COMMISSION OF THE EUROPEAN COMMUNITIES

MINES SAFETY COMMISSION

FOURTH REPORT

on

specifications and testing conditions relating to

fire-resistant fluids used for power transmission

Luxembourg, 26 March, 1971

(This report supersedes the third report dated 10 October, 1967)

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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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(Annexed to this report is a list of the names of the members of the group of experts, the "Mine Fires" Working Party and the Mines Safety Commission.)

PART I - GENERAL

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.

2. TECHNICAL FLAMMABILITY CRITERIA

The flammability of the fluids is determined by tests a) and b).

a) 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 Annex I.

b) 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 Annex II.

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 Annexes XVII and XVIII.

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.

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 Annex III.

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 Annex IV.

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 Annex V.

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 Annex VI. 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 Annex VII.

f) Determination of anti-corrosive power

The anti-corrosive power of fire-resistant fluids is to be determined by the method described in Annex VIII 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 Annex IX (A)

b) for aqueous hydraulic fluids (forms A, B and C), in Annex IX (B).

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 Annex X.

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.

Testing stations usually carry out the tests advocated in Annex XI based on the 4-ball machine and the Vickers pump test bench. Work is proceeding on the development of other methods for testing fatigue in rolling contacts.

k) Determination of foaming tendency

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

The method of determination is described in Annex XII.

l) Determination of emulsion stability

Emulsion stability is determined for forms A and B only by the method described in Annex XIII.

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 Annex XIV.

PART II - SPECIFICATIONS AND TEST CONDITIONS

GENERAL

Article 1 - Conditions of approval

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 (Articles 3 to 7 inclusive and Part III of the report)

   b) long-term tests in normal service (Article 8).

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)
	- Centre d'études médicales minières des Houillères du Bassin du Nord/Pas-de-Calais, Sin-le-Noble (Nord)
Luxembourg	
Italy	
Netherlands	

3. Authorization for the use of these fluids in coal mines is subject to the production of the certificate mentioned in para. 1.

Article 2 - Identification, classification and working temperature ranges

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

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 3	20 to 40	centistokes at + 50 °C
THI 4	50 to 70	centistokes at + 50 °C

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.

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

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 Annexes VIII and XIII.

SPECIFICATIONS AND TEST CONDITIONS

Articles 3 to 6 show the results to be obtained in the laboratory tests listed and described in Article 7 and Part III of the report respectively.

Article 3 - Flammability criteria

1. Flammability of fluid atomized under pressure (Annex I)

The fluid must be given the rating "1" or "2" according to paragraph 4 of Annex I, in five consecutive tests.

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. Flame propagation in a mixture of fluid and coal dust (Annex II)

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.

Article 4 - Health criteria

1. a) The toxicity tests of water-containing products are carried out by the techniques described in Annex XVIII. Water-free products are examined by the techniques described in Annex XVIII.

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

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.

Article 5 - Technological criteria

1. Softening point (Annex III)

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. Viscosities (Annex IV)

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.

3. Vapour presssure (Annex V)

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.

4. pH (Annex VI)

The pH must lie between 7 and 10 inclusive.

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

5. Shear strength (Annex VII)

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

6. Anti-corrosive power (Annex VIII)

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.

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.

Increase in parts insoluble in benzene $\leq$ 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.

8. Behaviour of packings and seals (Annex X)

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.

9. Wear protection (Annex XI)

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

10. Foaming tendency (Annex XII)

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

11. Emulsion stability (Annex XIII)

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.

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.

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.

14. Density (standard method)

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

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.

16. De-aeration power

The de-aeration power is measured at 50 °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.

Article 6 - Tolerances in the measurement of individual characteristics

Methods:

M 1 - Annex I - Combustion of atomized jet over a flame: no tolerance

M 2 - Annex II - Flame propagation: ± 5 mm of the propagation length of the flame

M 3 - Annex III - Softening point: ± 3 °C

M 4 - Annex IV - Viscosities: ± 10%

M 5 - Annex V - Vapour presssure: ± 10%

M 6 - Annex VI - pH: ± 0.1 pH

M 7 - Annex VII - Shear strength: ± 3% of viscosity values

M 8 - Annex VIII - Anti-corrosive power: ± 2 mg/sample

M 9 - Annex IX - Ageing: ± 0.5 mg KOH/g; ± 0.3 pH; ± 0.2% insolubles in benzene

M 10 - Annex X - Behaviour of packings and seals: ± 25% of mean value determined

M 11 - Annex XI - Wear protection: still being determined

M 12 - Annex XII - Foaming tendency: ± 25%

M 13 - Annex XIII - Emulsion stability: ± 50% of the height of the separated layer

M 14 - Annex - Miscibility and compatibility: no tolerance

M 15 - - Water content: not yet determined

M 16 - Ash content: ± 20%

M 17 - Annex XIV - De-aeration power: ± 5 min.

Article 7 - List of analysis and test methods

Tests	Method described in the following annexes of Part	Germany	Belgium	France	Netherlands	U.S.A.
3.1 Spray combustion	I					
3.2 Flame propagation	II					
5.1 Softening point	III					
5.2 Viscosity	IV	DIN 53015 E				
5.3 Vapour presssure	V					
5.4 pH	VI					
5.5 Shear strength	VII					
5.6 Anti-corrosive power	VIII					
5.7 Ageing of water-free fluids	IX(A)	DIN 51587			NEN 3024	ASTMD 943-54
water-containing fluids	IX(B)					
5.8 Behaviour of packings and seals	X	DIN 53521 E				
5.9 Wear protection	XI					
5.10 Foaming tendency	XII					ASTMD 892-63
5.11 Emulsion stability	XIII					
5.12 Miscibility and compatibility		DIN 51582 DIN 51777				
5.13 Water content				T 60-113		ASTMD 1533
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	XIV					
4.2 Toxicological test: fluids containing water (A+C)	XVII					
fluids containing no water (D)	XVIII					

Article 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 waarying 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.

Article 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

ANNEX I

DETERMINATION OF IGNITABILITY OF FLUID ATOMIZED UNDER PRESSURE

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.

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. 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.

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

DETERMINATION OF IGNITABILITY OF FLUID ATOMIZED UNDER PRESSURE

Diagram A

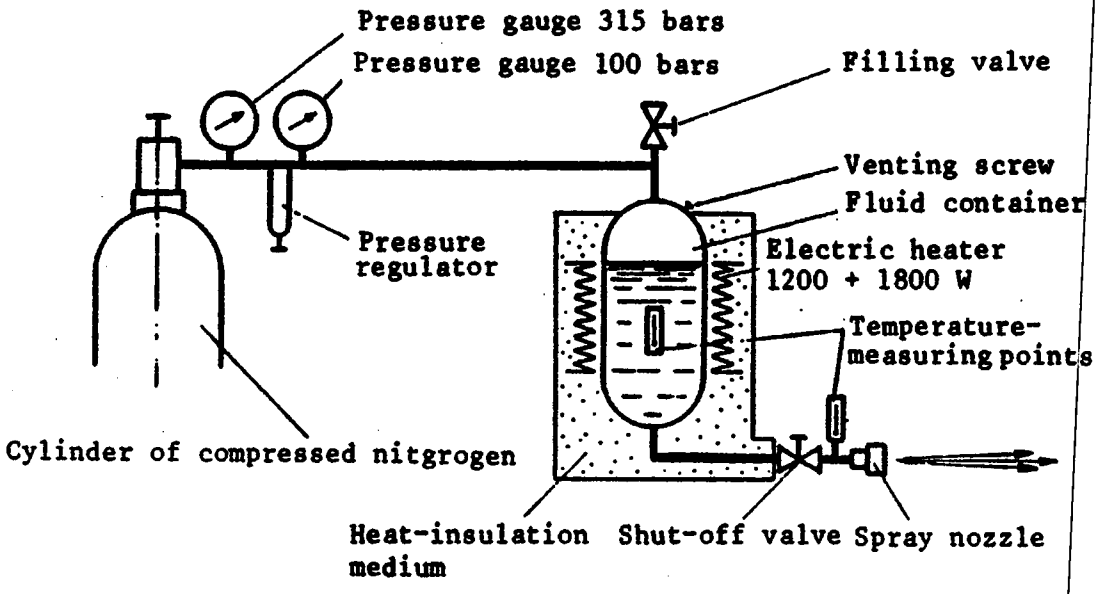
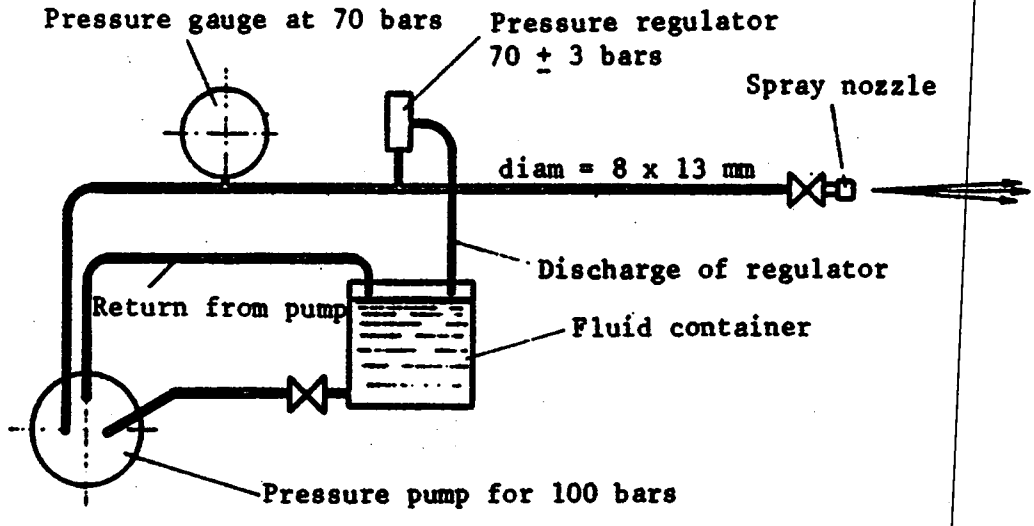


