and 700°C.

(d) Final scoring and conditions of approval

Each product tested is given a rating of 0 to 170, determined by the sum of all the scores obtained in each of the above tests, the latter being weighted by the coefficients given in the following table:

Oral toxicity	LD 50	1
Irritant effect	eyes	5
	skin	5
Aerosol	cold	2
	hot	2
Thermal decomposition	200° C	1
	700° C	1

Any product which scores 10 before weighting in any one test, or 50 after weighting, is rejected.

4.2.4 TESTING PHOSPHORIC ESTERS FOR NEUROTOXIC EFFECTS

The investigations are carried out on chickens (minimum age 10 months) kept in pens allowing them free movement. Birds with abnormalities in their mode of walking are not suitable for the experiment.

The fluid under test is diluted with peanut oil and applied to 6 chickens, using a probang. The dose is 10g/kg, given twice a day for three days, and this is followed by observation and weight monitoring period of 21 days.

During the observation period any walking defects are carefully noted. If the test is positive the birds display ataxia, walking difficulties, and loss of equilibrium.

The birds displaying positive clinical reactions are killed by bleeding, 21 days after application of the test fluid. For the purpose of the histological examination the brain and spinal chord with anterior horn cells are extracted and fixed for 48 hours in 10% formol. Then cross-sections are made through the cervical, thoracic and lumbosacral sections. Coloration is by the Marchi method, with the Lillie modification. The sections are dried and set in gelatine and frozen in sections (10 - 15 μ). A yellow-orange colour reaction indicates demyelination. After 21 days the birds displaying negative clinical reactions receive a repeat of the initial dose and undergo a further 21-day observation period. If motor disturbances still do not become evident, the fluid under test may be regarded as not neurotoxically harmful.

A control sample of six similar birds is kept. Over the same period, in similar conditions.

In order to test for clearly positive reactions a further control group is given a single oral dose of 0.5 g/kg triorthocresyl phosphate.

If two of the six chickens which have received the injection show positive clinical or histological reaction (excluding the case of concurrent neurolymphomatosy), the liquid is regarded as neurotoxic, and other tests are not necessary.

INTRODUCTION TO PARTS V, VI and VII

DEFINITIVE NOTE

This part of the Specification deals with British fire resistant fluids used for power transmission underground and on the surface, and the testing procedures which are applied to them.

Although it covers the most important of these requirements, it should be noted that the suitability of any fluid for a particular application would subsequently be assessed by experience in the actual equipment concerned.

PART V - GENERAL

(For fire resistant fluids used in mines, meeting United Kingdom requirements)

GENERAL

5.1 SCOPE

This specification covers fire resistant fluids for use in machinery and hydraulic equipment.

5.2 CLASSIFICATION

Each fluid shall be classified by the prefix Fr followed by a letter followed by a number to indicate respectively that (i) the fluid is fireresistant, (ii) the type of fluid and (iii) its viscosity. The letter in the classification shall be one of the following:

A - emulsions of the oil-in-water type (dilute emulsions) i.e. emulsions containing, typically, 95% water and 5% oil by volume.

B - emulsions of the water-in-oil type (invert emulsions) i.e. emulsions in which the proportion of water, typically 40%, is much less than that in type A emulsions.

C - solutions of glycols in water, including glycol containing small amounts of mineral oil.

D - fluids that do not contain water.

The number following the letter in the classification shall be whichever of the numbers e.g. 32,46,68,100,150 or 200 is nearest to the viscosity of the fluid in centistokes at 47.8°C (100°F) as stated by the manufacturer. (Note: the numbers in the classification follow B.S.S. (B.S. 4236). Viscosity classification for industrial liquid lubricant. In the case of type B fluids (invert emulsions) that are suitable for use at low temperatures, the letters LT shall be added. Thus Fr B46LT would be the classification of a fire resistant water-in-oil (invert) emulsion suitable for low temperature use and having a viscosity in centistokes at 37.8°C that is nearer to 46 than to 32 or 68.

5.3 TECHNICAL FLAMMABILITY CRITERIA

When the fluid is tested in accordance with Part VI.6.15 the flame from the fluid shall not persist for longer than 30 seconds after removal of the igniting flame from the spray. When the fluid is tested in accordance with Part VI.6.16, the mean persistence of flame shall be less than 60 seconds.

5.4 HEALTH CRITERIA

The toxic properties of fire resistant hydraulic fluids should be such that they do not present a significant hazard to health when used in mining machinery.

The use of fire resistant fluids should not result in hazardous pollution of the environment within or outside mines.

The assessment of the health hazard from any particular fluid is dependant on its chemical constituents and on the likely operational exposure of individuals. These points are specified in detail in Part VII.7.2 to 7.5 together with guidance on toxicological testing required for approval by the Competent Authority for the use of the fluid in mines. The Competent Authority for Health criteria shall be a body approved by the Department of Energy for this purpose.

5.5 TECHNOLOGICAL CRITERIA

The performances of fluids in the various tests for technological criteria are specified in Part VI.6.1 to 6.14. Descriptions of the test procedures themselves are given in Part VI.6.15 to 6.22.

5.6 CONTAINERS

5.6.1 Materials

These requirements apply to containers used for emulsifying oils used to make Type A Fluids, and to Types B, C and D fluids. Any container in which the fluid is supplied, together with the associated bungs, caps, seals, labels and other fittings shall not be made from aluminium, magnesium or titanium nor from alloys containing these metals as major constituents. Where the containers or their fittings are made from alloys containing aluminium and/or magnesium and/or titanium as minor constituents, the total content of these three elements shall not exceed 15 per cent of the alloy by weight and the content of magnesium and titanium together shall not exceed 10 per cent by weight.

Note: alloys within these limits include:

Diecasting alloys in accordance with BS 1004[*].
Aluminium bronzes such as AB1-I, AB1-C, AB2-1 and AB2-C in accordance with BS 1400[**].
Copper - manganese - aluminium alloys such as CMA1-1, CMA1-C, CMA2-I and CMA2-C in accordance with BS 1400.
Aluminium brass in accordance with BS 885[***].

The containers and their fittings shall not be painted with aluminium paint. The internal cleanliness of all containers before they are filled shall be of a high standard. Any coating of the internal surfaces of the barrels shall have no deleterious effect on the fluid.

Type B fluids shall be supplied only in new containers.

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[*] BS. 1004: Zinc alloys for diecasting and zinc alloy diecastings.

[**] BS. 1400: Copper alloy ingots and copper and copper alloy castings.

[***] BS. 885: Brass tubes for general purposes.

5.6.2 Marking

Type A Fluids: In the case of emulsifying oils used for making fluids of type A, the containers shall be coloured salmon pink all over (colour reference 447 of BS 381C: Colours for specific purposes) and the information set out below shall be marked clearly and indelibly on the containers:

Description of contents, e.g. No. 19 Superior Emulsifying Oil.

Approval Number.

Manufacturer's Name.

Batch Number.

Month and Year of filling.

Protect from Frost (where applicable).

Types B, C and D Fluids: containers shall be white with a coloured band around the belly to indicate the contents as follows:

Black		for type B fluids
Green	(colour 217 to BS 381C)	for type C fluids
Blue	(colour 175 to BS 381C)	for non-toxic phosphate ester in type D
Yellow	(colour 309 to BS 381C)	for other fluids in type D.

The information set out below shall be marked in black characters on the white background in the location shown in Table 1.

Approval numbers	in characters 500 mm high
Definition (See Part V.5.2) Supplier's Name Batch Number The words 'Put to use before ..'	in characters 25 mm high

Any other markings or colours on the containers shall not cause confusion with those required by this specification.

