COMMISSION OF THE EUROPEAN COMMUNITIES

COM(74) 966 final

Brussels, 4 July 1974

PROPOSAL FOR A COUNCIL DIRECTIVE

on the approximation of the laws of the Member States relating to seamless aluminium alloy gas cylinders

(submitted to the Council by the Commission)

PROPOSAL FOR A COUNCIL DIRECTIVE ON THE APPROXIMATION OF THE LAWS OF THE MEMBER STATES RELATING TO SEAMLESS ALUMINIUM ALLOY GAS CYLINDERS

EXPLANATORY MEMORANDUM

I. Introduction

On the 5th January 1973, the Commission forwarded to the Council the proposal for a general directive concerning approximation of the laws of the Member States relating to common provisions for pressure vessels and the methods for inspecting them together with the first special directive on unwelded steel gas cylinders.

Within the framework of harmonisation of the various classes of mass-produced pressure vessels and with a view to eliminating the many barriers that there are to freedom of movement owing to the diversity of the legislative requirements, administrative and statutory governing design and inspection, in force in the Member States of the Community, the Commission has, accordingly, prepared the proposal for a directive relating to welded unalloyed steel gas cylinders.

This is the third proposal for a special direct and concerns light alloy cylinders. It is similar to the lines of the proposal of the directive relating to unwelded steel gas cylinders but is being expanded to take into consideration technical aspects and safety factors peculiar to this type of gas cylinder.

As against the use of steel, the use of aluminium alloys for the production of cylinders is of comparatively recent origin. Given the increasing growth in the demand for light alloy cylinders, principally because of their lightness characteristics and smooth appearance, it is absolutely essential to extend this proposal for a special directive which will allow the free circulation of cylinders produced from light alloys, between all Member States.

Indeed, the different leglislative provisions at the present time in existence within the Member States are as result, particularly in the light of experience acquired that the production of the various alloys are not the same, is a very serious restriction of trade.

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It is therefore necessary to draw up a list of aluminium alloys admissible for the manufacture of cylinders as well as to specify the inspection and tests to be carried out to meet safety requirements; the corrosive behaviour of certain alloys particularly to be subjected to a special examination.

The employment of alloys, otherwise of a lesser commercial importance, having a tensile strength greater than 500 N/mm^2 is not allowed to avoid the risk of crack propogation on the stress-resistant cylinder casing.

In order not to hamper technical progress, the Directive also makes provision for the use of alloys other than those prescribed, provided that they pass an intercrystalline corrosion-resistance test simulating the likely effects of weather near the sea.

As with steel gas cylinders, this proposal for a Directive provides for "optional" harmonization and applies only to empty cylinders.

2. Remarks on the proposal for a Directive

In view of the analogy which exists, this proposal for a directive follows the plan adopted for the Directives on steel gas cylinders. The articles define in particular:

- the scope of application of the Directive;
- the cylinder which may be placed in free circulation and the means of ensuring such circulation;
- the capacity and pressure values of the cylinder determining the inspection procedures to which it will be subjected.

After defining the alloys approved for cylinder production and giving the broad lines of the construction rules and processes, Annex I specifies the methods of carrying out the tests and the criteria of acceptance for the cylinders tested.

Theprocedures for EEC approval and verification and the requirements for marking cylinders are then laid down.

Annex II defines the intercrystalline corrosion resistance test to be undergone by other than approved alloys in order to be used in the production of cylinders.

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Annexes III and IV give forms of certificates of EEC approval and verification.

3. Consultation with circles concerned

With a view to the preparation of the present proposal for a directive, a very detailed examination of the different national regulations will first be carried out. Conclusions drawn from the work of the principal standardisation and controlling bodies will be utilised to a great extent. Already, at the preparatory stage of the work, on the spot contacts are made frequently with consumer associations, controlling bodies, as well as professional circles. This procedure enables the Commission's services to prepare a homogenious draft directive which forms the subject matter for detailed discussions at the principal meetings of the Commission's working group of experts, to which consumer associations, controlling and standardisation bodies and the European association of Manufacturers are invited.

4. Consultation of the European Parliament and the Economic and Social Committee.

Pursuant to Article 100(2) of the Treaty, the opinions of thesetwo bodies are required. Implementation of the proposal provisions would, in the case of certain Member States, necessitate amendments to their legislation.

LIST OF THE PRINCIPAL LEGISLATIONS IN FORCE IN THE MEMBER STATES RELATING TO SEAMLESS ALUMINIUM ALLOY GAS CYLINDERS

BELGIUM :

General Regulation for work protection (R.G.P T.) :
Kingdom Orders of 11th February 1946 and 27th September 1947 (Articles 349 to 363).

Federal Republic of GERMANY :

Regulation for mobile containers and for compressed gas filling equipment (Druckgasverordnung) of 20th June 1968.

DENMARK :

a) Order for mobile containers designed for holding compressed gas, liquified or dissolved, n° 338 of 11th December 1935 and the following amendments :

n° 75 of the 19th March, 1959,
n° 73 of the 21st March 1961,
n° 345 of the 27th September 1963;

b) Instruction n° 17/1972 relating to labelling and periodical tests.