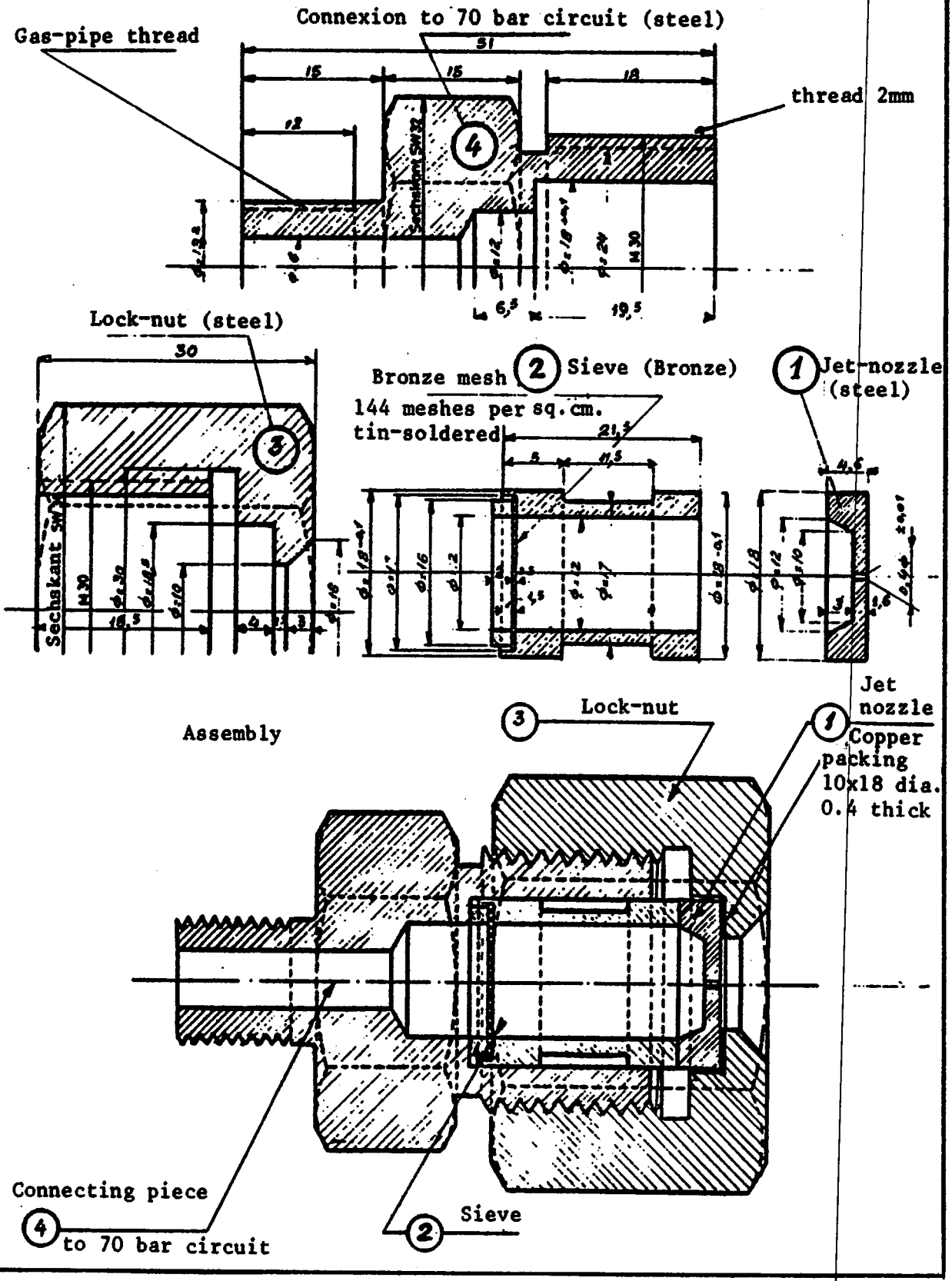
Diagram B



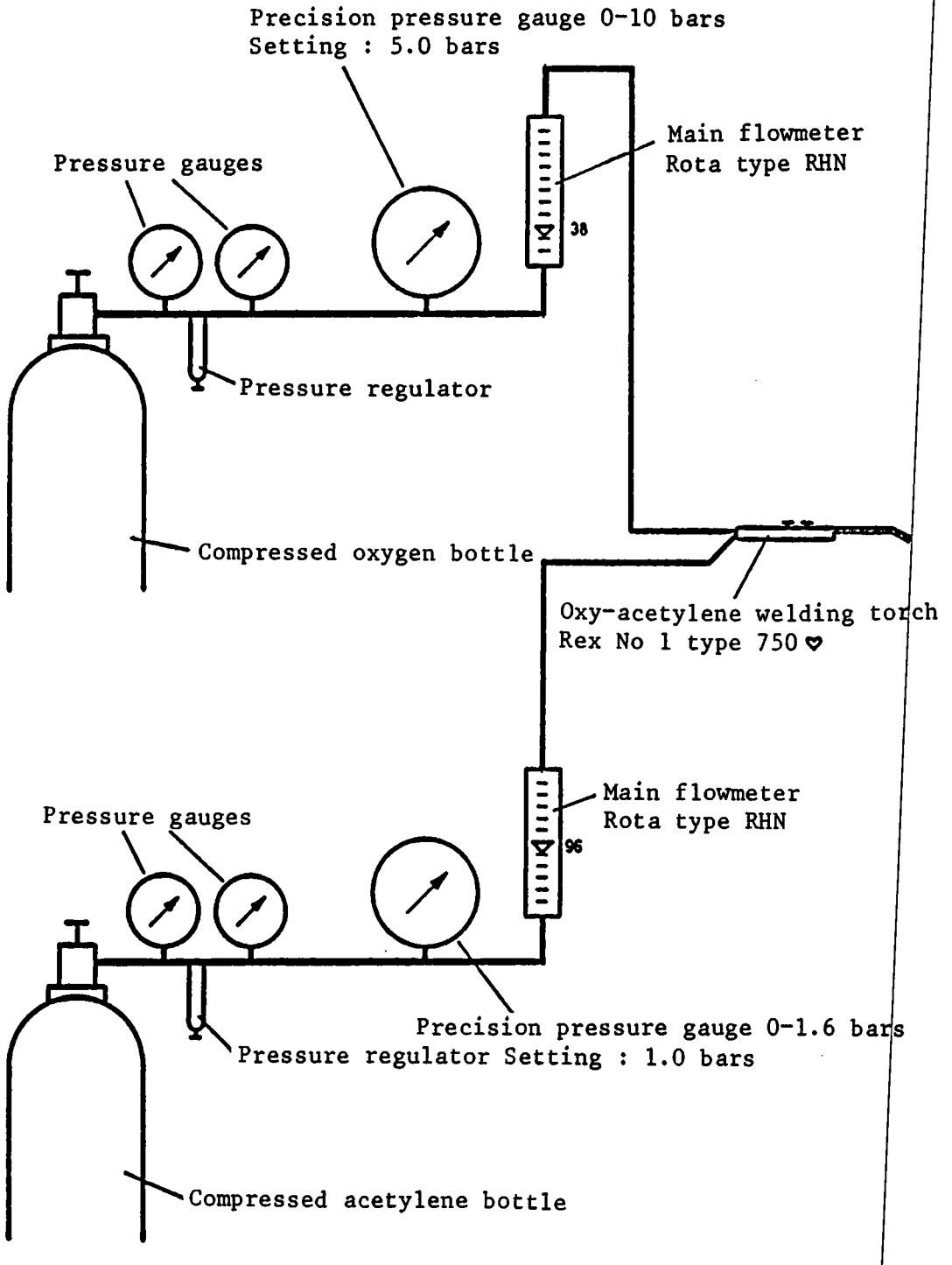
DETERMINATION OF IGNITABILITY OF FLUID ATOMIZED UNDER PRESSURE

Components of test nozzle

Scale 2:1



DETERMINATION OF IGNITABILITY OF FLUID ATOMIZED UNDER PRESSURE



ANNEX II

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

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.

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. 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 %
Ash, dry basis	10.3 % tolerance ± 10 %
Volatile matter, dry basis	27.1 % tolerance ± 10 %
Particle size	0 to 100 mircrons

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

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 in quality 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.

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.

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.

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.

NOTES

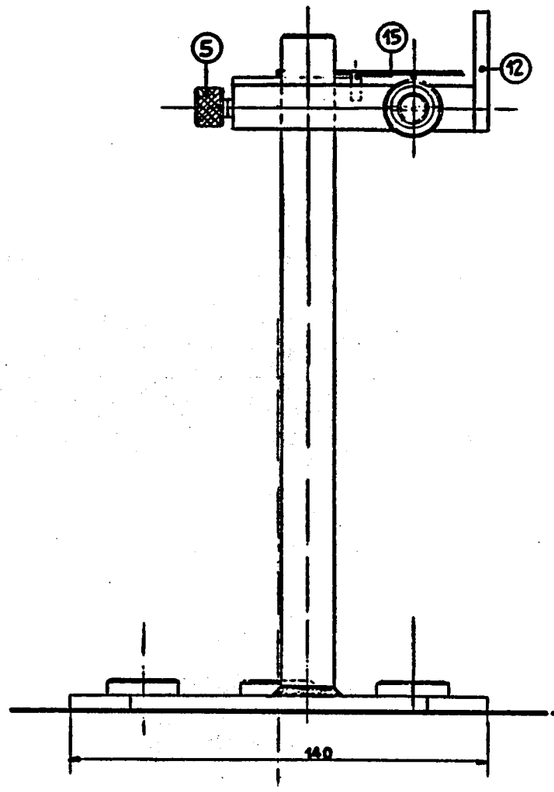
a) In preparing the test piece care must be taken to press the paste uniformly.

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.

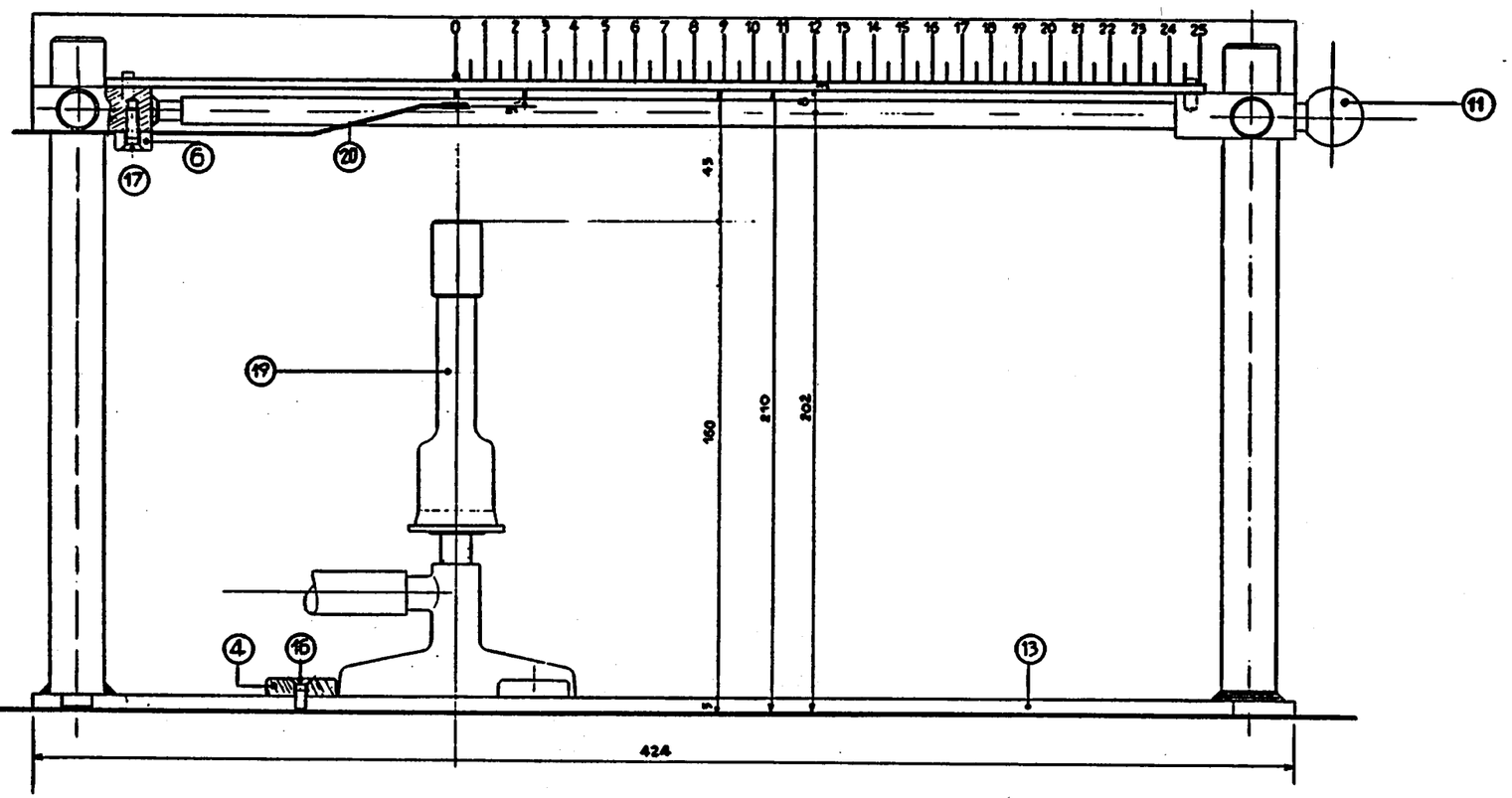
c) After ten or so determinations, it is advisable to renew the datum mark on each support by means of a metal scriber.