TABLE 1

Size of Container	Size of Coloured Band	Location of Marked Information
205 litres (45 gal) barrels	100 to 200 mm wide	On the end near the bung and on the side near the bung.
23 litres (5 gal) drums	75 to 100 mm wide	On the side as near the bung as practicable and confined to one half of the circumference.
4.5 litres (1 gal) containers	50 to 75 mm wide	On the side as near to the bung as practicable.

5.7 COLOUR

The colour of the fluid shall be subject to agreement between the manufacturer and the purchaser.

5.8 CONDITIONS OF APPROVAL, INCLUDING TESTS IN SERVICE

As prerequisites to the granting of approval of a particular fluid for use in the mines of the National Coal Board, the fluid must comply with the requirements of NCB Specifications No 463 or 570 as appropriate and in addition must perform to the satisfaction of the National Coal Board in particular installations or test rigs.

Complete copies of the NCB Specifications may be purchased from: Engineering Secretariat, National Coal Board, The Lodge, South Parade, Doncaster, Yorkshire DN1 2 DX, England.

The series of tests to be carried out prior to the granting of approval must be conducted by a competent body, such as, at present, the following:

National Coal Board, Mining Research & Development Establishment, Stanhope.

Bretby, Nr. Burton-on-Trent, Staffordshire.

Department of Energy, Safety in Mines Research Establishment.

Harpur Hill, Buxton, Derbyshire.

5.9 SUMMARY OF REQUIREMENTS

The following table indicates which tests have to be satisfied by fluids of the various types. In the case of an 'X' marking the fluid has to be capable of satisfying requirements when delivered and at the end of its stated storage life. In the case of an 'Y' marking, the manufacturer of the fluid shall state the particulars stipulated in the relevant clause.

Clause Part V or Part VI	Requirement	Fluids of type Fr			
		A	B	C	D
5.3	Spray Ignition Test	-	X	X	X
5.3	Wick Test	-	X	X	X
6.2	Pour Point	-	Y	Y	Y
6.3	Viscosity	X(oil)	Y	Y	Y
6.4	pH	X(water)	-	-	-
6.5	Corrosion	X	X	X	X
6.6	Thermal Stability				
	(a) low temperature cycling	-	X	X	-
	(b) normal ambient temperature	-	X	-	-
	(c) medium temperature	X	X	-	-
	(d) high temperature	-	-	-	X
6.7	Compatibility	X	Y	Y	Y
6.8	Wear Protection	-	-	-	-
6.9	Miscibility	X	-	-	-
6.10	Water Content	Y(oil)	X	X	-
6.11	Shear Strength, Foaming Tendency, De-aeration, Density, Ash Content	-	-	-	-
6.12	Storage Life	-	Y	Y	Y
6.13	Freedom from contamination	X(oil)	X	X	X
6.14	Flash Point	X(oil)	-	-	Y
5.7	Colour	Y(oil)	Y	Y	Y
5.6	Containers, materials	X(oil)	X	X	X
5.6.2	Containers, marking	X(oil)	X	X	X
5.4	Health Criteria	X(oil)	X	X	X

5.10 NOTES ON THE SELECTION AND USE OF TYPE A FLUIDS WHICH COMPLY WITH THE FOREGOING REQUIREMENTS

5.10.1 The specification has been prepared to cover emulsifying oils ('soluble' oils) suitable for adding to water to make emulsions (type A fluids) for use in powered roof supports and for other such hydraulic purposes. When selecting an oil from those which comply with this specification, local knowledge of the conditions of use, both with regard to the nature of the water and the duty in the machinery, must be taken into account.

5.10.2 Classes of Emulsifying Oil

Two classes of oil are covered:

No. 18: ordinary emulsifying oil which is suitable for waters varying between very soft and rather hard.

No. 19: superior emulsifying oil which is suitable for waters which are very hard.

Note: synthetic oils are not included.

5.10.3 Characteristics of Emulsifying Oil

In addition to the requirements summarised in Part V.5.9, the basic mineral oil should have the following characteristics:

(a) it shall be free from deleterious impurities;

(b) it shall not deposit waxes in service;

(c) its anti-foaming properties shall be satisfactory for use in powered roof supports;

(d) it shall contain a sufficient concentration of anionic emulsifying agent together with a coupling agent, or other additive to ensure ready self-emulsifying properties.

5.10.4 Characteristics of Water used for Mixing Type A Fluids

Experience has shown that the general characteristics of the waters for which emulsifying oils are suitable may be summarised as in the following table.

Table 5.10.4

Characteristic	Class of Emulsifying Oil	
	No. 18	No. 19
pH	6 to 9	
Limit for chloride content * Limit for sulphate content *	approx. 200 mg/litre chlorine ion approx. 400 mg/litre sulphate ion	
Total Hardness, as $CaCO_3$ equivalent *	approx. 250 mg/litre	approx. 750 mg/litre

* Note: values higher than those tabulated may be acceptable in particular cases where tests show the stability of the fluid is satisfactory.

The above summary does not necessarily imply that the stability of a type A fluid in service can be guaranteed with all waters of the hardness stated or that the emulsifying oil will be unstable with waters of greater hardness. Experience and local knowledge may enable satisfactory judgement as to performance in service to be made on the basis of the thermal stability test specified in Part VI.6.17, but in cases of doubt it may be necessary for the test to be repeated with the water actually to be used. Where the water is exceptionally corrosive, it may also be advisable to repeat the test for rust preventing characteristics, Part VI.6.5, with the water actually to be used.

5.10.5 Mixing and Use

The emulsion should be used at 5% oil content by volume, (i.e. 5 parts of oil to 95 parts of water), for all normal applications. The oil content should be checked regularly, e.g. by pocket refractometer, and should under no circumstances be allowed to fall below 3%.

Although the oils specified are of the self-emulsifying type it is nevertheless essential to ensure that the added oil is correctly mixed either by an injector or by thorough general agitation.

The fluid should not be used in conjunction with any rubber or compound for which the volume change is outside the limits allowed by Part VI.6.7.

Attention is drawn to the risk of damage to equipment containing the fluid when exposed to frost, e.g. hydraulic rams may contain a residue of fluid following works tests.

PART VI - SPECIFICATION AND TESTING METHODS

(for fire resistant fluids meeting United Kingdom requirements)

6.1 INTRODUCTION

PART VI Sections 6.2 to 6.14 deal with specifications of the fluids.
PART VI Sections 6.15 to 6.23 describe the Test Methods used for determining the requirements summarised in Part V Section 5.9.

6.2 POUR POINT

The manufacturer shall state the pour point of fluids of types B, C, and D as measured by the Test Method of the Institute of Petroleum (IP) No. 15.

6.3 VISCOSITY

Type A Fluids: the kinematic viscosity of the emulsifying oil used for making up fluids of type A shall not exceed 350 centistokes (1,421 Redwood (No. 1) seconds) at 21.1°C (70°F).

Types B, C and D Fluids: the manufacturer shall state the kinematic viscosity in centistokes at 25°C, 37.8°C and 50°C as measured by Test Method IP 71, using U-tube viscometers, type BS/U or reverse flow U-tube viscometers, type BS/IP/RF. Reverse flow type BS/IP/RF viscometers shall be used for all type B fluids (invert emulsions) and other non-Newtonian fluids. The size of viscometer shall be stated and shall be chosen to give the shortest flow time consistent with the specified minimum. When measured at 37.8°C the viscosity shall be within ± 10 per cent of the stated value.

6.4 CONTAMINATION AND pH OF WATER

The water used for forming fluids of type A shall be free from suspended contaminants and the pH of the water shall be within the range 6 to 9.

6.5 CORROSION

Type A Fluids: an emulsion of the oil at a concentration of 2 per cent by volume shall be made up in a 0.05N (2.9 g/litre) solution of sodium chloride in distilled water and subjected to a test in accordance with Test Method IP135/64: Rust Preventing Characteristics - Lubricating Oil, except that approximately 330 ml of this emulsion shall be used in place of the mixture of lubricating oil and water. No rusting or pitting on duplicate steel test specimens shall be produced after 24 hours.