FRANCE :

a) Amended legislation of 28th October 1943;
b) Amendment Order of 18th January 1943;
c) Amendment Order of 23rd July 1943;
d) Order of 12th November 1962

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ITALY :

a) R.D.L. (Royal Order-in-Council) n° 1331 of 9th July 1926; (Establishment of the National Association for the Control of combustion);
b) R.D. (Royal Decree) n° 824 of 12th May 1927 (Approval of legislation for the enforcement of Royal Order-in-Council n° 1331 of 9th July 1926);
c) D.M. (Ministerial Order) of 20th August 1933;
d) R.D. n° 2421 of 11th December 1933;
e) D.M. of 1st August 1935;
f) D.M. of 27th October 1969.

IRELAND :

Common Law and particularly the "Rylands v Fletcher" Statute.

LUXEMBOURG :

Grand Duchy Order of 24th October 1938.

UNITED KINGDOM :

Gas Cylinder Conveyance Legislation 1931, as amended in 1947 and 1939, and certain Exemption Orders.

Home Office Specifications HOAL 1, 2, 3 and 4.

PROPOSAL FOR A COUNCIL DIRECTIVE ON THE APPROXIMATION OF THE LAWS OF THE MEMBER STATES RELATING TO SEAMLESS ALUMINIUM ALLOY GAS CYLINDERS

THE COUNCIL OF THE EUROPEAN COMMUNITIES,

having regard to the Treaty establishing the European Economic Community, and in particular Article 100 thereof;

having regard to the proposal from the Commission;

having regard to the Opinion of the European Parliament;

having regard to the Opinion of the Economic and Social Committee;

Whereas in the Member States the construction and methods of control of gas cylinders are subject to mandatory provisions which differ from one Member State to another and consequently hinder trade in such cylinders; whereas it is therefore necessary to approximate these provisions;

Whereas the Council Directive of ..... on the approximation of the laws of the Member States relating to common provisions on pressure vessels and to the methods of control of these vessels lays down in particular the procedures of EEC approval and verification for these vessels; whereas, in accordance with that Directive, the technical requirements to be satisfied by EEC type seamless aluminium alloy gas cylinders of 0.2 to 150 litres capacity should be laid down, so that these cylinders can be freely put into circulation, marketed and used, after they have undergone the proposed controls and have been given the proposed marks and symbols;

HAS ADOPTED THIS DIRECTIVE:

Article 1

1. This Directive applies to the stress-resistant shells of refillable seamless aluminium alloy gas cylinders (i.e., formed from a single piece), of a capacity of 0.2 to 150 litres inclusive and designed to contain compressed, liquefied or dissolved gases, the effective pressure of which is greater than 1 bar gauge at a temperature of 15°C. These seamless gas cylinders are hereinafter termed "cylinders".

2. The provision of this Directive shall not apply to:
- cylinders manufactured from an aluminium alloy with a tensile strength greater than 500 N/mm^2;
- cylinders which have been manufactured by a method involving the addition of metal during the process of sealing the bottom end.

Article 2

Cylinders which conform to the requirements laid down in the Annex to this Directive shall be EEC-type cylinders.

Article 3

No Member State may, for reasons connected with constructions or controls, refuse, prohibit or restrict the placing on the market and entry into appropriate service of EEC-type cylinders bearing the EEC approval symbol and the EEC inspection mark, provided that the provisions of Article 4 are complied with.

Article 4

EEC-type cylinders shall be subject to:

(a) EEC approval when their hydraulic test pressure is not greater than 225 bars and if their capacity is not less than 0.2 litresand not more than 1 litre;

(b) EEC aproval and EEC inspection

(1) when their hydraulic test pressure is not greater than 225 bars and if their capacity is more than 1 litre and not more than 150 litres;

(2) when their hydraulic test pressure is greater than 225 bars, irrespective of their capacity.

Article 5

1. Member States shall put into force the laws, regulations and administrative provisions needed in order to comply with this Directive within eighteen months of its notification and shall fortwith inform the Commission thereof.

2. Member States shall ensure that the texts of the main provisions of national law which they adopt in the field covered by this Directive are communicated to the Commission.

Article 6

This Directive is addressed to the Member States.

ANNEX I

1. TERMS AND SYMBOLS USED IN THIS ANNEX

1.1. Yield stress

The term "yield stress" means the conventional yield stress ($R_p0.2$) i.e. the value of the stress (σ) which gives rise to a non-proportional elongation to 0.2 % of the gauge length of the test-piece.

1.2. The symbols used in this Annex have the following meanings:

P_h = relative hydraulic test pressure (design pressure), in bars;

P_r = relative cylinder bursting pressure measured in the bursting test, in bars;

R_e = minimum value of the yield stress ($R_p0.2$) guaranteed by the cylinder manufacturer, in N/mm^2;

R_m = minimum tensile strength guaranteed by the cylinder manufacturer in N/mm^2;

a = minimum thickness of the wall of the cylindrical part of the cylinder, in mm;

D = maximum outside diameter of the cylinder, in mm;

R_t = actual tensile strength, in N/mm^2;

d = diameter of the former for the bend test.

2. TECHNICAL REQUIREMENTS

2.1. Materials

2.1.1. The materials used for the manufacture of the cylinders shall be aluminium alloys which, after the proposed heat treatment, are sufficiently resistant to atmospheric corrosion.