TESTS OF FLAME PROPAGATION IN A COALDUST-FLUID MIXTURE

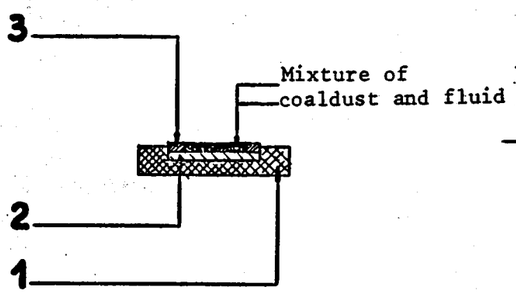
Right hand side elevation



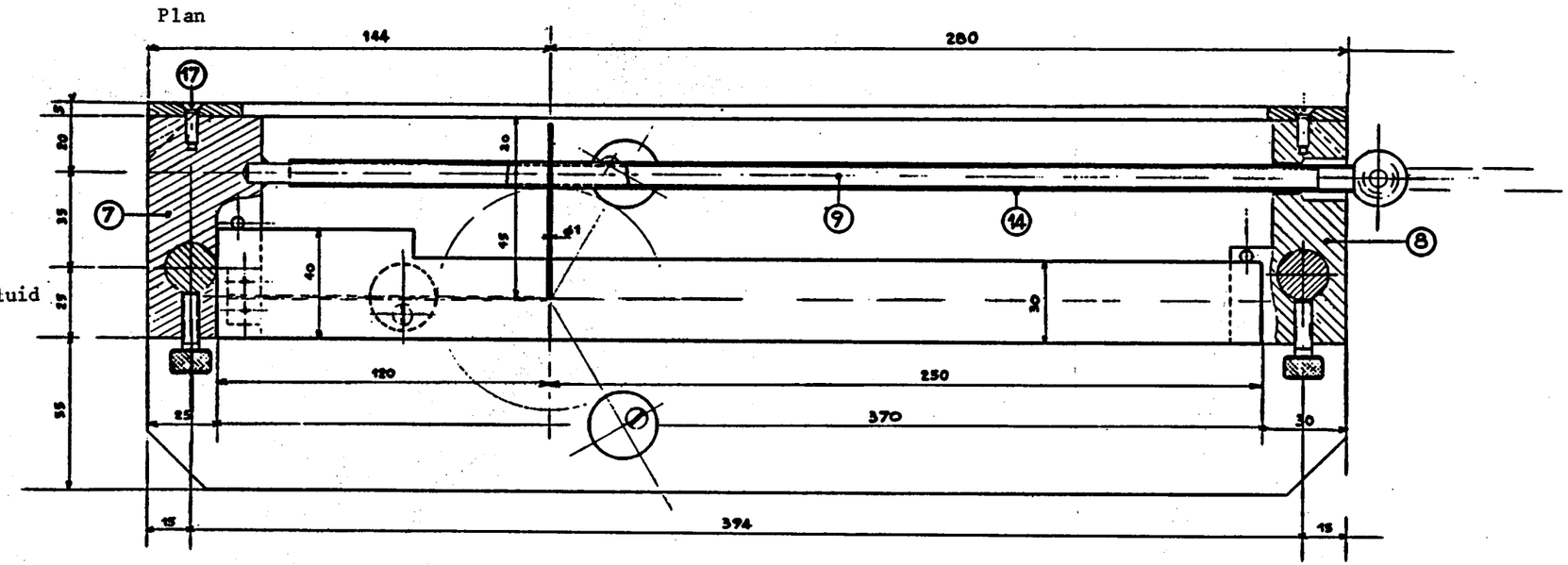
Front elevation



Preparation of test pieces



Plan

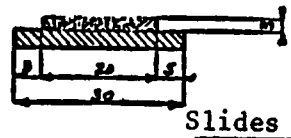


Annex II

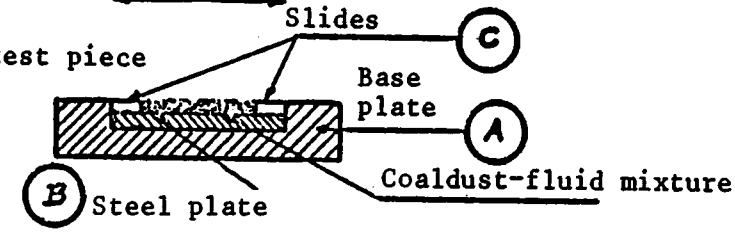
MAKING OF TEST PIECE

Scale 1:1 and 1:2

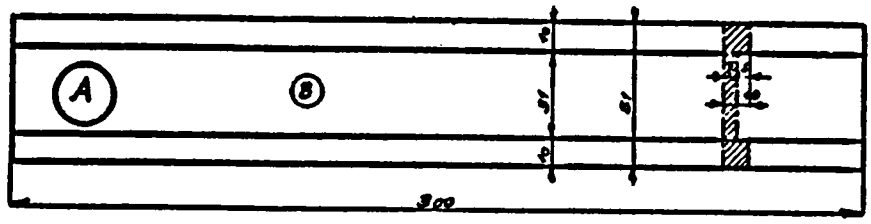
Finished test piece



Jig for making test piece

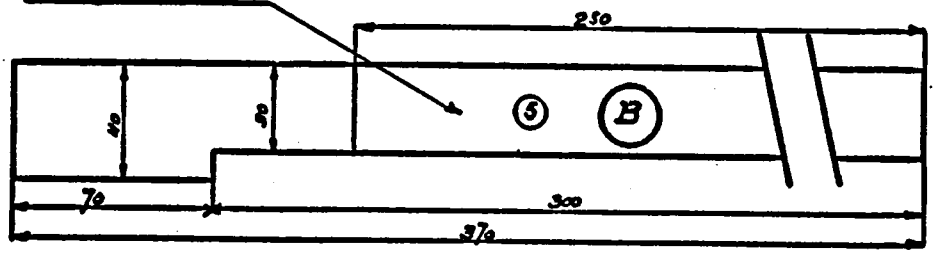


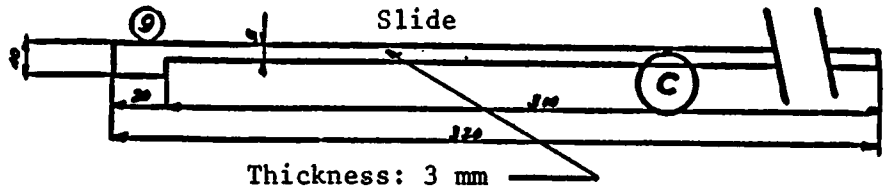
Base plate



Thickness: 3 mm

Steel plate





Thickness: 3 mm

B

Burner closed : approx. 150 mm

BURNER FOR FLAME PROPAGATION TEST APPARATUS

C

ANNEX III

DETERMINATION OF THE SOFTENING POINT

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 a bath of liquid 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.

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. 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.

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 wipped 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 - 78 °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.

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.

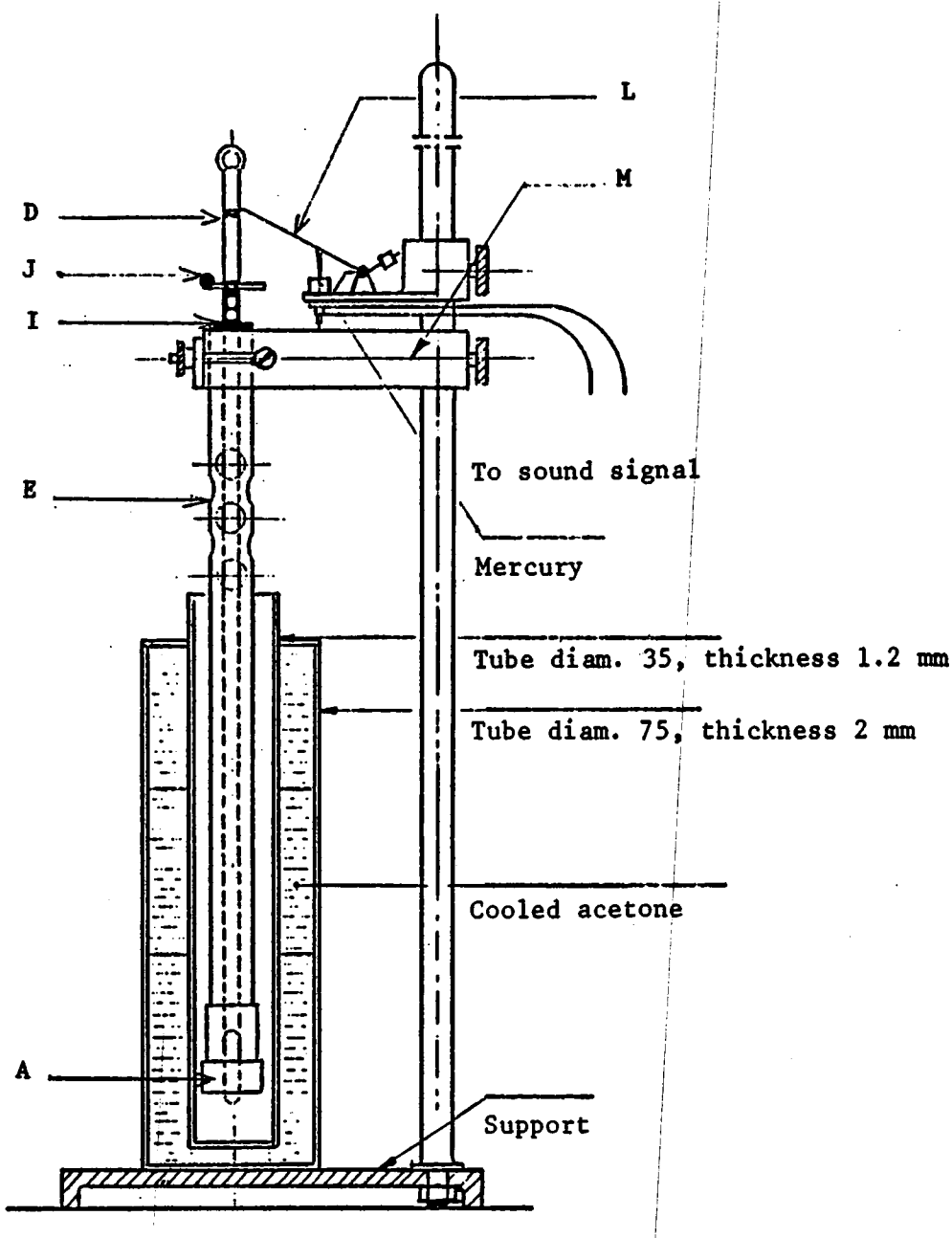
6. SPECIAL CASE OF PRODUCTS WITH SOFTENING POINTS ABOVE 0 °C

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 ^{o}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.