Note: Especially careful surface preparation of the test specimens is essential to avoid any possible confusion between original surface imperfections and any pitting produced in the test. Test specimens (of En 3B steel to BS 970[*]) can be obtained from Stanhope Seta Ltd., Station Road, Chertsey, Surrey.

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[*] BS 970: Wrought Steels - En series

Types B, C and D Fluids: No specific requirements are laid down for general corrosion - preventing characteristics apart from those for fluids of types B and C mentioned below, but the performance of the fluids will be assessed under appropriate conditions. No specific requirements are laid down for rust-preventing characteristics in the vapour phase condition but the performance of the fluid will be assessed under appropriate conditions.

The rust-preventing characteristics in the immersed condition of fluids of types B and C shall be tested for 24 hours in accordance with Test Method IP 135. There shall be no rusting or definite pitting on duplicate test specimens. In this test 330 ml of the fluid alone shall be used instead of a mixture of oil and water.

6.6 THERMAL STABILITY

(a) Low Temperature Cycling

Fluids of types FrB...LT and FrC shall be stable when tested as prescribed in section 6.18.

(b) Normal Ambient Temperatures

Fluids of type FrB shall have a medium term stability at normal ambient temperature (20°C - 2°C) to the extent of satisfying the following requirements when tested in accordance with section 6.18.

(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; and

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

(c) Medium Temperatures

Fluids of type FrA shall be stable when tested in accordance with section 6.17.

Fluids of type FrB shall be stable under medium temperature conditions (70°C) to the extent of satisfying the following requirements when tested in accordance with section 6.19.

(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 the volume of accumulated free water shall not exceed 1 ml.

(d) High Temperatures

Type D Fluids: fluids of type D must withstand high temperature conditions without undue chemical degradation. When tested in accordance with PART 6.20 the fluids shall satisfy the following requirements:

(i) the total acid number shall not exceed the values given in Table 1;

(ii) the percentage increase in kinematic viscosity at 37.8°C (100°F) and 98.9°C (210°F) shall not exceed the values given in Table 1;

(iii) the percentage change in specific gravity shall not exceed the values given in Table 1;

(iv) the wick test results shall satisfy the requirements of clause 2 of PART V.5.3 for flammability criteria.

The thermal stability of the fluid may also be assessed by any other tests which may be considered by the NCB to be necessary and appropriate to the chemical nature of the fluid.

TABLE 1

Temperature of test, °C	Time of test, hours	Total Acid No. Maximum value	Kinematic Viscosity at 37,8°C (100°F) Maximum percentage increase	Kinematic Viscosity at 98,9°C (210°F) Maximum percentage increase	Specific Gravity 15.6°C/ 15.6°C (60°F) 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	288	1.5	3.0	3.0	1.5
93.3°C	432	2.0	4.0	4.0	2.0

6.7 COMPATIBILITY

Effects on packings, seals and other materials.

Type A Fluids: the volume change in nitrile rubber compound to BS 2751[*], grade BA6, shall not be less than 2 per cent and not more than 6 per oent in any single test when two test pieces are immersed in a 5 per cent emulsion (by volume for 168 hours at 70°C in accordance with BS 903[**], part A16. The test pieces shall be cut from 3/16 inch (5mm) sheet and shall be rectangular, 50 mm long by 25 mm wide. The emulsion shall have no deleterious effect of any kind on leather or other seals such as those used in powered supports.

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[*] BS 2751: vulcanised butadine/acrylonitrile rubber compounds

[**] BS 903: methods of testing vulcanised rubber; part A16: Determination of swelling in liquids.

Types B, C and D Fluids: the manufacturer shall state the materials (including other hydraulic fluids and water) used in hydraulic systems, with which the fluid is known to be compatible and the materials with which it is known to be incompatible.

6.8 WEAR PROTECTION

Tests are carried out in the laboratory in types of equipment for which the fluids are intended. It is expected that laboratory scale tests will be specified as a result of this work.

6.9 MISCIBILITY

Type A Fluids: a type A fluid shall mix freely with any other fluid of the same make and type which has been in normal service use.

Note: In view of the variability of compounds within grade BA6 it is recommended that the rubber compound for this test be obtained from one supplier, namely Messrs. Metropolitan Flexible Products, Queen Street, Great Harwood, Blackburn, Lancashire, England. The National Coal Board will hold in stock sheets of compound 1 foot square by 3/16 inch thick (305 mm square by 5 mm thick) and oil suppliers should order these from The Head of Approvals Section, National Coal Board, The Lodge, South Parade, Doncaster, Yorkshire, DN1 2DX, England.

6.10 WATER CONTENT

Type A Fluids: the manufacturer shall state the water content of the emulsifying oil.

Type B Fluids: the manufacturer shall state the water content of fluids of type B as a percentage by volume when measured by the method given in PART VI.6.21. When the fluid is tested in accordance with this method the water content shall be within ± 1.5 of the stated percentage value.

The manufacturer shall state the chemical composition and concentration of any substance in the fluid which may lead to a difference between the True Water Content and the Apparent Water Content as measured in accordance with PART VI.6.21. In such cases the True Water Content shall be found from the Apparent Water Content and from a measurement of the concentration of water in the distillate.

Type C Fluids: the manufacturer shall state the water content of fluids of type C as a percentage by volume when measured in accordance with PART VI.6.22. When the fluid is tested in accordance with this method the water content shall be within ± 2 of the stated percentage value.

6.11 SHEAR STRENGTH, FOAMING TENDENCY, DE-AERATION, DENSITY AND ASH CONTENT

No requirements for these properties are contained in this specification.

6.12 STORAGE LIFE

6.12.1 Types B, C and D Fluids: the manufacturer shall state the storage life of the fluid under the conditions stated below.

The storage life shall apply to fluid stored in the sealed containers in which it is delivered. In the case of fluids of type B the storage life shall be not less than 6 months. In the case of fluids of types C and D the storage life shall be not less than 2 years. At the end of the storage life the fluid shall be capable of satisfying all appropriate requirements as indicated in the Summary of Requirements, PART V.5.9.

6.12.2 Notes on Storage

The containers holding fire resistant fluid should be stored in cool dry conditions indoors at a temperature not less than $4^{\circ}C$ ($40^{\circ}F$).

Any instructions by the supplier must be followed. The oil for Type A fluids is not fire resistant before it is mixed with water and due precautions must be taken.

Every effort must be made to put fluids of types B, C and D to use before the date marked on the container. Where there is fluid remaining in the container after this date it should not be used until approval has been given by a competent person that the fluid is satisfactory with particular reference to deterioration such as separation or contamination from rusting on the inner surface of the container, etc.

6.13 FREEDOM OF FLUIDS FROM CONTAMINATION

Fluids shall be free from contaminants. The purchaser shall have the right to carry out tests to determine the nature and quantity of any contamination.

6.14 FLASH POINT

Type A Fluids: the emulsifying oil before mixing with water shall have a closed flash point not less than $65.6^{\circ}C$ ($150^{\circ}F$).

Type D Fluids: the manufacturer shall state the closed flash point as measured by the Institute of Petroleum Test Method I.P. 34.

6.15 METHOD OF TEST FOR FIRE RESISTANT PROPERTIES BY MEANS OF THE SPRAY IGNITION TEST

6.15.1 Preparation of Samples for Test

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.

An 1100 ml sample shall be placed in a 1500 ml glass beaker and heated to the test temperature by means of an electrical immersion heater at a rate of temperature increase of 3°C ± 1/2°C/min. The surface area of the immersion heater shall be not less than 170 cm^2 and the fluid shall be mechanically stirred during heating (a two blade stirrer, having a diameter of 40 mm and running at 400 rev/min is satisfactory).