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2.1.2. Aluminium alloys with the following chemical compositions are acceptable for the manufacture of gas cylinders:

Type of alloy		Chemical composition							
		Cu	Mg	Si	Fe	Mn	Zn	Cr	Ti + Zr
a)	min.	-	2.4	-	-	0.3	-	-	-
Al-Mg 3 Mn	max.	0.10	3.8	0.55	0.5	1.0	0.2	0.25	0.2
b)	min.	-	4.0	-	-	0.3	-	-	-
Al-Mg 4.5 Mn	max.	0.10	5.1	0.5	0.5	1.0	0.2	0.25	0.2
c)	min.	-	0.4	0.6	-	0.4	-	-	-
Al-Si 1 Mg	max.	0.10	1.4	1.6	0.5	1.0	0.2	0.35	0.2
d)	min.	5.2	0.2	-	-	0.15	-	-	-
Al-Cu-Mg	max.	6.0	0.4	0.2	0.3	0.35	0.10	0.10	0.25

For the interpretation of the results of the chemical analysis of these alloys, reference shall be made to page 4 of the ISO recommendation R209-1971, page 4.

2.1.3. Any other aluminium alloy may be used for the manufacture of gas cylinders provided that it first passes the atmospheric corrosion resistance tests described in Annex II to this Directive or, failing this, complies with a specification laid down by the Committee for Adaption to Technical Progress referred to in Article 19 of the Council Directive of ..... on the approximation of the laws of the Member States relating to common provisions on pressure vessels and to the methods of control of these vessels.

2.1.4. The cylinder manufacturer shall obtain and provide cast analysis certificates for the aluminium alloys supplied for the manufacture of theicylinders.

2.1.5. The inspection authority shall have the opportunity of making independent analyses. These analyses shall be carried out either on specimens of the alloy taken at the time of casting such as those supplied by the alloy manufactures to the cylinder manufacturer or on the finished cylinders.

2.2. Heat and Mechanical Treatments

The manufacturer shall state the type of manufacture: cold working, type of heat treatment (temperature and duration) and type of cooling.

2.3. Calculation of the Parts under Pressure

2.3.1. The thickness of the cylinder walls shall not at any point be less than that calculated for the cylindrical part by the formula :

$$a = \frac{P_h \cdot D}{\frac{20\,R_e}{1,3} + P_h}$$

The value of R_e shown in the formula shall in no case be greater than 0,85 R_m whatever the type of alloy used.

2.3.2. The minimum wall thickness shall not in any case be less than 1.5, 2 and 3 mm respectively for cylinder diameters of 50 mm or less, greater than 50 and less than 150 mm, and 150 mm or more.

2.3.3. The thickness and the shape of the bottom and the top end shall be such as to satisfy the requirements of the tests provided for in item 3.2 (bursting test) and 3.3 (pressure cycling test) of this Annex.

2.3.4. In order to obtain a satisfactory stress distribution, the thickness of the cylinder walls shall increase progressively in the transition part situated between the cylindrical part and the end; the wall shall be free from defects.

2.4. Construction and Workmanship

2.4.1. Each cylinder shall be examined for thickness and for external and internal surface defects :

- the wall thickness shall not at any point be less than that specified on the drawing;
- the internal and external surface of the cylinder shall be free from defects which would adversely affect the safe use of the cylinder.

2.4.2. The out-of-roundness of the cylindrical shell shall be limited to such a value that the difference between the maximum and minimum outside diameters in the same cross section is not more than 2 % of the average of these diameters.

2.4.3. When a footring is provided, it shall be sufficiently strong and made of a material compatible with that of the cylinder. The shape should preferably be cylindrical and shall give the cylinder sufficient stability. The footring shall not allow water to accumulate or permit ingrees of water between the footring and the cylinder.

3. TESTS

3.1. Mechanical Tests

3.1.1. General requirements

All the mechanical tests for checking the properties of the alloy of gas cylinders shall be carried out on test pieces taken from the finished cylinders.

3.1.2. Types of tests and evaluation of test results

Every test cylinder shall undergo one tensile test in a longitudinal direction and four band tests in a circumferential direction.

3.1.2.1. Tensile test

3.1.2.1.1. The tensile test shall be carried out on a test specimen of the type shown in Fig. 1 of this Annex with a gauge length $L_o = 5{,}65 \sqrt{F}$, F being the right cross-section of the test piece. The two faces of the test piece representing the inside and outside surfaces of the cylinder shall not be machined.

3.1.2.1.2. The elongation in percent shall not be less than:

- 12 % for the alloys referred to in (a), (b) and (c) of the table in item 2.1.2 and for the alloys subject to the requirements in 2.1.3;
- 10 % for the alloys referred to in (d) of the table in item 2.1.2.

3.1.2.2. Bend test

3.1.2.2.1. The bend test shall be carried out on specimens obtained by cutting into two equal parts a ring of width 3a; in no case may the width of the test piece be less than 25 mm.

Each strip may be machined only on the edges.

3.1.2.2.2. The bend test shall be carried out by means of a former of diameter d and two cylinders separated by a distance of d + 3a. During the test, the inside face of the ring shall be placed against the former.