Annex III

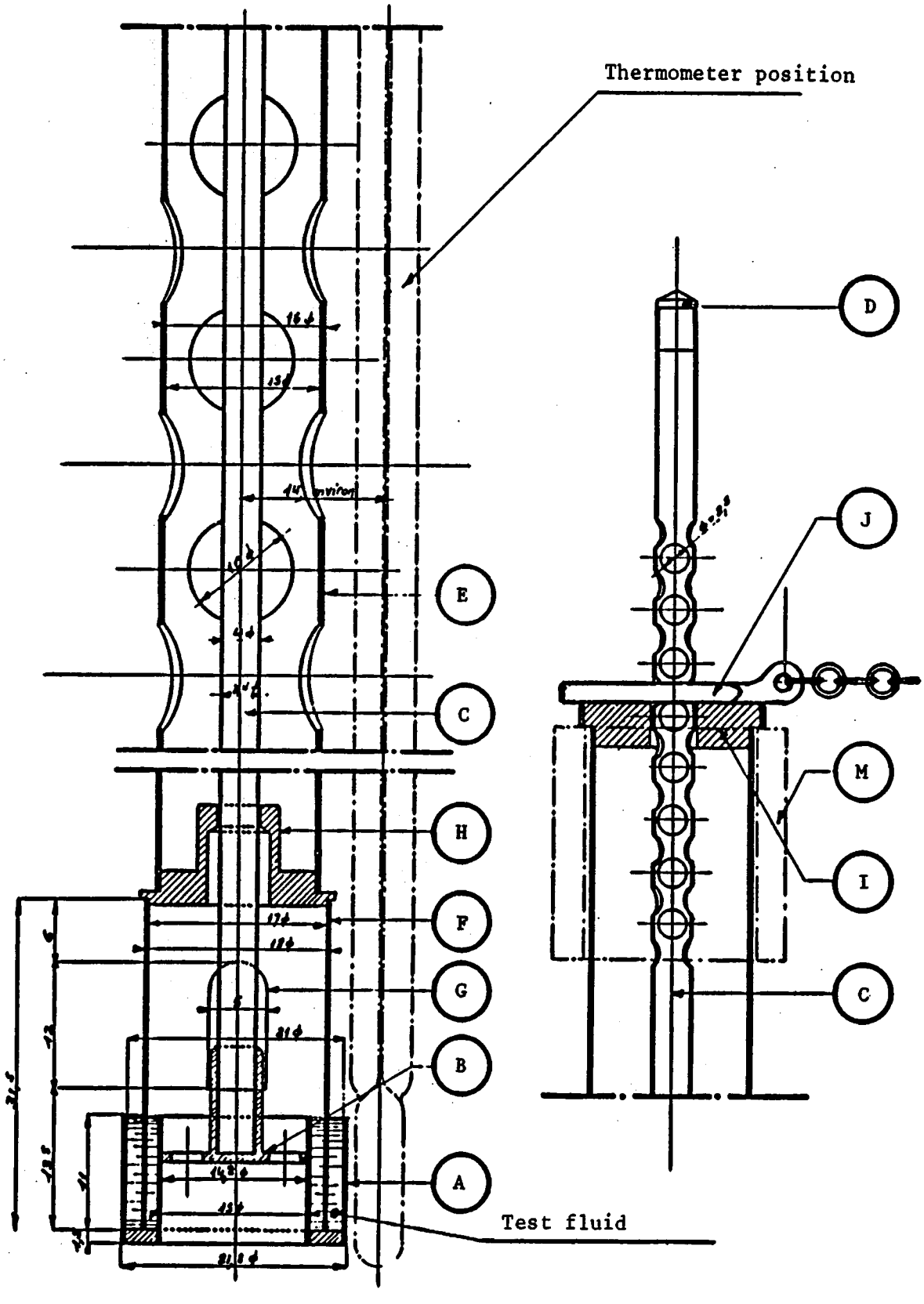
DETERMINATION OF THE SOFTENING POINT



GENERAL DIAGRAM

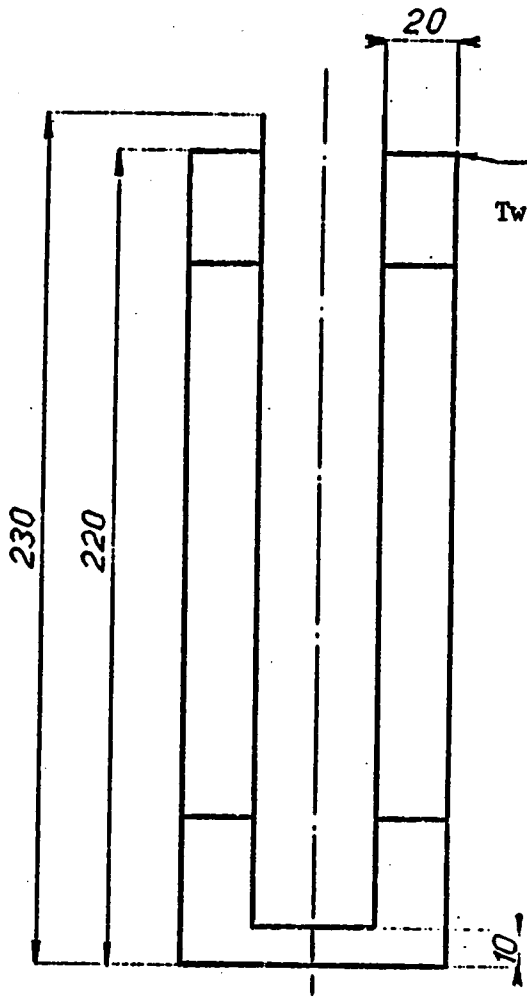
Annex III

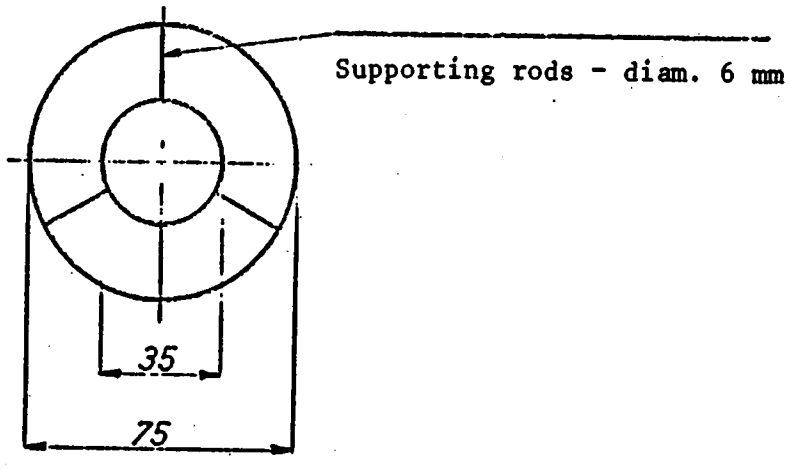
DETERMINATION OF THE SOFTENING POINT



Annex III

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

ANNEX IV

DETERMINATION OF KINEMATIC VISCOSITIES

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

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

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

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. 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).

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.

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.

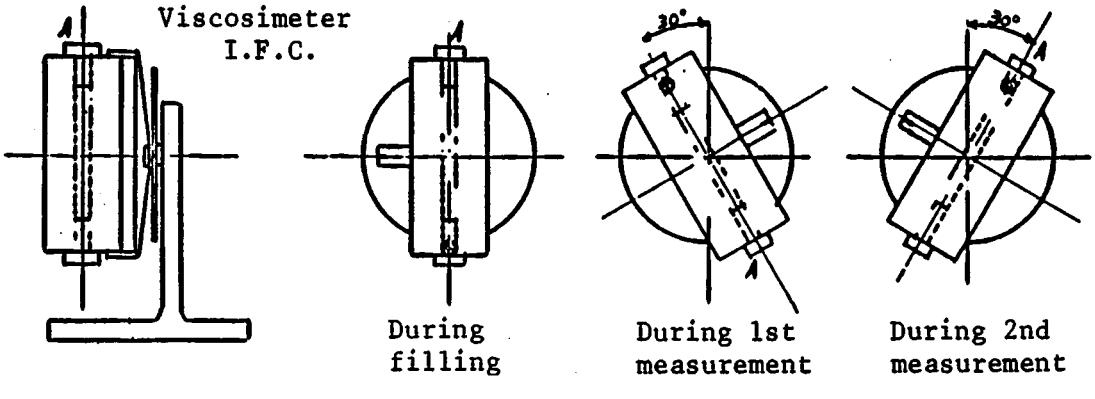
c) Cleaning the calibrated tube and the balls

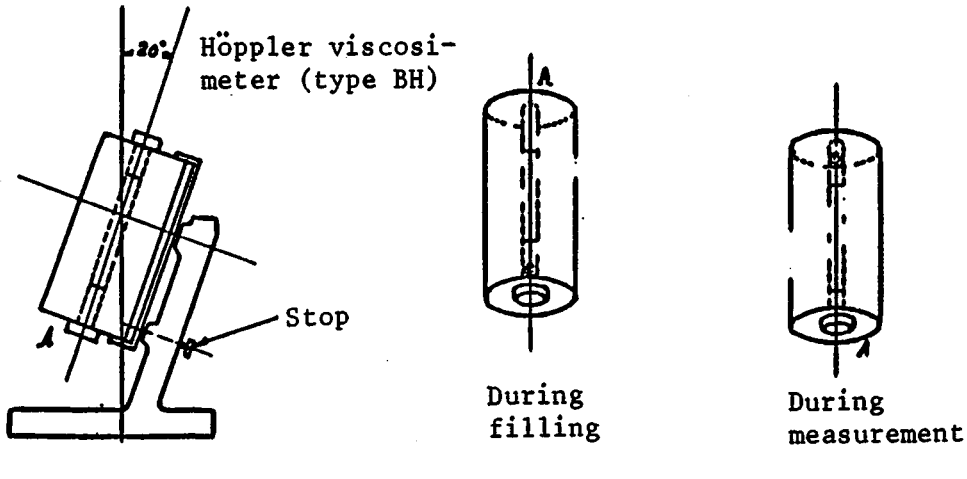
The tube is cleaned with solvents and rinsed with pure ether. In the case of aqueous solutions, clean with a hot solution of soda containing 5% concentrated ammonia to remove any grease, and then rinse with distilled water.

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

MEASUREMENT OF KINEMATIC VISCOSITY

Annex IV





ANNEX V

DETERMINATION OF VAPOUR PRESSURE

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.

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. 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.

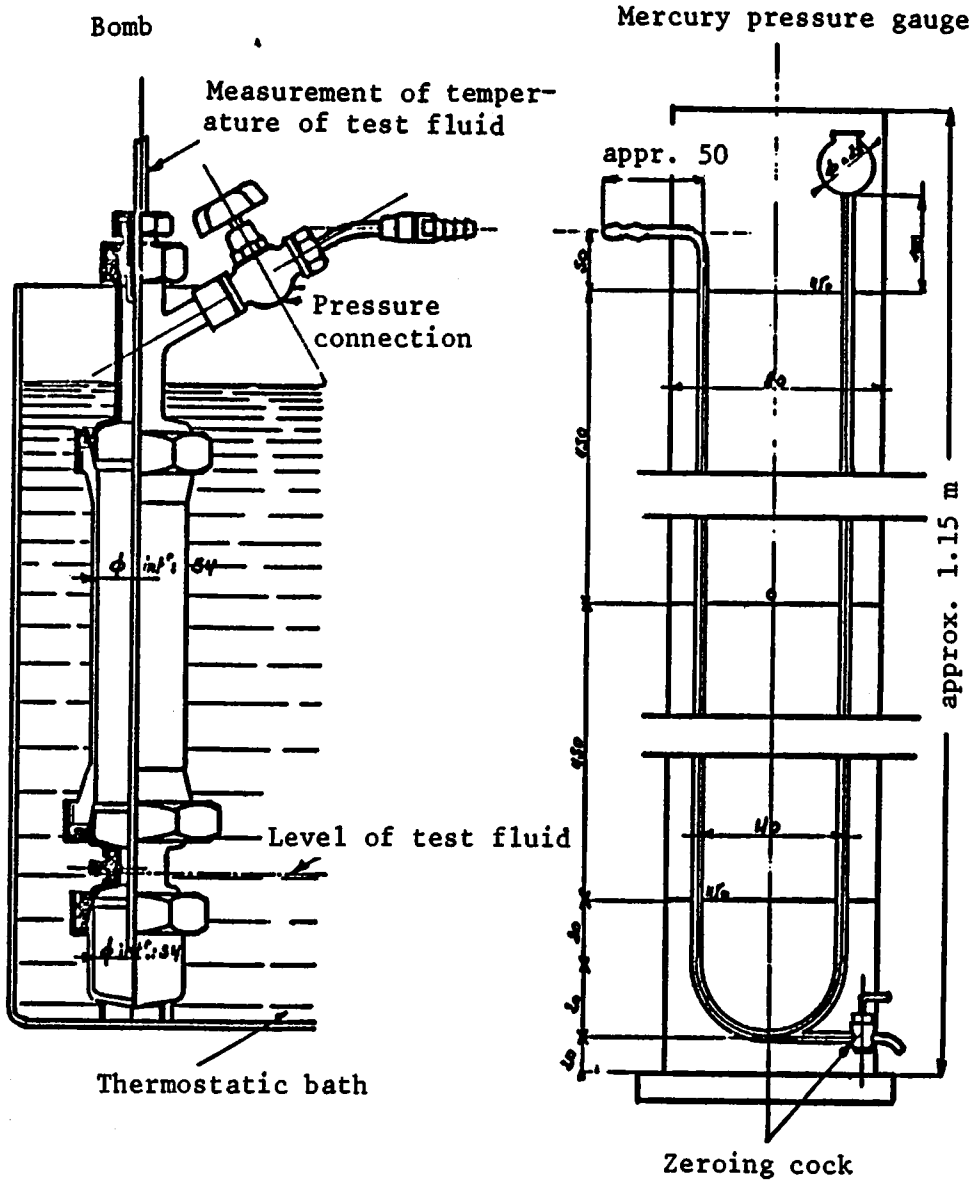
The corrected value must be converted into bars.