For water-based fluids or emulsions containing water, the test temperature shall be 65°C, and 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 shall be made of any such change.

6.18.2 Test apparatus

The fluid, heated to the test temperature, shall be sprayed from a 2.5 U.S. gal/h[*] at 100lbf/in^2, (or 689/KN/m^2) 80° hollow-cone spray oil-burning nozzle type F.80 supplied by the Monarch Manufacturing Co., c/o Perfection Parts, 59 Union Street, London, S.E.1, at a spraying pressure of 6.89 MN/m^2 (1000 lbf/in^2) maintained throughout the test 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 21 kN/m^2 (3 lbf/in^2) or 'Saffire' lightweight model with a No. 10 nozzle with both gases at 28 kN/m^2 (4 lbf/in^2) supplied by British Oxygen Co. Limited).

The test shall be carried out in an enclosure with a forced draught in the direction of spray propagation. The diameter of the enclosure downwind of the apparatus should be not less than 1.22 m (4ft) and the arrangement such that the spray is visible along its whole length. The fixed ventilation should be such as to carry fumes free of the operator, and a velocity of 0.4 to 0.5 m/s (80-100ft/min) is recommended.

6.15.3 Test 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 final test temperature, as described in PART VI.6.15.1 it shall immediately be transferred to the pressure vessel and the test carried

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[*] 2.5 U.S. gal/h = 9.4625 litres/hour

out. When the spray is established, the igniting flame shall be repeatedly applied to and withdrawn from different positions along the length of the spray and the maximum duration of burning after withdrawal of the igniting flame noted.

6.15.4 Acceptance criteria

To pass this test, the spray must not continue to burn for more than 30 seconds after removal of the igniting flame.

6.16 METHOD OF TEST FOR FIRE RESISTANT PROPERTIES BY MEANS OF THE WICK TEST

6.16.1 Summary of method

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.

6.16.2 Sampling

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

6.16.3 Apparatus

(a) Woven asbestos tape of nominal width 25.4 mm (1 in.) and nominal thickness 0.38 mm (0.015 in.) complying with B.S. 1944:1953 and approved by the National Coal Board. Approved tape will normally be kept available by the National Coal Board for purchase by recognised fluid suppliers only. Orders for tape shall be for one or two coils (not more as each coil contains about 36 yards or 33 meters of tape) and should be sent to Purchasing and Stores Department, N.C.B. Mining Research and Development Establishment, Ashby Road, Stanhope Bretby, Burton-on-Trent, Staffordshire. No despatch will be made until an official order has been received.

(b) A reservoir as shown in Figure 1 PART VI.6.16.6. The reservoir shall be 25 mm (1 in) wide and 19 mm (0.75 in.) deep. 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 (7 in.). Each clip shall have a reference mark at a height of 28.5 mm (1.125in) above the base of the reservoir.

(c) A burner equipped with a B.O.C. No. 1 welding nozzle. The burner may be fabricated from a length of tube, one end of which is threaded to accomodate the nozzle as shown in Figure 2, PART VI.6.16.6 The burner shall be connected by means of flexible tubing to a cylinder of liquid propane via 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 bunsen burner jet. The flame at the nozzle is not aerated.

(d) A testing jig shown in Figure 2. 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 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.

(e) A soaking bath having the approximate dimensions 30 mm (1.25 in.) wide, 200 mm (8.0 in.) long and 40 mm (1.75 in.) deep.

(f) A stop watch.

6.16.4 Wick preparation

Each wick shall be 190 mm (7.5 in.) long and its edges shall be free from knots. Each wick shall be suspended in still air with its long edges vertical and shall be ignited by applying a small flame to its lower edge. The flame shall be allowed to spread vertically so that the whole of the wick is burnt free of combustible material. It is recommended that fifteen wicks should be prepared. No wick shall be used for test purposes until at least 1 hour after preparation.

6.16.5 Adjustment of testing 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 is in the igniting position the axis of the burner nozzle is horizontal and level with the top of the wick to within 1.5 mm (0.06 in.) (see PART VI.6.16.Fig.3). The horizontal distance between the tip of the nozzle and the top of the wick shall be 6.5 ± 1.5 mm (0.25 ± 0.06 in) as shown in Fig. 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.

6.16.6 Flame height

With the torch in the setting position the igniting flame shall be adjusted to a height of 25.5 ± 1.5 mm (1.00 ± 0.06 in.). The flame height should be checked between tests and adjusted as necessary.

6.16.7 Test conditions

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 manipulation of the apparatus.

The tests shall be carried out at a temperature of $20^\circ \pm 2^\circ C$.

6.16.8 Procedure

The sample of fluid shall be thoroughly mixed and the soaking bath filled to a depth of approximately 30 mm (1.25 in.). A batch of at least five wicks shall immediately be immersed in the fluid in the soaking bath. The wicks 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 but 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 and 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 (0.50 ± 0.06 in.) 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 (0.75 in.) 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 wick 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 second.

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 burnt portion of the wick does not extend more than 20 mm (0.75 in.) from the point of ignition two further tests may be carried out on that wick edge, applying the igniting flame at positions 50 mm (2 in.) from the mid point of the wick edge. Should any residual flame on the wick traverse a previously burnt 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 time 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.

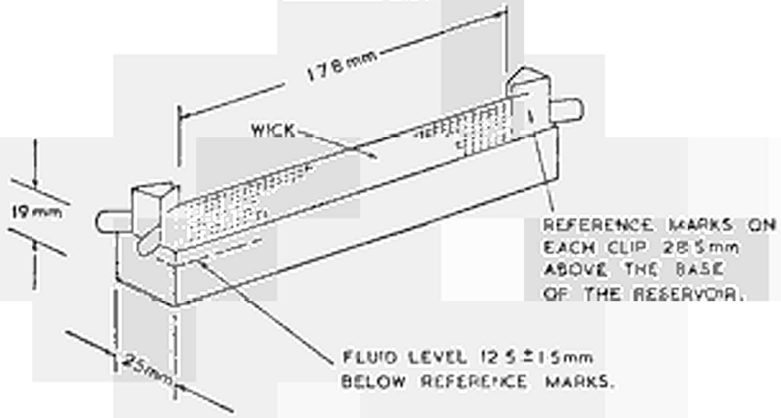

Fig. 1 Reservoir

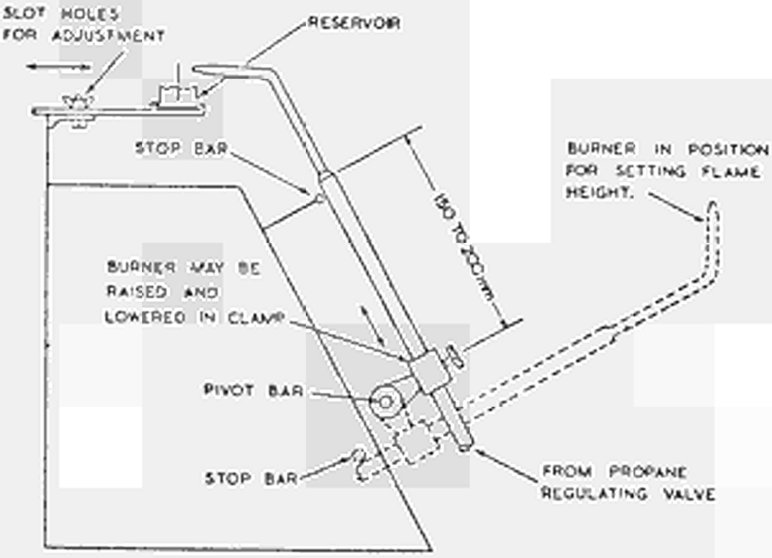

Fig. 2 Test rig

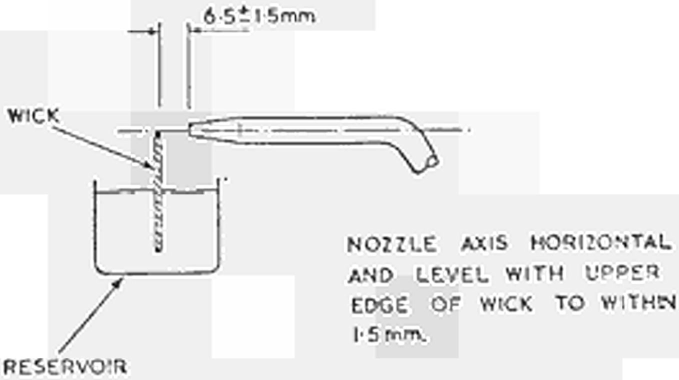

Fig. 3 Burner nozzle in igniting position

6.16.10 Acceptance Criteria

To pass this test the mean persistance of flame shall not be more than 60 seconds.