3.1.2.2.3. The test piece shall remain uncracked when bent inwards around the former until the interior edges are separated by a distance not greater than the diameter of the former.

3.1.2.2.4. The ratio (n) between the diameter of the former and the thickness of the test piece shall conform to the values given in the following table :

Actual tensile strength R_t in N/mm^2	Value of n
up to 330 inclusive	6
above 330 to 400 inclusive	7
above 400 to 500 inclusive	8

3.2. Bursting test under hydraulic pressure

3.2.1. Test conditions

3.2.1.1. The bursting test under hydraulic pressure shall be carried out in two successive stages by means of an apparatus such that the pressure in the cylinder increases regularly until the cylinder bursts and that the curve of the pressure variation with time is recorded.

3.2.1.2. During the first stage, the rate of pressure increase until the point of which plastic deformation begins shall be about 1 bar/s.

From that point, (second stage) the pumping rate shall be increased to four times that of the first stage and kept constant until the cylinder bursts.

3.2.2. Performance of test

The bursting test under hydraulic pressure shall afford:

- an examination of the pressure/time curve, in order to determine the pressure at which plastic deformation of the cylinder commences, the bursting pressure and the deformation of the cylinder during the test;
- an examination of the fracture and the shape of its edges.

3.2.3. Test acceptance conditions

3.2.3.1. The pressure corresponding to the initiation of plastic deformation shall not be less than four-thirds of the hydraulic test pressure.

3.2.3.2. The measured bursting pressure (P_r) shall not be less than five-thirds of the test pressure (P_h).

3.2.3.3. The bursting test shall not cause any fragmentation of the cylinder.

3.2.3.4. The main fracture shall not show any signs of brittleness i.e., the edges of the fracture shall not be radial but shall be sloped in relation to a diametrical plane and shall show signs of striction throughout their thickness.

3.2.3.5. The fracture shall not reveal a characterized defect in the metal.

3.2.3.6. The fracture shall begin and develop in the cylindrical part and shall not extend into either end of the cylinder.

3.2.4. Data which may help in the interpretation of the test

3.2.4.1. The measurement of the volume of water used from the start of the pressure build-up to the moment of bursting, this gives an indication as to the volumetric expansion of the cylinder.

3.2.4.2. The measurement of the increase in the circumference of the cylinder in the area of the fracture.

3.3. Pressure Cycling Test

3.3.1. The pressure cycling test shall be carried out on two cylinders, which are guaranteed by the manufacturer to be reasonably representative of the minimum thickness values envisaged in the design, using a non-corresive fluid.

3.3.2. The test shall be carried out by subjecting the cylinder to successive reversals of hydraulic pressure between the initial or residual pressure and the pressure known as the test pressure.

The test parameters shall satisfy either one or the other of the following conditions, the choice being left to the Member State:

1) - total number cycles : 80.000
   - test pressure : 2/3 Ph + residual pressure
   - frequency : 15 cycles/minute

2) - total number cycles : 15.000
   - test pressure : Ph + residual pressure
   - frequency : 5 cycles/minute

The residual pressure (the pressure at the end of the cylinder decompression phase) shall not exceed 5 % of the value of the test pressure.

The temperature measured on the outside wall of the cylinder shall not exceed 50°C during the test.

3.3.3. No fracture shall occur during the test.

3.4. Hydraulic test

3.4.1. The value of the hydraulic pressure test shall be equal to the design pressure (P_h).

3.4.2. The water pressure in the cylinder shall increase regularly until the test pressure is reached.

3.4.3. The cylinder shall remain under the test pressure for a time sufficiently long to demonstrate that the pressure shows no tendency to fall and that leak-tightness has been achieved.

3.4.4. Any cylinder which does not satisfy these test requirements shall be rejected.

4. EEC APPROVAL

4.1. The applicant for approval shall submit the documentation necessary for the verification prescribed below, together with a number not exceeding eight prototype cylinders, fully representative of future production, which may be necessary for carrying out the tests prescribed below, and any additional information required by the Member State.

4.2. In the course of the EEC approval process, the Member State shall :

- verify that the calculations provided for in item 2.3 are correct;
- verify that the conditions laid down in items 2.1 and 2.2 are satisfied;
- perform on the cylinders submitted as prototypes:
  - the tests provided for in item 3.1.2. except in respect of the number of tensile tests, three of these tests being required;
  - the test provided for in item 3.2, on two cylinders;
  - the test provided for in item 3.3, on two cylinders, except where only the length parameter of the cylinder differs and is situated in the range of lengths of the types of cylinders which have already been approved;
- issue the EEC approval certificate conforming to the model in Annex III to this Directive.

5. EEC INSPECTION

5.1. For the purpose of EEC inspection the cylinder

manufacturer shall :

5.1.1. make available to the inspection authority:

- the EEC approval certificate;
- the analysis certificates of the aluminium alloys supplied for the manufacture of the cylinders and when the cylinder is manufactured with the alloy referred to in (d) of the table in item 2.1.2 the results of intercrystalline corrosion tests carried out according to one of the methods described in section 1 in Annex II to this Directive, on test pieces taken from one cylinder of each tempering charge;
- the means of identifying the aluminium alloy cast from which each cylinder is manufactured;
- the documents necessary to certify that the cylinders submitted for EEC inspection have undergone the same heat treatment as those submitted for the approval procedure;
- the list of cylinders stating the numbers and the marks provided for in item 6.