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

Annex V

DETERMINATION OF VAPOUR PRESSURE



ANNEX VI

DETERMINATION OF pH

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.

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. 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]

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.

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.

6. EXPRESSION OF RESULTS

The results are given to the nearest 0.1 pH unit.

ANNEX VII

DETERMINATION OF SHEAR STRENGTH

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.

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. 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.

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 or 200 bars are being conducted by way of research.

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).

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).

7. EXPRESSION OF RESULTS

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

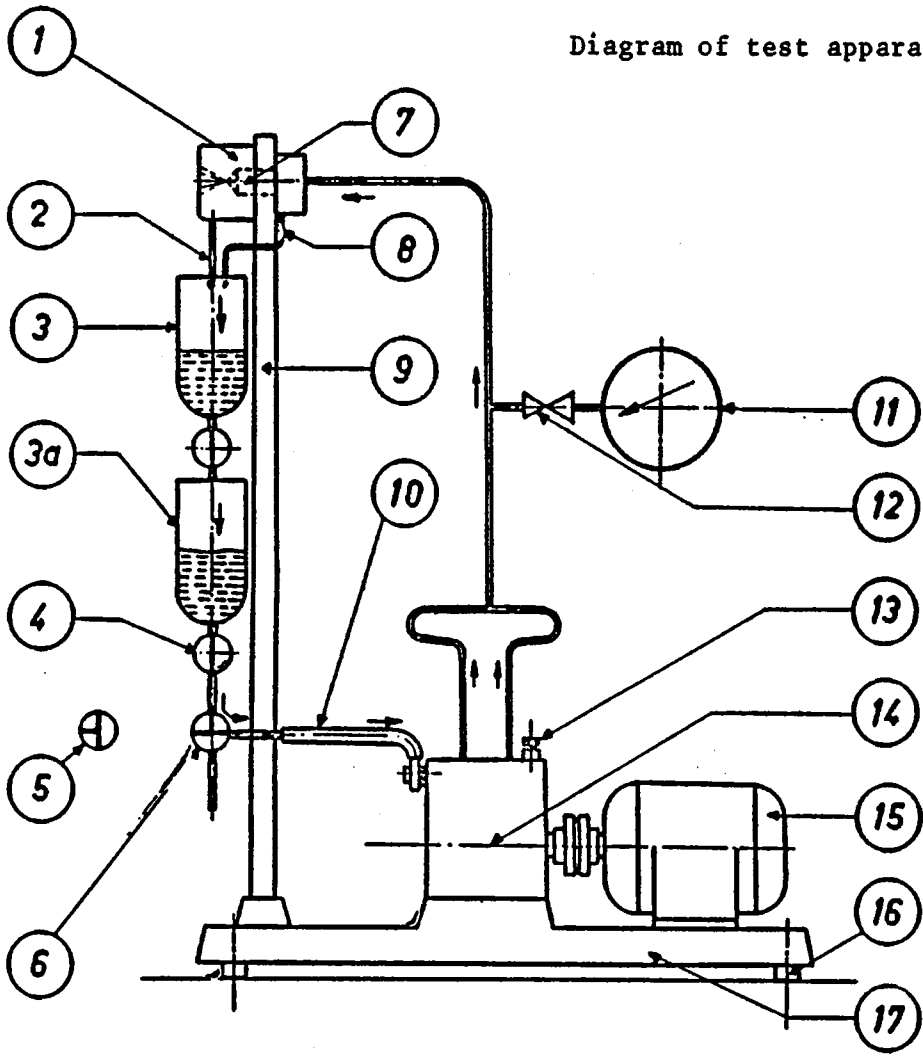
Viscosities	: at - 20, 0, 20 and 50 °C for forms C and D; at 20 and 50 °C for forms A and B.
Softening points	: for all groups
pH	: for forms A and C; and neutralization number for forms B and D.
Water content	: for forms A, B and C.

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

Annex VII

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

ANNEX VIII

DETERMINATION OF ANTI-CORROSIVE POWER

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).

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. APPARATUS AND CHEMICALS

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

b) Watch-glasses, to cover the beakers;

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.

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.

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.

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.

7. PROBABLE FUTURE CHANGES TO THE TEST

It is likely that in the future 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 apparatus 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.

ANNEX IX (A)

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.

1. FIELD OF APPLICATION

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

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. 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 (γ = 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 (γ = 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.

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 lenghts 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).

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.

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). 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. After 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. 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.

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.

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

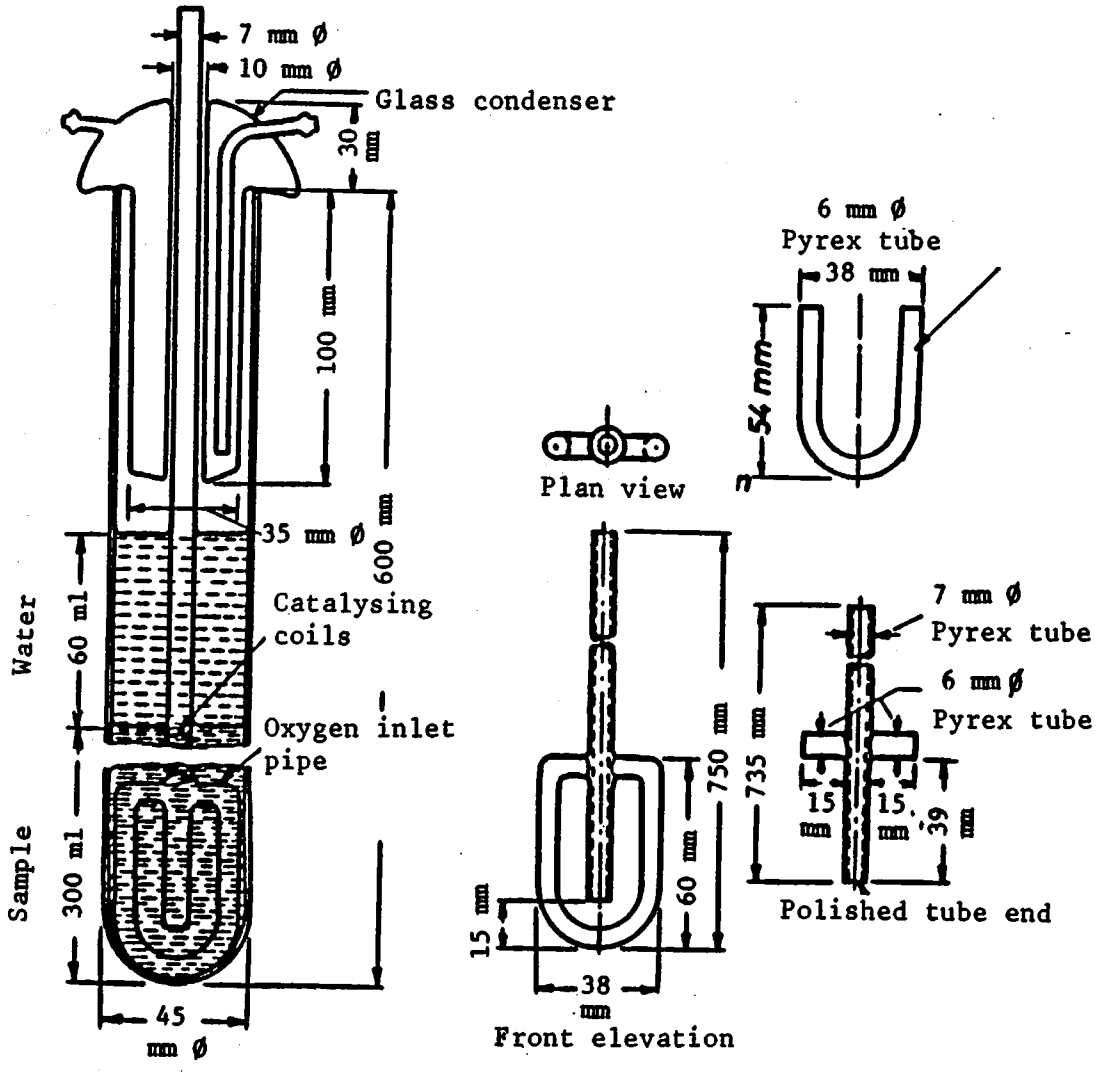


Fig. 1: OXIDIZING CELL

ANNEX IX (A)