6.17 METHOD OF TEST FOR STABILITY OF OIL-IN-WATER EMULSIONS (TYPE FrA FLUIDS) AT MEDIUM TEMPERATURE

An emulsion of the oil at a concentration of 5 per cent by volume shall be made up with one of the following solutions depending on the classification of the oil. (Soluble oils designated No. 18 are ordinary emulsifying oils which are suitable for use with water varying between very soft and rather hard. Soluble oils designated No 19 are superior emulsifying oils which are suitable for use with very hard waters).

No 18 Oil: solution of concentration 308 mg/litre $MgSO_4$. $7H_2O$, 215 mg/litre $CaSO_4$. $2H_2O$ and 330 mg/litre NaCl equivalent to 250 mg/litre $CaCO_3$ and 200 mg/litre chlorine ion.

No 19 Oil: solution of concentration 924 mg/litre $MgSO_4$. $7H_2O$, 645 mg/litre $CaSO_4$. $2H_2$ and 330 mg/litre NaCl equivalent to 750 mg/litre $CaCO_3$ and 200 mg/litre chlorine ion.

Note: Calcium sulphate is difficult to dissolve in water, and even with occasional shaking it may take up to 2 days to dissolve the prescribed amount.

The emulsion shall be put in a flask with a graduated neck to BS 676[*] and of a nominal capacity of 150 or 200 ml. The flask shall be filled to approximately the middle of the graduated scale, stoppered to prevent evaporation, and allowed to stand for 168 hours at a temperature of 70 ± 2°C. No sign of any oil separation, no creaming in excess of 0.1 ml per 100 ml of emulsion, or flocculation or water separation shall occur.

6.18 METHOD OF TEST FOR MEDIUM TERM STABILITY FOR WATER IN OIL EMULSIONS (TYPE FrB FLUIDS) AT NORMAL AMBIENT TEMPERATURES

6.18.1 Summary of method

A 500 ml graduated measuring cylinder shall be filled with 450 ml of emulsion over a 50 ml layer of water. After 1000 hours storage, 50 ml samples shall be pipetted from two defined levels in the upper and lower regions of the emulsion column. The water contents of these samples shall be determined and compared with the measured initial water content. Readings of the surface oil layer volume and the volume of accumulated free water shall also be taken.

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[*] BS 676: Flasks with graduated necks

6.18.2 Apparatus

(a) Test vessels	500 ml unstoppered graduated measuring cylinders to B.S. 604.
(b) Pipettes	50 ml capacity to B.S. 1583 grade A or B. NOTE: Precautions should be taken to guard against the danger from implosions whilst the pipettes are under vacuum.
(c) Vacuum Pump	capable of maintaining 50 kN/m^2 (15 in. of mercury), fitted with a vacuum gauge and a means of adjusting the applied suction.
(d) Sampling Jig	consisting of retort stand and clamps as in figure 1, PART VI.6.18.
(e) Stop clock	
(f) Equipment and reagents for water determination by the Dean & Stark method, IP74.	

6.18.3 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 od 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.

6.18.4 Storage

The cylinders shall stand undisturbed for 1000 hours at a temperature of 20°C ± 2°C. They shall be protected from direct sunlight and draughts and stored at a location remote from sources of heat.

6.18.5 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, i.e. the volume of accumulated free water, after 1000 hours standing time.

6.18.6 Sampling

Samples shall be taken from the cylinders after 1000 hours standing time, using the following procedure:

A 50 ml pipette shall be mounted vertically in clamp A of the sampling jig shown in figure 4. 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 positioned directly beneath clamp B. Clamp B shall be released and the pipette raised and positioned with its stem on the vertical axis of the measuring cylinder.

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 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°C ± 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 Dean & Stark method. The residual fluid shall be rinsed from the pipette into the flask using several charges of water-free petroleum spirit (90° - 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.

6.18.7 Water content measurements

The percentage water content by volume Xs, of each pipetted sample shall be determined to the nearest 0.1 by the Dean & Stark method, as given in Test Method 1P74.

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

6.18.8 Calculating and reporting

The following calculations shall be made:

The mean Xi of the two values of initial water content.

The changes in water content Xd which shall be calculated from Xs - Xi.

The means of the two determinations of Xd 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 1000 hours.
2. The mean change in percentage water content at the 125 ml level after 1000 hours.