5.2. In the EEC inspection

5.2.1. The control authority shall :

- ascertain that approval has been obtained and that the cylinders conform to this approval;
- check the documents which give the data concerning the materials;
- ascertain whether the technical requirements set out in Section 2 have been met, and verify in particular, by an external and internal visual examination of a sample number of between 5 and 10 % of the cylinders in each batch whether the construction and the examination carried out by the manufacturer in accordance with item 2.4.1. are satisfactory;
- be present at the tests prescribed in items 3.1 and 3.2 and check the way they are carried out;
- ascertain whether the information supplied by the manufacturer in the list in item 5.1.1 (fifth indent) is correct. Between 5 an 10 % of the cylinders in each batch shall be subjected to this check;
- issue the EEC inspection certificate conforming to the model in Annex IV to this Directive.

5.2.2. For the execution of the two types of tests prescribed in items 3.1 and 3.2, two cylinders shall be taken at random from each batch of 402 cylinders made from the same cast and having been subjected to the same heat treatment and from each remaining batch of less than 402 cylinders.

One of the two cylinders shall be subjected to the tests prescribed in item 3.1 (mechanical tests) and the other to the test prescribed in item 3.2 (bursting test).

If one of the tests fails even partially to give satisfaction, two further cylinders shall be taken at random from the same batch and subjected to the type of test which was failed.

If either of these two further cylinders fails to give satisfaction, all the cylinders of that batch shall be rejected unconditionally.

5.2.3. The selection of specimens and all the tests shall be carried out in the presence of a representative of the inspection authority.

5.2.4. All the cylinders in the batch shall be subjected to a hydraulic test as specified in item 3.4. in the presence and under the supervision of a representative of the inspection authority.

5.3. Exemption from EEC inspection

For the cylinders referred to in Article 4 (a), of this Directive and in accordance with Article 15 (a), of the Council Directive of ..... on the approximation of the laws of the Member States relating to common provisions on pressure vessels and to the methods of control of these vessels, the manufacturer shall carry out, on his own responsibility, all the test and inspection operations prescribed in item 5.2.

The manufacturer shall provide the inspection authority with all the documents and the test and control reports.

The presence of a representative of the inspection authority (cf. items 5.2.3. and 5.2.4. of this Annex) shall not be required.

6. MARKING

6.1. When all the prescribed checks have been carried out by the inspection authority, and providing that the results are satisfactory, the inspection authority shall issue a certificate stating the checks which have been carried out.

6.2. The markings shall be grouped together and none may be stamped on the stress-resistant shell of the cylinder.

6.3. A symbol shall be stamped on the metal of the top end of each cylinder or on an identification plate, if any, of a minimum thickness of 3 mm; the symbol referred to in item 5.3 of Annex I to the Council Directive of ..... on the approximation of the laws of the Member States relating to common provisions on pressure vessels and to the methods of testing these vessels shall be used for the cylinders referred to in Article 4 (a) of this Directive, and the symbol of EEC type approval $\mathcal{E}$ referred to in Annex I item 5.1., followed by the symbol of EEC inspection "e" referred to in Annex II item 3.1.1.1. (a) of the above-mentioned Directive shall be used for the cylinders referred to in Article 4 (b) of this Directive.

These symbols shall be followed by the following markings :

6.4. Markings relating to the construction

6.4.1. in respect of the metal

A number indicating the value of R_e in N/mm^2, on which the calculation was based.

6.4.2. in respect of the hydraulic test

The value of the test pressure in bars in figures larger than the others and above them, the date (month and year) of the first hydraulic test.

6.4.3. in respect of the type of cylinder

Below the figure relating to the test pressure, the weight of the cylinder in kg, without the valve and tap , and the minimum capacity in litres guaranteed by the cylinder manufacturer.

The weight and the capacity shall be expressed to three significant figures, the third one being fixed "by rounding down" for the capacity and "rounding up" for the weight.

6.4.4. in respect of the origin

The manufacturer's mark and the serial number.

6.4.5. example :