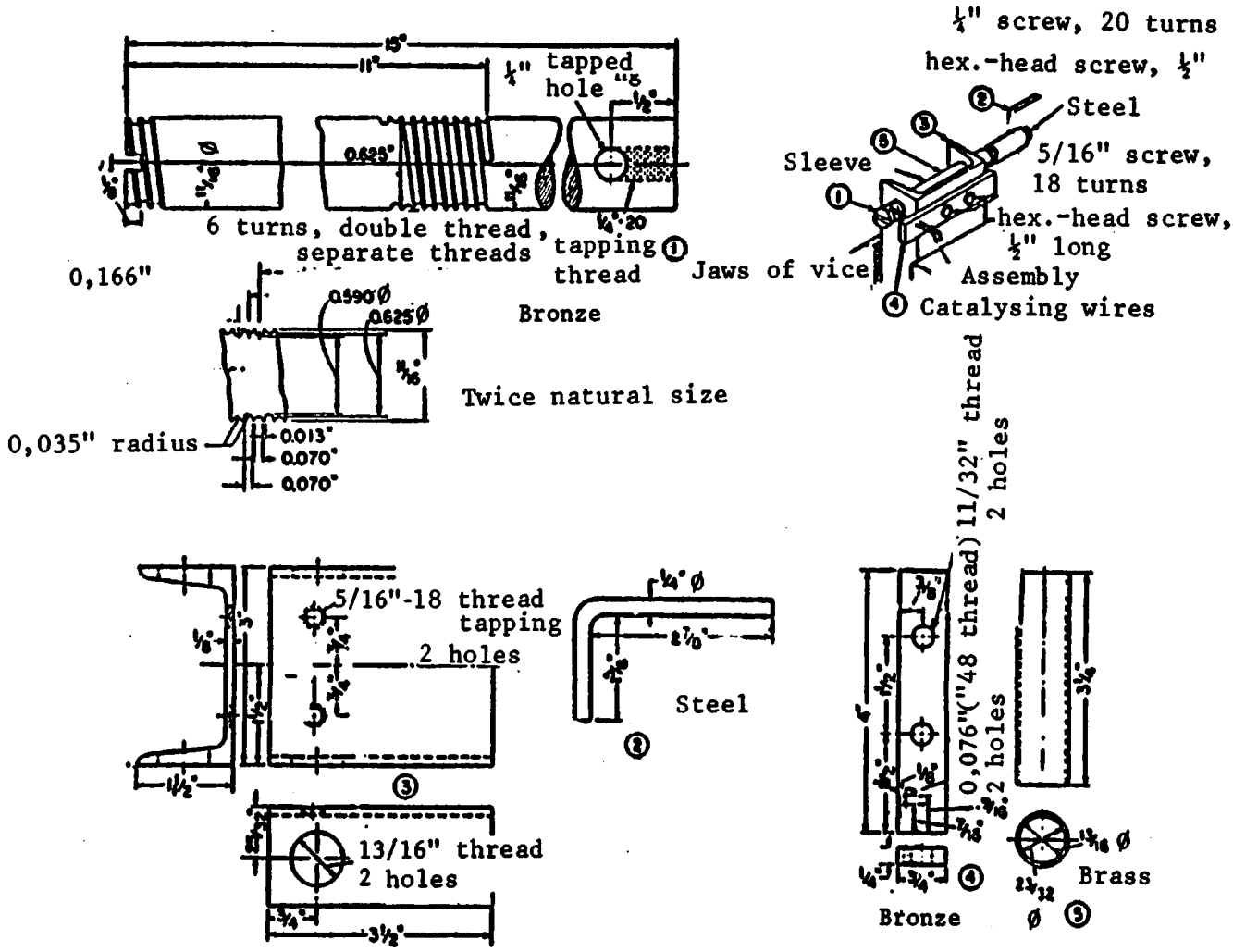


Fig. 2: SPINDLE FOR WINDING CATALYSING COILS

ANNEX IX (B)

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.

1. FIELD OF APPLICATION

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

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. APPARATUS AND CHEMICALS

a) Oxidizing cell (fig. 1)
The cooling jacket of the oxidizing cell differs from that described in Annex IX (A) 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.

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).

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.

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 remaider 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). 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. It is advisable to carry out a blank filtration test with distilled water, and to determine the variation in weight.

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.

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

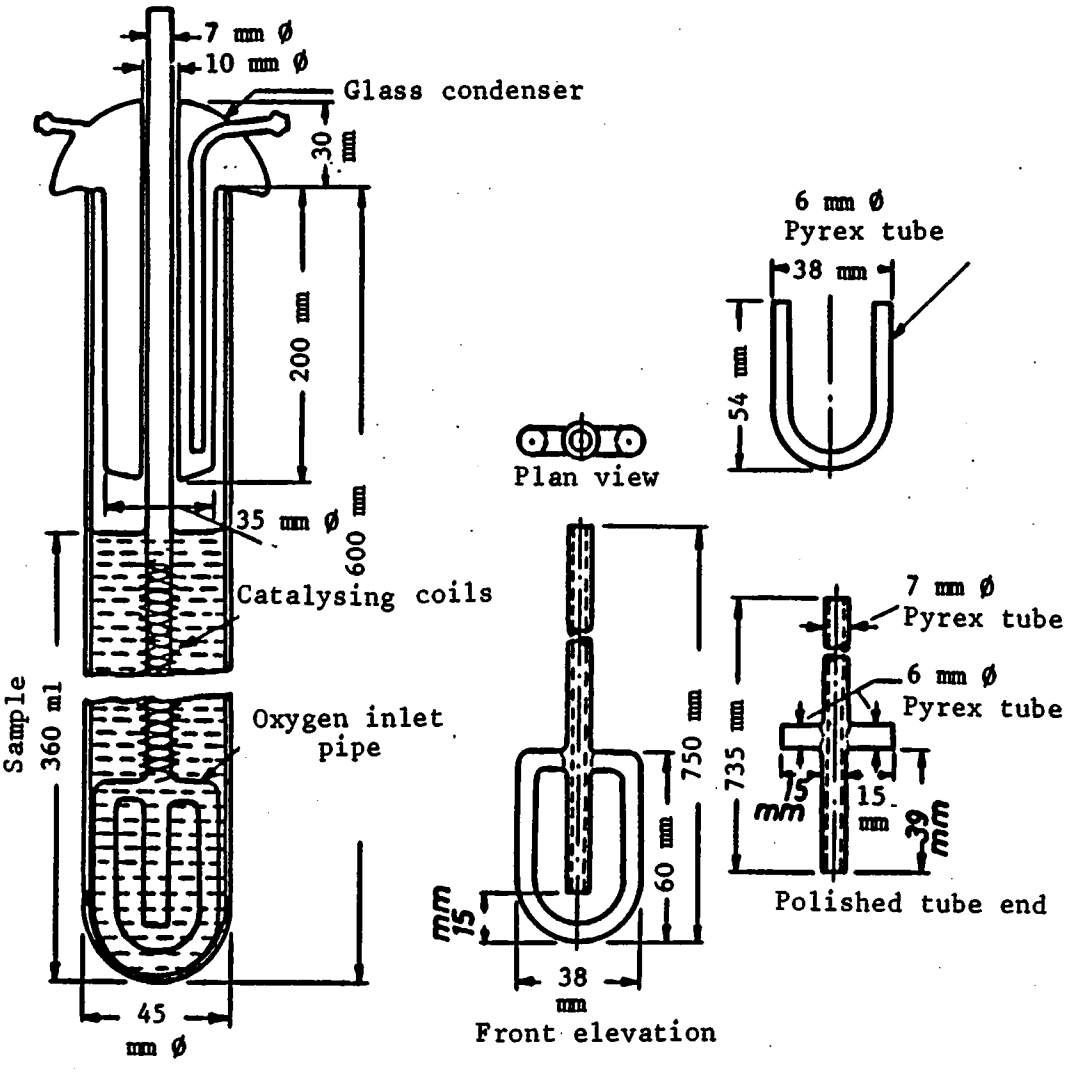


Fig. 1: OXIDIZING CELL

Annex X (B)

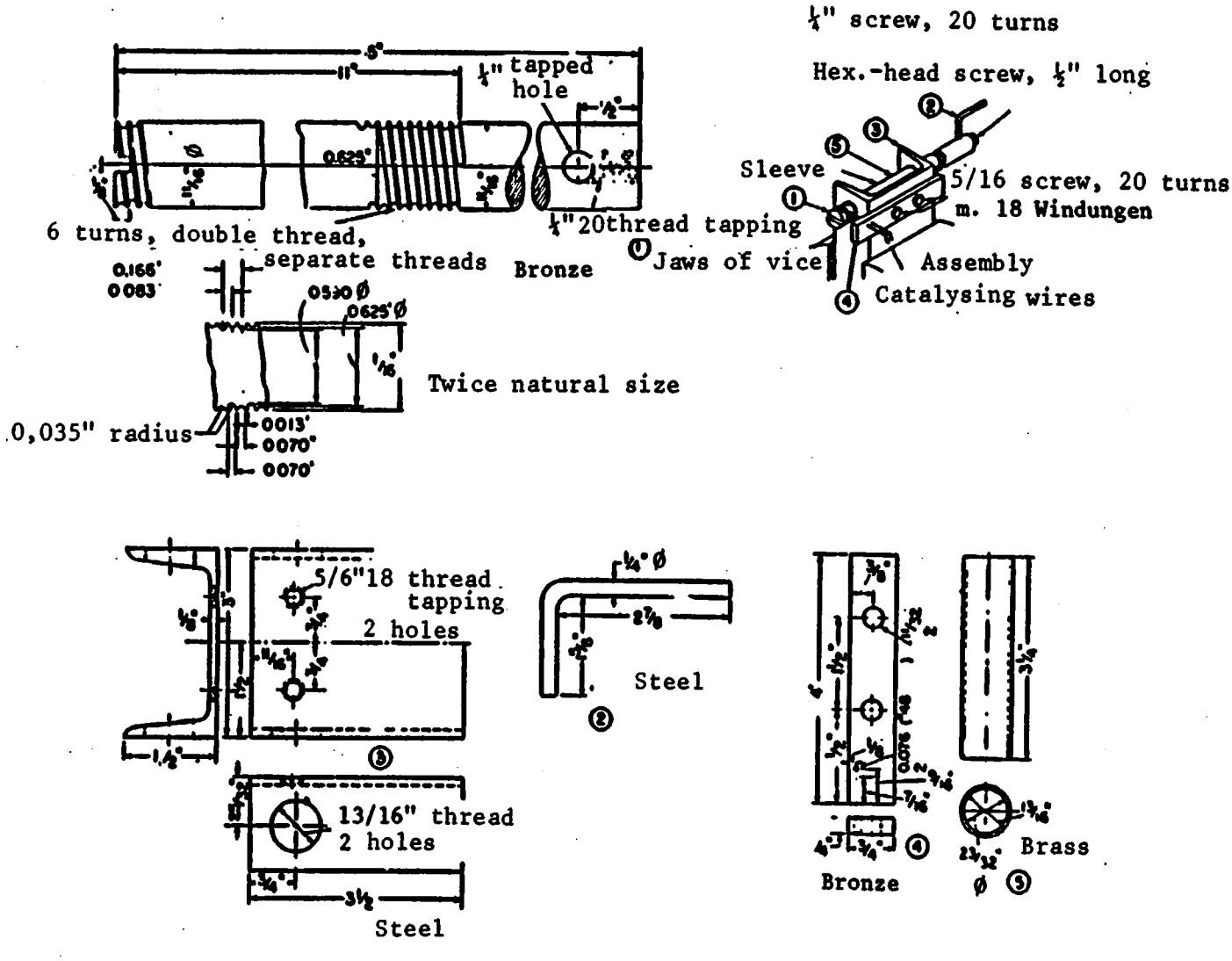


Fig. 2: SPINDLE FOR WINDING CATALYSING COILS

ANNEX X

METHOD FOR DETERMINING THE BEHAVIOUR OF PACKINGS AND SEALS

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.

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. 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.

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.

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:

$$H = H_1 - H_2$$

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.

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.

ANNEX XI (A)

A. ASSESSMENT OF EXTREME PRESSURE CHARACTERISTICS
(4-ball machine)

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.

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. 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 105 Cr2 (W1), hardness 64 ± 2 HRC, made by SKF, Schweinfurt.

d) Chronometer reading in tenths of a second.

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.

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.

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.