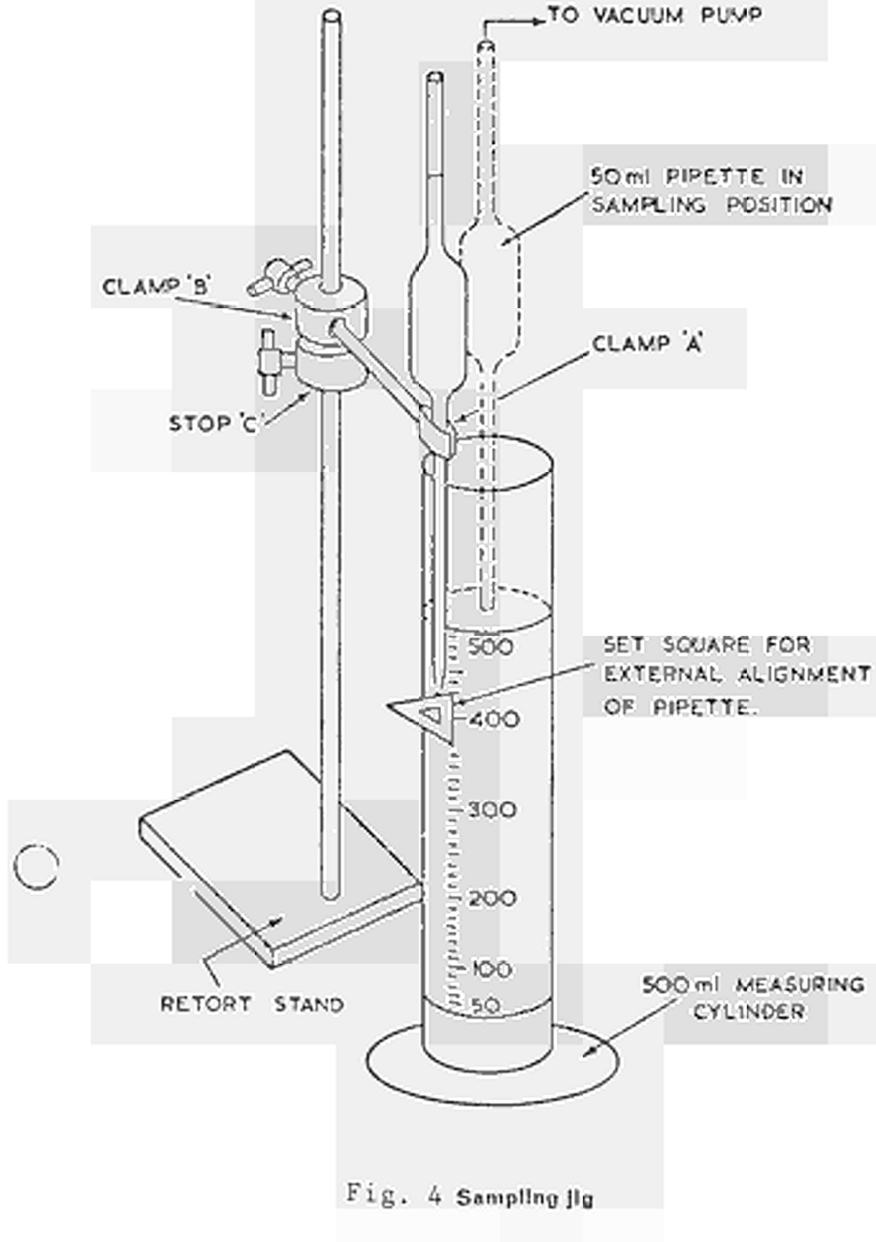

Fig. 4 Sampling jig

3. The mean surface oil layer volume (in ml) after 1000 hours.
4. The mean volume of accumulated free water (in ml) after 1000 hours.

6.19 METHOD OF TEST FOR STABILITY OF WATER-IN-OIL EMULSIONS (TYPE FrB FLUIDS) UNDER MEDIUM TEMPERATURE CONDITIONS

6.19.1 Summary of method

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.

6.19.2 Apparatus

(a) Test Vessels	100 ml Crow receivers to BS 568:1972 Class B.
(b) Polyethylene film	- approximately 0.05 mm thick.
(c) oven	An air oven capable of maintaining a temperature of 70°C within ± 1°C. An oven to BS 2648:1955 is suitable.

6.19.3 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.

6.19.4 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 undisturbed 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.

6.19.5 Expression of 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.

6.20 METHOD OF TEST FOR STABILITY FOR LIQUIDS WHICH DO NOT CONTAIN WATER (TYPE FrD FLUIDS) AT HIGH TEMPERATURES

Summary of Method

Samples of the fluid will be subjected to heat ageing for various periods of test at three different temperatures of test. Air is bubbled through the fluid during the period of test.

Various fluid properties are measured before and after the heat ageing treatments.

6.20.2 Sampling

A sample of not less than 10 litres of fluid shall be supplied for test. Care should be taken that this sample is representative of the bulk.

6.20.3 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 fig. 1, PART VI.6.20 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 cm^3/min ($\pm$ 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.

6.20.4 Procedure

The following procedure shall be carried out for each of the test conditions given in table I PART VI.6.20 using a fresh sample of fluid for each test condition.

The heating vessel shall be filled with 850 ml of fluid. The thermometer and air nozzle shall be positioned as shown in fig. I PART VI.6.20 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 cooling water shall be circulated through the condenser.

The fluid shall then be heated to the temperature of test, using the heating mantle. On reaching the temperature of test, 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 with $\pm$ 2°C of the temperature of test for the period of test by the operation of the temperature controller (see note 1). At the end of the period of test the heating and air bubbling shall be discontinued and the fluid allowed to cool naturally to room temperature.

The tests described below for determinations of fluid properties shall then be carried out on the fluid.

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 temperature of test is reached. This reduction in power will also assist in reducing the temperature oscillations which may occur if an on - off type of controller is used.

6.20.5 Determination of Fluid Properties

The following fluid properties shall be measured for a samle of untreated fluid and for the samples of fluid subjected to heat ageing for all of the test conditions given in Table I,PART VI.6.20.

1. Total Acid Number by potentiometric titration, method IP 177/64.* (or, if appropriate, Total Base Number by potentiometric perchloric acid titration, method IP 276/71 T.*
2. Kinematic viscosity at 37.8°C (300°F) and 98.9°C (210°F) by means of U tube viscometer, Method IP 71/73.*
3. Specific gravity 15.6°/15.6°C (60/60°F) by means of hydrometer, method IP 160.*

Note: * IP - Institute of Petroleum.

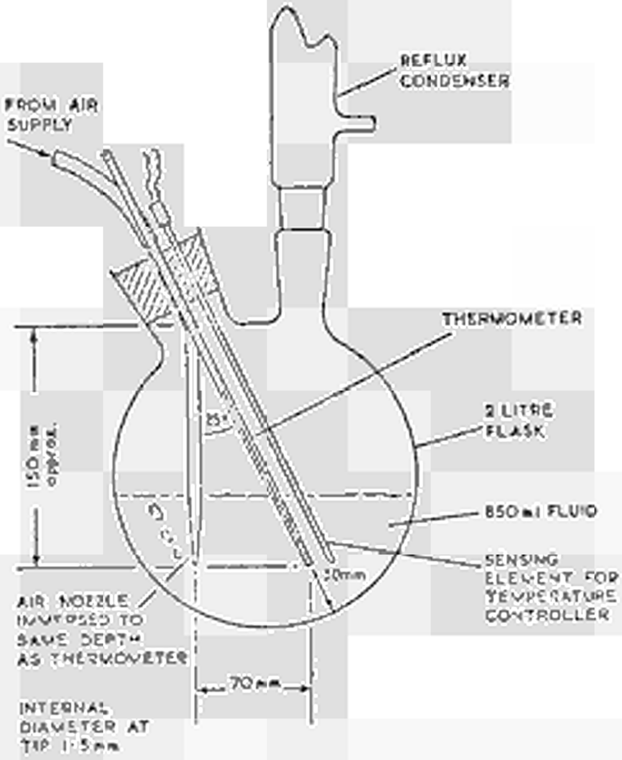

Fig. 1. Apparatus for testing thermal stability of synthetic fluids

The fire resistant properties of the fluid shall be measured for a sample of untreated fluid and for the samples of fluid subjected to heat ageing for the test conditions in Table I which are marked with an asterisk (*). The Wick Test described in PART VI.6.16 shall be used.

6.20.6 Expression of Results

The results of all the determinations required by PART VI, 6.20.5 together with the percentage change in specific gravity and viscosity shall be reported.

Table I

Conditions of Testing

Temperature of Test	Periods of Test hours		
250°C (482°F)	20*	30*	40*
149°C (300°F)	72	144	216*
93.3°C (200°F)	144	288	432*

*See Determination of Fluid Properties, 3. above.

6.21 METHOD OF MEASUREMENT OF WATER CONTENT OF WATER-IN-OIL EMULSIONS (TYPE FrB FLUIDS)

6.21.1 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.

6.21.2 Test method

The sample supplied for test shall be placed in a container having a capacity of at least twice that of the sample and vigorously shaken before extracting sub-samples for test.

The Apparent Water Content shall be measured by the Dean & Stark distillation method. This shall be carried out in accordance with Test Method IP74 except that the distillation shall be continued until the side tube of the receiver is free from condensed droplets and no further increase in volume has taken place in a period of five minutes. It is recommended that a 50 ml sub-sample should be used and a 10 ml receiver with stopcock is also recommended.

For fluids which do not contain any water soluble substance which will be distilled during the Dean & Stark distillation the Water Content shall be the Apparent Water Content. In the case of fluids containing ethylene glycol for example the fractional concentration of water in the distillate may be found from specific gravity measurements. The total distillate from a Dean & Stark distillation is thoroughly mixed and then separated from the carrier fluid. To obtain sufficient distillate it may be necessary to mix this with the distillate from a second Dean & Stark distillation. It is essential that in each case the distillation should be carried to completion and that in each case the total distillate should be thoroughly mixed before separation from the carrier fluid. The specific gravity of the distillate is then measured using a 25 ml capillary-stoppered pyknometer in accordance with Test Method 1P190. The concentration of water in the distillate is then found graphically from measurements of the specific gravities of solutions of ethylene glycol of known concentration. A method based on the refractive index of the distillate is also satisfactory.