04/72

$\mathcal{E}$ e 270 225.... 6.851

10,4/40,5

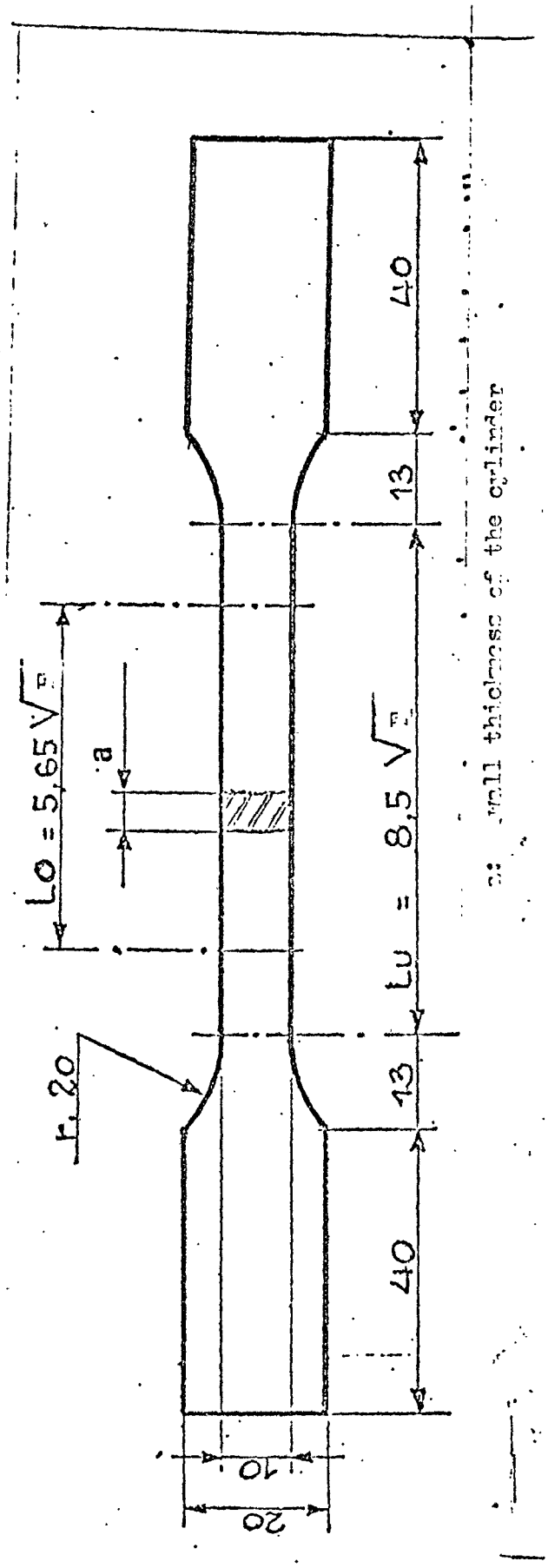

fig. 1 : Tensile test piece

a : wall thickness of the cylinder

A N N E X II

TESTS TO EVALUATE SENSITIVITY TO INTERCRYSTALLINE CORROSION

The method described below consists of immersing the specimens taken from the cylinder under test in two different corrosive solutions at the same time, examining them after a specified etching time for any signs of intercrystalline corrosion and determining the type and intensity of such corrosion. The propagation of intercrystalline corrosion is determined metallographically on polished surfaces cut transversely to the etched surface.

1.1. Sampling

Specimens are taken from the pointed top, the body and the bottom of the cylinder (Fig.1), so that the tests with solution A defined in 2.1.1. and solution B defined in 2.1.2. can be carried out on metal from three parts of the cylinder.

A sufficient number of specimens must be taken to meet the requirements set out in 2.2.1 and 2.2.2.

Each specimen must be of the general form and the dimensions given in Fig. 2.

The faces a1 a2 a3 a4, b1 b2 b3 b4, a1 a2 b2 b1, a4 a3 b3 b4 are all sawn with a band saw and then carefully trimmed with a fine file. The surfaces a1 a4 b4 b1 and a2 a3 b3 b2 which correspond respectively to the inner and outer faces of the cylinder are left in their original rough state.

1.2. Preparation of surface before corrosive etching

1.2.1. Products required

HNO_3 for analysis, density 1.33
HF for analysis, density 1.14 (at 40 %)
Deionized water

1.2.2. Method

In a one-litre pyrex beaker, prepare the following solution :

HNO_3 : 63 cm3
HF : 6 cm3
H_2O : 929 cm3

Heat the solution to 95° C

Dip each specimen suspended on an aluminium wire for one minute in this solution.

Then wash in running water followed by deionized water.

Immerse the specimen in nitric acid as defined in 1.2.1. above for 1 minute, at room temperature, to remove any copper deposit which may have formed.

Rinse in deionized water.

To prevent oxydation of specimens, on completion of preparation they should be plunged in the corrosion bath intended for them (see 2.2 below).

2. PERFORMANCE OF TEST

2.1. Two corrosive solutions are to be used, one with 57 g/l sodium chloride and 3 g/l hydrogen peroxide (= solution A), and the other with 30 g/l sodium chloride and 5 g/l hydrochloric acid (= solution B).

2.2. Preparation of corrosive solutions

2.2.1. Solution A

2.2.1.1. Products required

NaCl, crystallized, for analysis
H_2O_2 100 - 110 medicinal volumes
$KMnO_4$ for analysis
H_2SO_4 for analysis, density 1.83
Deionized water

2.2.1.2. Titration of hydrogen peroxide

Since hydrogen peroxide is not very stable, it is essential to titrate it before use. This is done as follows.

Take 10 cm3 of hydrogen peroxide with a pipette, dilute to 1,000 cm3 (in a gauged flask) with deionized water, thus obtaining a hydrogen peroxide solution which will be called C. With a pipette, place in an erlenmeyer flask :

- 10 cm3 of the hydrogen peroxide solution C
- about 2 cm2 of sulphuric acid, density 1.83.

A solution of permanganate at 1.859 g/l is used for the titration. The permanganate itself serves as an indicator.