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

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

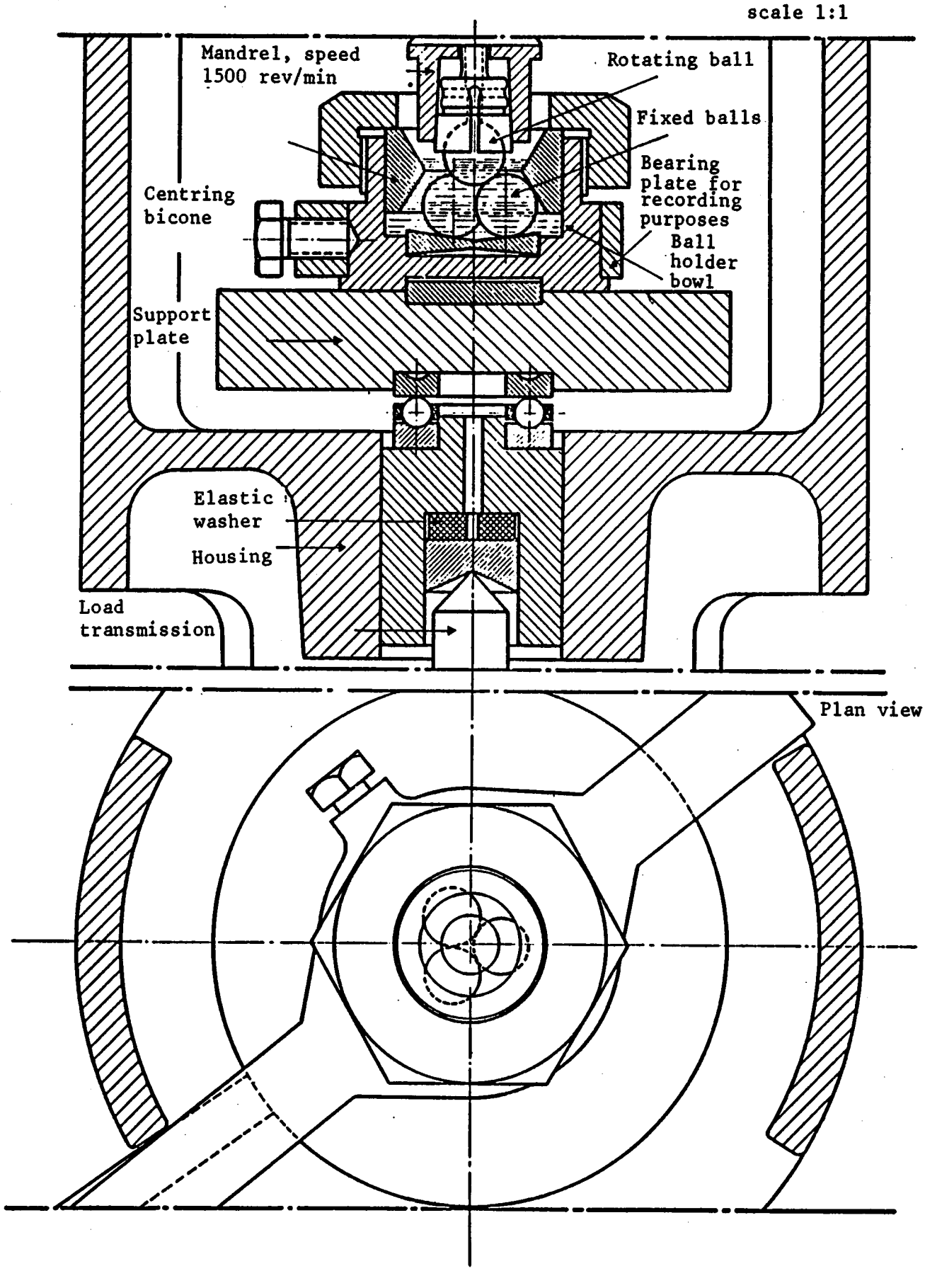
c) Expression of results

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

Annex XI (A)

ASSESSMENT OF EXTREME PRESSURE CHARACTERISTICS

4-Ball machine
scale 1:1



ANNEX XI (A)

4 BALL MACHINE

CORRECTED MEAN LOADS

Applied load P (kg)	Diam. of indentation						Mean diam d (mm)	Factor K = P.dH	Corrected load Pc = 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								191.09	
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		_____

ANNEX XI (B)

B: ASSESSMENT OF ANTI-WEAR CHARACTERISTICS
(VICKERS pump test bench)

1. AIM

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

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. 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.

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. 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.

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.

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.

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.

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. 912014 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, making sure that the lid stays perpendicular to the shaft.

The operating temperature may not vary from ± 20 °C. After 30 min the pressure in the pump 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.

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.

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.

Annex XI (B)

ASSESSMENT OF ANTI-WEAR PROPERTIES

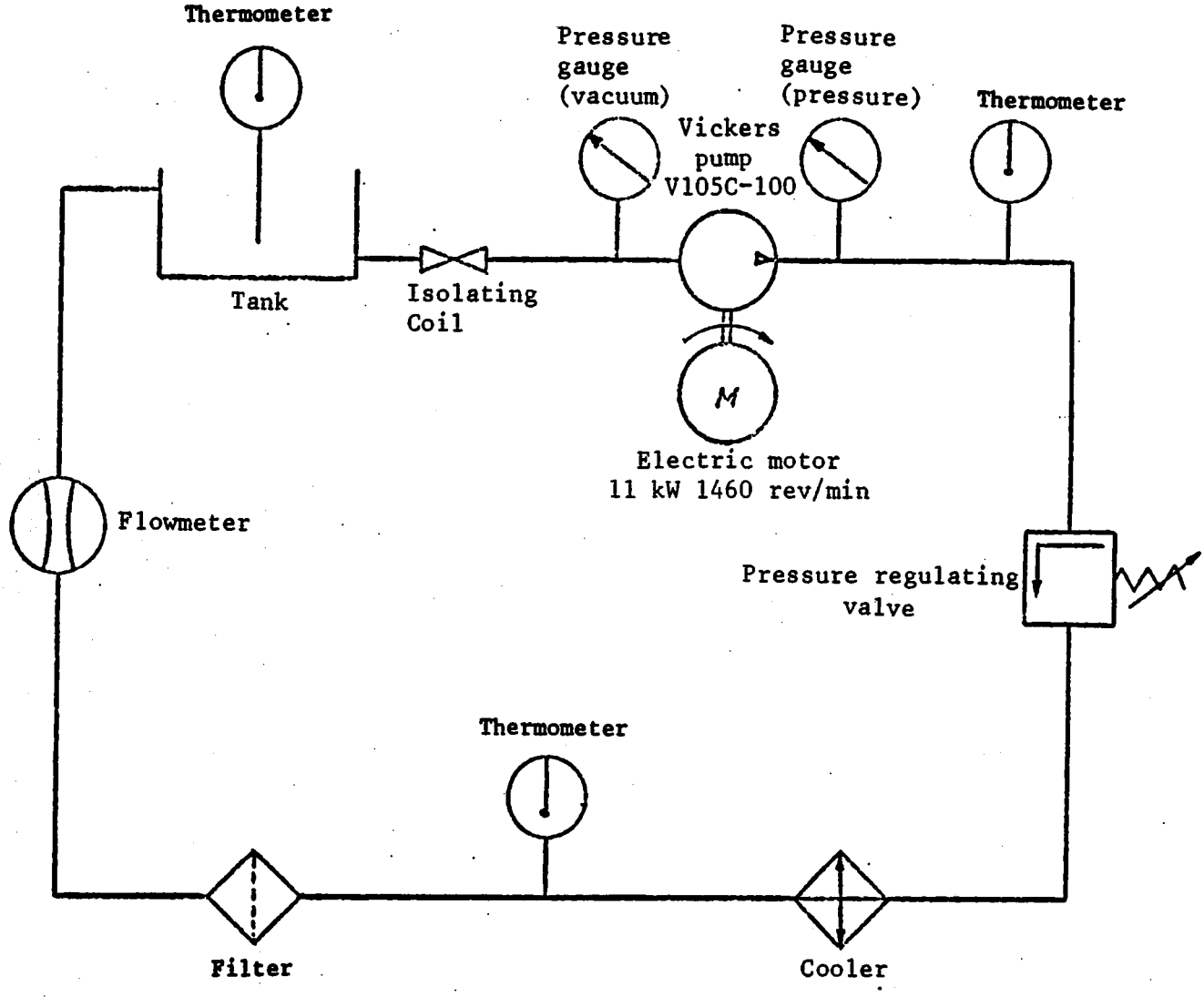


Diagram of test assembly

ANNEX XII

DETERMINATION OF FOAMING TENDENCY

(Taken from ASTM 892 - 63)

1. APPLICATION

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

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. 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.

4. CHEMICALS

a) Petroleum ether or heptane.

b) Chemically pure acetone.

c) pure benzene.

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 benzene 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). The tube and stone are then dried in dry (oil-free) air. The pipe is finally wiped with a clean, 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.

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°, and for form D fluids at 95°, 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.

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

ANNEX XIII

DETERMINATION OF EMULSION STABILITY

1. PRINCIPLE

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

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. 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.

ANNEX XIV

DETERMINATION OF DE-AERATION ABILITY

1. APPLICATION

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

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. 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.

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).

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.

6. EXPRESSION OF RESULTS

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

$$L_n = \frac{100\ (d_o - d_n)}{d_o - d_1}$$

L_n = content of air dispersed in the fluid (% volume) after n min (n = I, 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_1 = 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.