6.22 METHOD OF MEASUREMENT OF WATER CONTENT OF TYPE FrC FLUIDS

6.22.1 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.

6.22.2 Summary of Method

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

6.22.3 Apparatus

(a) The titration assembly shown in PART VI.6.22, fig. 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 desiccant. Anhydrous magnesium perchlorate is recommended.

(b) The electrical circuit, also shown in PART VI 6.22 fig. 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. An 'Agla' syringe is recommended.

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

6.22.4 Reagents

(a) Karl Fischer reagent - with a water equivalent of approximately 5 mg wager/ml. Karl Fischer reagent modified, supplied by Hopkin & Williams Ltd. is recommended.

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

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 10 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 seconds.

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: 10 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.

6.22.5 (b) Determination of Water Content

10 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 water glycol (Type FrC) 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.

6.22.6 Reporting

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

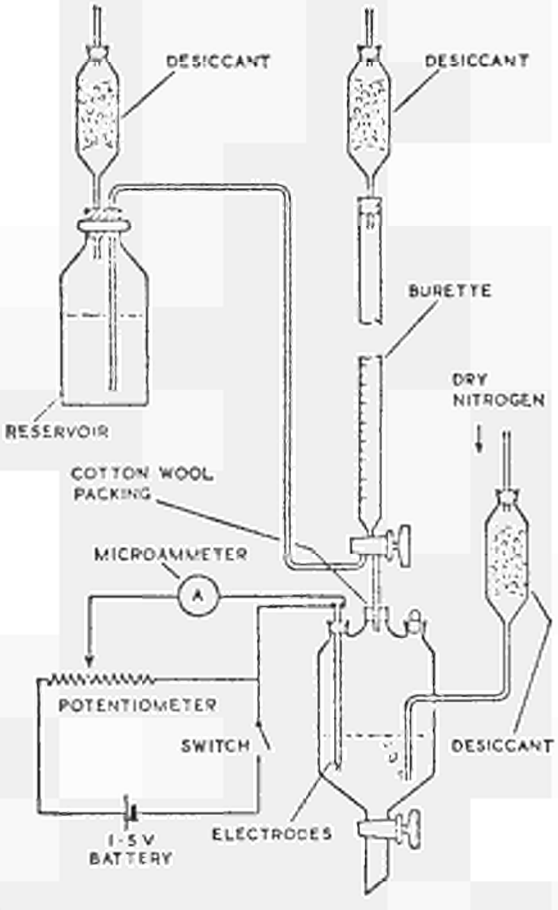

Fig. 1 Apparatus for measurement of water content of Type Fr. C Fluids

PART VII - SPECIFICATION AND TESTING METHODS RELATING TO HEALTH HAZARDS

(for fire resistant fluids for use in mines meeting United Kingdom requirements)

7.1 OPERATIONAL HEALTH HAZARD - GENERAL COMMENTS

Fire resistant fluids used in mining machinery may result in the following:

1. Short term exposure of the skin and mucous membranes to fluid or fluid mist (which may be at the ambient or higher temperatures) with subsequent acute irritation or damage to these tissues.

2. Short term exposure of the eyes to the fluid or fluid mist with subsequent corneal or conjunctival irritation or injury.

3. Continuous or frequent exposure of the skin or mucous membranes to fluid over long periods of time with subsequent irritation of the skin or the development of allergic antization reactions.

4. Inhalation of high concentration of the fluid mist (which may contain thermal decomposition products) for short periods or of low concentration of fluid mist for longer periods (eg. 1 - 2 hours) with subsequent irritation or injury of the upper respiratory tract, the tracheobroncheal tree or lung tissue.

5. Percutaneous absorption of fluid or of one or more components of the fluid to an extent sufficient to cause damage to internal organs or systems or accumulation in the body tissues.

6. Absorption of fluid or one or more components of the fluid following its ingestion, with damage to the gastrointestinal tract or internal organs and systems or with undesirable accumulation in body tissues.

7. Contamination of the environment inside and subsequently outside the mines. This is likely to happen only with substances which are not easily degradable chemically or biologically.

7.2 COMPOSITION OF FLUIDS

A satisfactory specification of the composition of the fire resistant fluid must be made available to the Competent Authority[*] by the manufacturer or supplier. All such fluids for use must comply with the specification. Toxicological studies must be carried out on this fluid. The specification should include:

1. The chemical name of the major and minor components and of principal chemical impurities.

2. The percentage composition.

3. Where the exact composition cannot be given the substances used in the manufacture and the manufacturing methods should be stated.

4. Chemical and physical data including chemical assay methods and spectral data sufficient to characterise the fluid and to allow comparison between batches.

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[*] See PART V.5.4.

7.3 ASSESSMENT OF HEALTH HAZARD

The Competent Authority[x] will assess the likely health hazards involved in using the fluid after

1. Reviewing the available toxicological data on all ingredients and chemical impurities in the fluid or formed in the fluid by interactions of the components by heating or by thermal decomposition in the presence of air.

2. Examining the results of any toxicological tests of the fluids and its reaction and decomposition products which it has found necessary to perform because of inadequate data.

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

A general outline of the test methods to be used in assessing health hazard is given in PART VII.7.5.

7.4 QUALITY CONTROL TESTING OF APPROVED FLUIDS

When a fluid has been accepted provisionally or finally for use in machinery in mines checks should be carried out at appropriate intervals to ensure that it complies with the specification for that fluid required by PART VII.7.2.

Generally chemical and physical methods can be used but sometimes a biological test suitable to the fluid may have to be devised.

7.5 METHODS OF TEST TO DETERMINE THE HEALTH HAZARD

It is recognised that satisfactory results from tests on fluids cannot guarantee freedom from ill effects to all men exposed to them. It is not possible to safeguard the occasional person who may suffer idiosyncratic reactions to chemical substances but it is possible by testing in accordance with the following guidelines to provide an acceptable measure of safety. Approval of the particular fluid can however only be provisional until a satisfactory period of freedom from health hazard has been demonstrated during a long period of use under operational conditions. During the period of provisional approval all cases of ill health which might be attributed to exposure to the fluid should be reported to the Competent Authority.

Toxicity tests needed to assess health hazards have to be designed in detail for a particular chemical substance under investigation. Because of this, toxicological protocols for fluids used in mining machinery are not specified in detail here. Expert toxicological advice should therefore be sought by manufacturers and if necessary guidance sought from the Competent Authority. It is emphasised that all aspects of the toxicology of individual fluids may not need experimental investigations; assessment may be possible

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[x] See PART V.5.4.

from present knowledge of the chemical and physical properties and the biological behaviour of components and impurities. The following notes are intended to assist in the design of those tests which may be needed; they should not be regarded as a rigid plan but only as a guide to be modified for each fluid investigated.

7.5.1 Systematic toxicity

This can be investigated by carrying out suitable haematological chemical and functional tests and by careful macroscopic and microscopic examination of their organs and tissues at autopsy of animals to which the fluid has been administered orally percutaneously or intraperitoneally.

7.5.1.1 Acute toxicity

The study should establish the approximate LD 50 value for the oral intraperitoneal and percutaneous routes in at least two common species of laboratory animal (eg. the method of Thompson W R and Weil C S - Biometrics 1952 8 51 or some other comparable method). Although the LD 50 figure is useful for comparative purposes it is equally important to determine the time course and characteristics of the poisoning, noting behavioural changes and also the pathological findings at autopsy of animals which died during treatment and of those which survived treatment. The study should make it possible to form an opinion as to the mode of action of the substance tested.