2.2.1.3. Explanation of titration

The reaction of the permanganate on the hydrogen peroxide in a sulphuric medium is expressed as :

$$2\ KMnO_4 + 5\ H_2O_2 + 3\ H_2SO_4 \longrightarrow K_2SO_4 + 2\ MnSO_4 + 8H_2O + 5\ O_2$$

which gives the equivalence : 316g $KMnO_4$ = 170 g H_2O_2

Therefore 1 g of pure hydrogen peroxide reacts on 1.859 g of permanganate; hence the use of a 1.859 g/l solution of permanganate, which, volume for volume, saturates 1 g/l of hydrogen peroxide.
Since the hydrogen peroxide was previously diluted 100 times, the 10 cm3 of the test sample represent 0.1 cm3 of the original hydrogen peroxide.

By multiplying by 10 the number of cubic centimeters of permanganate solution used for the titration, the titre T of the initial hydrogen peroxide in g/l is obtained.

2.2.1.4. Preparation of the solution

Method for 10 l :

Dissolve 570 g of sodium chloride in deionized water to obtain a total volume of about 9 l. Add the quantity of hydrogen peroxide calculated below. Mix and then make up the volume to 10 l with deionized water.

Calculation of hydrogen peroxide volume to be placed in solution

Quantity of pure hydrogen peroxide required: 30g.

If the hydrogen peroxide contains T grammes of H_2O_2 per litre, the volume required, expressed in cubic centimetres, will be :

$$\frac{1{,}000 \times 30}{T}$$

2.2.2. Solution B

2.2.2.1. Products required :

NaCl, crystallized, for analysis
HCl, pure concentrated, 37 % HCl
Deionized water

2.2.2.2. Preparation of the solution :

Method for 10 l :

Dissolve 300 g of sodium chloride and 50 g of HCl (50 g ≙ 0.5 %) in 9 l of deionized water, mix well and make up to 10 l.

2.3. Etching conditions

2.3.1. Etching in solution A

The corrosive solution is placed in a crystallizer (or possibly a large beaker) and this in its turn is placed in a water bath. The water bath is stirred with a magnetic stirrer and the temperature is regulated with a contact thermometer.

The specimen is either suspended in the corrosive solution by an aluminium wire or placed in the solution so that it rests only on the corners, the second method being preferable. The etching time is 6 hours and the temperature fixed at 30 ± 1° C. Care should be taken to ensure that the quantity of reagent is at least 10 cm3 per cm2 of specimen surface.

After etching, the specimen is washed in water, immersed for about 30 seconds in 50 % dilute nitric acid, washed again in water and dried by compressed air.

2.3.2. A number of specimens can be etched at the same time provided that they are of the same type of alloy and that they are not in contact. The minimum quantity of reagent per unit of specimen surface must, of course, be respected.

2.3.3. Etching in solution B

The corrosive solution is poured into a suitable glass container (e.g. a beaker). The test is carried out at room temperature. While it is impossible to avoid variations in room temperature during the test, it is preferable to carry out the test in the water bath, the temperature of which is adjusted to 23°C by a thermostat. Etching time is 72 hours.

The specimens are kept immersed in the corrosive solution in accordance with 2.3.1. After etching, the specimens are very carefully washed with deionized water and dried by compressed air free of grease. Care must be taken to see that the ratio quantity of corrosive solution/specimen surface in ml/cm2 is 10 : 1 (see 2.3.1.).

3. PREPARATION OF SPECIMENS FOR EXAMINATION

3.1. Products required

Casting dishes with, e.g., the following dimensions :

- external diameter : 40 mm
- height : 27 mm
- wall thickness : 2.5 mm

Araldite DCY 230)
Hardener HY 951) or equivalent

3.2. Method

Each specimen is placed vertically in a casting dish on its face a1 a2 a3 a4. Around it is poured a mixture of araldite DCY 230 and hardener HY 951 in the proportion 9 to 1.

The setting time is about 24 hours.

A certain amount of material is removed from the face a1 a2 a3 a4, preferably by lathe, so that the section a'1 a'2 a'3 a'4 examined under the microscope cannot show corrosion from the surface a1 a2 a3 a4. The distance between the faces a1 a2 a3 a4 and a'1 a'2 a'3 a'4, i.e. the thickness removed by the lathe, must be at least 2 mm (Figs. 2 and 3).

The section for examination is polished mechanically with alumina first on paper and then on felt.

4. MICROGRAPHIC EXAMINATION OF SPECIMENS

The examination consists of noting the intensity of intercrystalline corrosion round the entire perimeter of the section. In so doing, account is taken of the properties of the metal on the outer and inner surface of the cylinder and in the thickness of it.

The section is first examined at low magnification (e.g., x40), in order to locate the most corroded areas, and then at a higher magnification, usually about x 300, in order to assess the nature and extent of the corrosion.