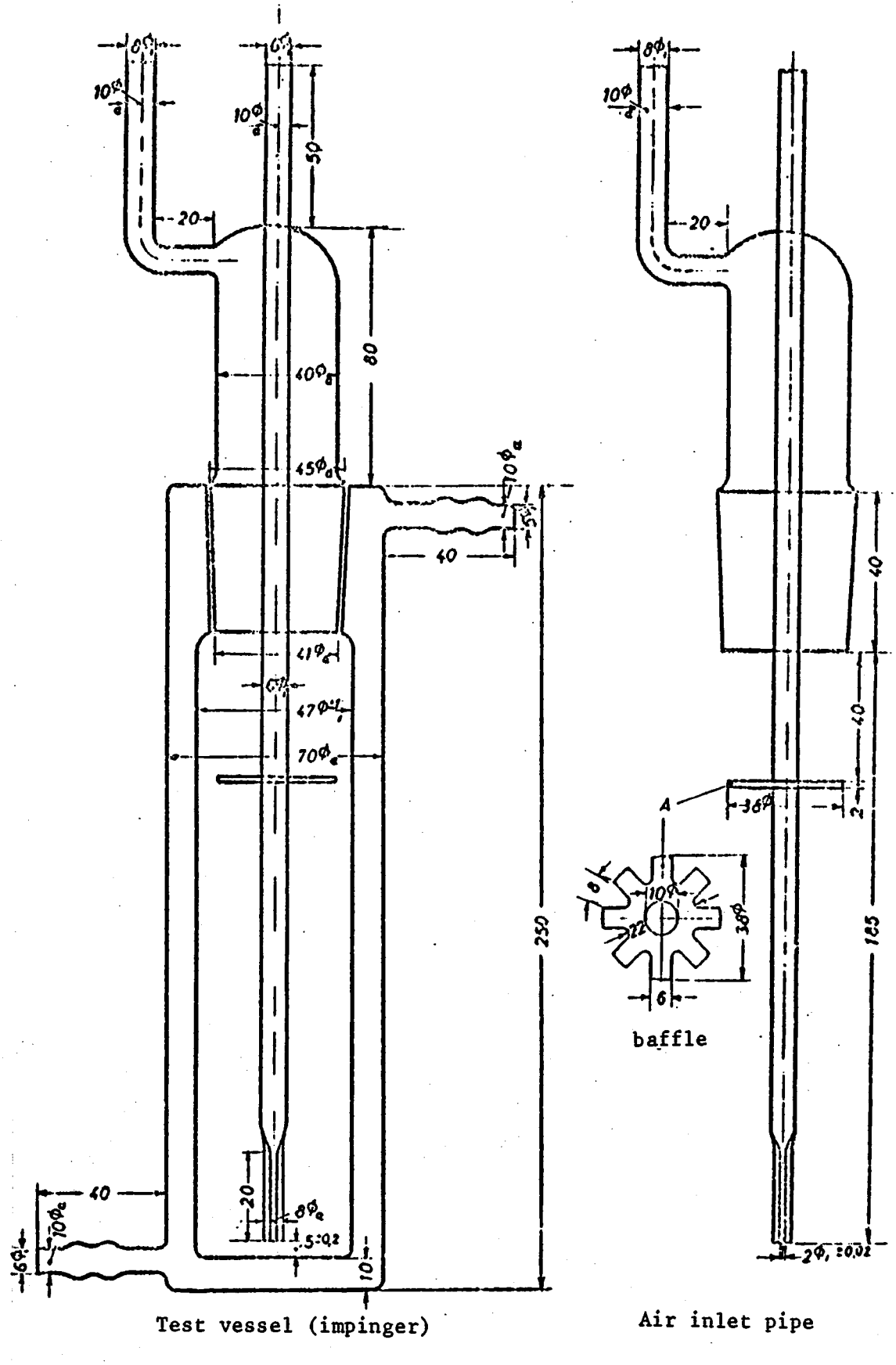


Fig. 1 - TÜV ESSEN IMPINGER USED FOR DISPERSION OF AIR IN OIL

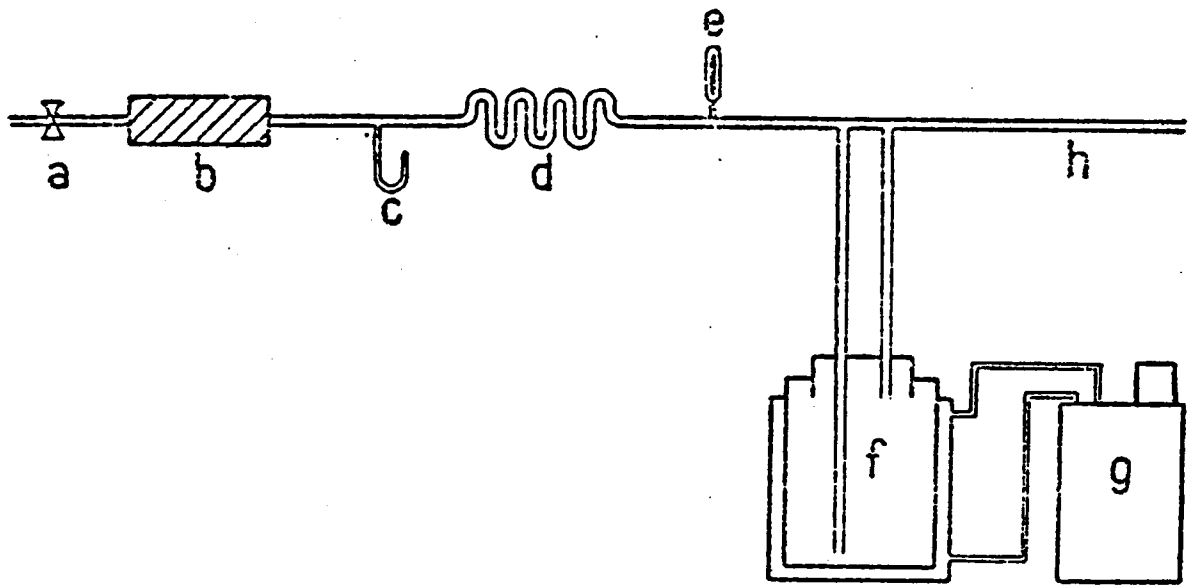


FIG. 2 - TEST ASSEMBLY. a) REGULATING COCK, b) FILTER, c) PRESSURE GAUGE, d) HEATER, e) THERMOMETER, f) IMPINGER WITH AIR INLET PIPE, g) THERMOSTAT, h) AIR OUTLET

ANNEX XVII

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

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.

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 approporiate) 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 2 P, 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 whiche 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

3. DETERMINATION OF TOXICITY

a) Overall toxicity

In a shallow vessel (capacity 2 litres) and at room temperature, dilutions of the fluid to a final volume of 1 litre at concentrations of:

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

are made up with well 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. Tests to determine the irritant effect on the eyes

Using a standard eye-dropper, a drop of test fluid is placed in the conjonctival 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. Test to determine irritant effect on the skin

Using the same animals as were employed for test 1, cuntaneous 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 shared 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
Complex lesions of the skin according to extent and duration	:	5 to 10
Death of the test animals	:	10

d) Aerosol toxicity test

The fluid is diluted half and half with distilled water and placed in an aerosol generator. The temperature of the generator is maintained at room temperature for fluids of form A, and at 70 °C for those of form C. The generator must disperse 20 to 30 ml/hour of fluid in the form of an aerosol containing about 90 % particles of diameter less than 3 microns. The entraining air flow is 1 m^3/hour.

After passing through a Vigreux tube, the aerosol is fed to a parallelepipedic Plexiglas chamber of approximately 140 l capacity. The dimensions recommended for this chamber are: length 800 mm, width 350 mm, height 500 mm. At the bottom of the front walls there are connections for the aerosol inlet and outlet pipes (diam. 20 mm). The inside of the chamber is accessible by a lateral door.

A cage with Plexiglas bars as wide apart as possible holds the animals; dimensions: base 300 x 200 mm. The size of the feet fixed to the base of the cage must allow the cage to be placed in the centre of the aerosol chamber.

Three male Wistar rats weighing about 200 g are placed in the cage and exposed to the aerosol in the chamber for three hours. Before the test and during the next 14 days of observation, the animals are weighed.

Another three rats of the same weight and age are kept as controls under identical conditions.

Disregarding any infections, the results are classified as follows:

No reaction observed	:	0
Symptoms of irritation such as: circulatory disorders, nervous affections, ceasing after one hour	:	1
after 6 hours	:	2
after 5 days	:	3
Loss of weight or insufficient gain in weight	:	4
Death of one animal in the 14 days	:	5
Death of two of the animals in the 14 days	:	6
Death of three of the animals in the 14 days	:	7
Death of one animal during the test	:	8
Death of one animal during the test and of one other in the 14 days	:	9

Death of two animals during the test	: 9
Death of one animal during the test and of the two others in the 14 days	: 10
Death of two animals during the test and of the other in the 14 days	: 10
Death of the three animals during the test	: 10

d) Thermal decomposition products

1. Procedure

Thermal decomposition of the test fluid takes place in an enclosed metal chamber, at one end of which the fluid is sprayed at a pressure of approximately 10 atm. on to a heating plate by means of a diesel type injector (conical spray nozzle with a minimum spraying angle of 30°). At the other end a mixture of vapours and air is drawn off. This air (10 litres/minute) is drawn off through openings in the middle of the chamber. A parition between the injector and the air inlet prevents the injected fluid from being drawn off before it has reached the heating plate.

The injector can spray at 0.35 to 3.5 ml per minute.

The temperature of the evaporation plate is monitored by means of a thermocouple.

The tests are carried out at 200° and 700 °C.

After the combustion chamber, a condenser unit is interposed in the suction line. After passing through this, the mixture of gases is fed to the test chamber.

The test is performed at an injection rate of 1 ml per minute; if the condensate exceeds one third of the volume injected, the test is repeated with a flow of 0.5 ml per minute.

2. Toxicological examination of the thermal decomposition products

The extracted gases (about 10 l/min) are fed into a stream of fresh air in the proportions of 1 part gas: 2 parts air. This mixture passes through the rat cage described in section c), after having been cooled to ambient temperature.

The procedure to be followed and the scoring are also as described in section 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 between 0 and 170 obtained from the sum of the scores from each of the above tests, which are first weighted by the coefficients given in the following table:

Oral toxicity	LD 50	1
Irritant effect	eye	5
	skin	5
Aerosols	hot or cold	4
Test on fish		2
Thermal decomposition	200°	1
	700°	1

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

ANNEX XVIII

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

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.

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 $\leq$ 5 g/kg : 5

LD 50 $\leq$ 0.05 g/kg : 10

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. Test of 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 inert 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 infection, 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 tests

1. Cold aerosol

The undiluted fluid is placed in an aerosol generator. The temperature of the generator is maintained at 50 °C. The generator must disperse 10 to 15 ml per hour of fluid in the form of an aerosol containing about 90 % of particles with a diameter less than 3 microns. The entraining air flow is 1 m^3/hour.

After passing through a Vigreux tube, the aerosol is fed into a plexiglas parallelepipedic chamber of approximately 140 litres capacity. The recommended dimensions for

this chamber are: length 800 mm, width 350 mm, height 500 mm. On the bottom of the front walls there are connexions for the aerosol inlet and outlet pipes (diam. 20 mm). The inside of the chamber is accessible by a lateral door.

A plexiglas cage with bars, as open as possible, holds the animals; dimensions: base 300 x 200 mm, heigth 200 mm. The feet on the base should have dimensions such that this cage can fit in the centre of the chamber containing the aerosol.

Three male Wistar rats weighing approximately 200 g are placed inside the cage and exposed to the aerosol in the chamber for 3 hours. Before the test and during the next 14 days of observation, the animals are weighed.

3 other rats of the same weight and age are kept as controls under identical conditions.

Disregarding any infections, the results are classified as follows:

No reaction observed	: 0
Symptoms of irritation such as circulatory disorders, nervous affections, ceasing: after 1 hour	: 1
after 6 hours	: 2
after 5 days	: 3
Weight loss or insufficient gain in weight	: 4
Death of one of the animals in the 14 days	: 5
Death of two of the animals in the 14 days	: 6
Death of the three animals in the 14 days	: 7
Death of one of the animals during the test	: 8
Death of one of the animals during the test and of another in the 14 days	: 9
Death of two animals during the test	: 9
Death of one animal during the test and of two others in the 14 days	: 10
Death of two animals during the test and of the other in the 14 days	: 10
Death of all three animals during the test	: 10

2. Hot aerosol

The test described above is repeated on a new group of animals with the generator fluid at a temperature of 150 °C. The aerosol produced is cooled by passing through the piping so that the temperature of the chamber is kept at ambient temperature.

Disregarding any affection, the results are scored as follows:

No reaction observed	: 0
Symptoms of irritation such as circulatory disorders, nervous affections, ceasing: after 1 hour	: 1
after 6 hours	: 2
after 5 days	: 3
Loss of weight or insufficient gain in weight	: 4
Death of one of the animals in the 14 days	: 5
Death of two of the animals in the 14 days	: 6
Death of the three animals in the 14 days	: 7
Death of one of the animals during the test	: 8

Death of one of the animals during the test and of another in the 14 days : 9

Death of one of the animals during the test and of the two others in the 14 days : 10

Death of two animals during the test and of the other in the 14 days : 10

Death of the three animals during the test : 10

c) Thermal decomposition products

1. Procedure

Thermal decomposition of the test fluids is carried out in an enclosed metal chamber. At one end of the chamber, the fluid is sprayed at a pressure of approximately 100 atm on to a heating plate by means of a diesel type injector (conical spray nozzle, with a minimum spraying angle of 30°). At the other end of the chamber a mixture of air and vapours is extracted. This air (about 10 litres/min) is extracted through openings situated in the middle of the chamber. A partition between the injector and the air inlet prevents the injected fluid from being extracted before it has reached the heating plate.

The injector can spray at 0.35 to 3.5 ml per minute.

The temperature of the evaporation plate is monitored by means of a thermocouple and can be set to values up to 700 °C.

A condenser unit is inserted in the extraction piping after the combustion chamber. After passing through this condenser, the mixture is fed to the test chamber.

The tests are carried out at 200 °C and 700 °C. The injection rate is 1 ml per minute. If the condensate exceeds one third of the volume injected, the test is repeated with a flow of 0.5 ml per minute.

2. Toxicological examination of the thermal decomposition products

The extracted gases (about 10 litres/min) are fed into a stream of fresh air in the proportions of 1 part gas: 2 parts air. This mixture passes through the rat cage described in section B 2), after having been cooled to ambient temperature. The procedure and scoring are also as in section B 2).

The scoring applies to both the test at 200° and the test at 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.

ANNEX

LIST OF EXPERTS

1. TECHNICAL EXPERTS

Dipl.-Ing. E. BREDENBRUCH (Chairman)
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Dipl.-Ing. K. GRUMBRECHT
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