7.5.1.2 Percutaneous Toxicity

An unused batch of fluid should be examined for percutaneous toxicity (eg the method of Noakes, D N, and Sanderson, D.M. - Brit. J. Indust. med. 1969, 26, 59). Both local and systemic effects should be looked for. The relationship between the acute oral or parenteral LD50 value and the acute percutaneous LD50 is useful in indicating how readily uptake occurs through the skin. More prolonged tests may be needed as many tests on fluids which have been in use over a considerable period of time (where it is suspected that decomposition products may be formed).

7.5.2 Inhalation toxicity

In investigating this aspect an apparatus has to be designed to produce mists of the same particle size and composition as occurs when the fluid is used under operational conditions. Mists produced by simple aerosol devices may be sufficient for preliminary investigations which may be carried out by methods such as those described in the following publications and papers.

a. Fifth report on Specification and testing conditions relating to fire-resistant fluids used for power transmission. Commission of the European Communities 1975.

b. Draize, J.H. Appraisal of the safety of chemicals in foods, drugs, and cosmetics (1959). Association of Food and Drug Officials of the U.S.A.

c. Gage, J.C. Brit.J.Indust.Med. 27, 1, 1970.

d. Calandra, J.and Kay, J A. Proc. Sci. Sect. T.G.A. 30, 41, 1958.

Animals should be exposed to several suitable concentrations of cold and hot aerosols and where appropriate to aerosols containing thermal decomposition products for short (a few minutes) and long (a few hours) periods and also the effect of repeated exposures should be examined. Behavioural changes and the histopathological effects on the whole respiratory tree should be noted. The time taken to recover from ill-effects produced should be noted.

7.5.3 Skin and eye irritancy

The degree of irritancy following single and repeated application to the skin should be investigated. The irritancy to the mucosal membranes and conjunctival sac may also need testing including whether corneal anaesthesia occurs. Suitable tests will be found in the following papers and publications.

a. Draize, J H: Appraisal of the safety of chemicals in foods, drugs and cosmetics (1959) Association of Food and Drug Officials of the USA.

b. Federal Register 21CFR parts 191.11, 29, 13009, 1964.

c. Federal Register 37 8534, 1972.

d. Federal Register 37 27635, 1972.

e. Wolven, A and Levenstein, I.J.Soc.Cos.Chem. 18, 199, 1967.

f. Schmid, O.J.Soc.Cos.Chem. 21, 835, 1970.

g. Elliott, G.E. and Gray, E, Toxicol.Appl.Pharmacol. 16, 362, 1970.

h. Finkelstein, P, Laden,K, and Miechowski, W.J. Invest.Derm. 40, 11, 1965.

i. Geddes, I.C., Brit.J.Anaesth. 27, 609, 1955.

j. Burton, A.B.C., Fd.Cosmet.Toxicol. 10, 209, 1972.

k. Beckley, J.H., Russel, T.J., and Rubin, L.F. Toxicol.Appl. Pharmacol. 15, 1, 1969.

7.5.4 Allergic sensitisation

The possibility that this aspect may need investigating may be suggested by the formula of one of the fluid components by previous experimental findings or by the experience of those in contact with the fluid during its manufacture. Suitable tests will be found in the following papers and publications.

a. Draize, J.H. The appraisal of the safety of chemicals in foods, drugs and cosmetics, (1959) Association of Food and Drug Officials of the U.S.A.

b. Landsteiner, K, and Jacobs, J, J.Exp.Med. 61, 643, 1935.

c. Buchler, E.V., Arch. Derm. 91, 171, 1965.

d. Stevens, M.A. Brit.J.Indust.Med. 24, 189, 1967.

e. Magnussen, B. and Kligman, A.M. J.Invest.Derm., 52, 268, 1969.

7.5.5 Other investigations

In some cases knowledge of the metabolism of components of the fluid may be needed in order to assess their potential hazard. Other aspects of the toxicity are less likely to need review or experimentation but the need for them will depend on the composition of the fluid and knowledge of the biological activity of chemically related compounds. For example, one commonly used fluid has constituents related to chemicals causing serious neurological injury and investigation of delayed neurotoxic changes were indicated. If any component in the fluid might be carcinogenic this aspect can be investigated by suitably designed long term tests (eg Appraisal of the Safety of Chemicals in Food, Drugs and Cosmetics - Draize J.H.and Hagan O.G. 1959 and W H O Technical Report Series No 220, 1969).

PART VIII - LIST OF EXPERTS

1. TECHNICAL EXPERTS

Dipl.-Ing. E. BREDENBRUCH (Chairman)
Leiter der Hauptstelle für das Grubenrettungswesen
des Steinkohlenbergbauvereins
D - 43 Essen-Kray
Schönscheidtstr. 28

G. BLANPAIN
Ingénieur de recherches
Laboratoire du Centre d'Etudes et Recherches
des Charbonnages de France à Verneuil-en-Halatte
BP 27
F - 60 Creil

R. PLOUCHARD
Ingénieur des Mines
Chef du Laboratoire Lubrifiant
F - 59 Sin-le-Noble/Nord

Prof.rer.nat. H.W. THOENES
Direktor in Rheinisch-Westfälischer
Technischer Überwachungsverein E.V.
D - 43 Essen
Steubenstrasse 53

Dipl.-Ing. K. GRUMBRECHT
Abteilungsleiter in
Versuchsgrubengesellschaft mbH
D - 46 Dortmund
Tremoniastr. 13

C. FRENAY
Ingénieur en Chef
Directeur des Mines
Administration des Mines, Div. de Liège
Avenue Rogier 10
B - 4000 Liège

J. BRACKE
Ingénieur principal divisionnaire
Institut National des Industries extractives
60 Rue Grande
B - 7260 Pâturages

Ir. VAN BLARICUM
Staatsteozicht op de Mijnen
Apollolaan 9
Heerlen (L.) Nederland

I HOWIESON
H M Deputy Principal Inspector of Mechanical Engineering
H M Inspectorate of Mines and Quarries
Department of Energy
Thames House North
Millbank
London S.W. 1

J.B. HALL
Chief Mechanical Engineer
The National Coal Board
The Lodge
Doncaster Yorks. DN.1. 2DX

D.G. WILDE
Principal Scientific Officer
The Safety in Mines Research Establishment
Department of Energy
Harpur Bill
Buxton, Derbyshire SK17 9SN

2. MEDICAL EXPERTS

Prof. Dr med. MALORNY
Director des Pharmakologischen Instituts der Universität Hamburg
D - 2 Hamburg
Martinistr. 52

Prof. Dr med. BENTHE
Pharmakologisches Institut der Universität Hamburg
D - 2 Hamburg
Martinistr. 52

Prof. Dr med. C.A. PRIMAVESI
Hygiene-Institut des Ruhrgebietes
D - 465 Gelsenkirchen
Rotthauserstr. 19

Dr AMOUDRU
Médicin-Chef des Charbonnages de France
9 Avenue Percier
F - 75 Paris 8e

Dr LEBOUFANT
Chef du departement Physique-Biologie
Laboratoire du Centre Etudes et de Recherches des charbonnages de France
Verneuil en Halatte (Boite Postale 27)
60 Creil (Oise)

Dr J.C. MARTIN
Medecin
Laboratoire d'histopathologie
Laboratoire du centre d'etudes et recherches des
charbonnages de France
Verneuil en Halatte (Boite Postale 27)
60 Creil (Oise)

Dr J.S. MCLINTOCK
Chief Medical Officer
The National Coal Board
Hobart House
London SW 1

Dr M. SHARRATT
Senior Medical Officer
Division of Environmental Health and Chemical Hazards
Alexander Flemming House
Elephant and Castle
London SE 1 6BY

OFFICE FOR OFFICIAL PUBLICATIONS OF THE EUROPEAN COMMUNITIES
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