ANNEX II

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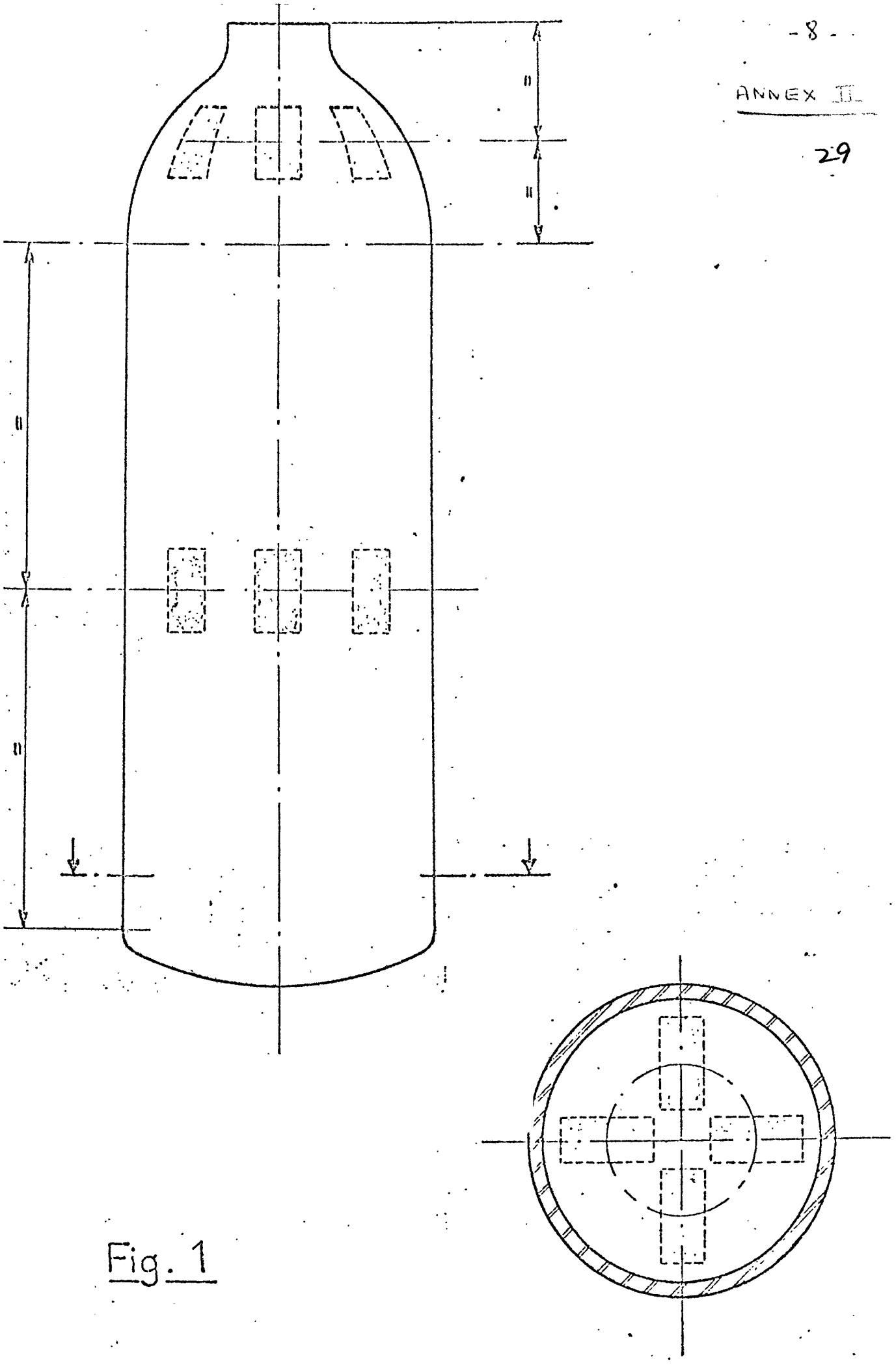

Fig. 1

ANNEX II

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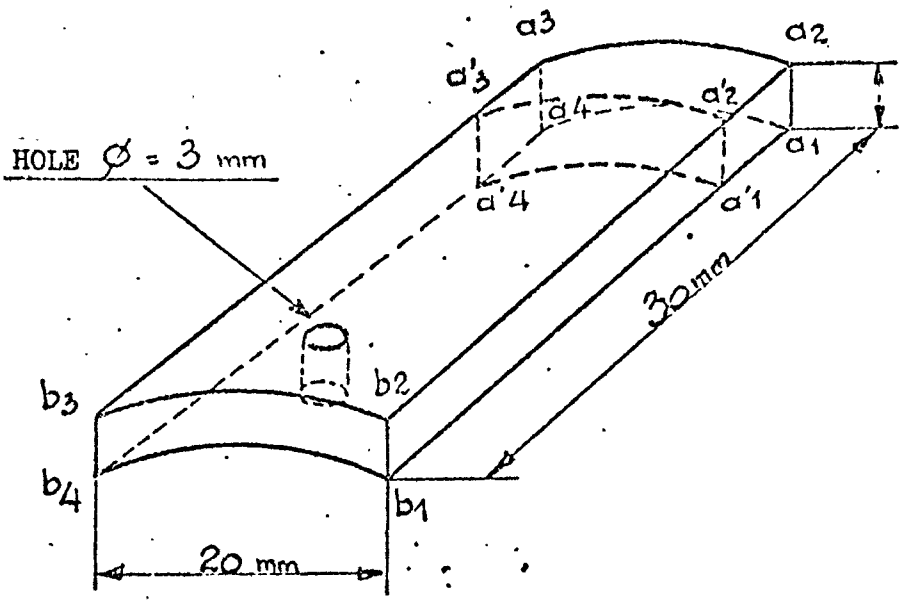

THICKNESS OF THE CYLINDER

Fig. 2

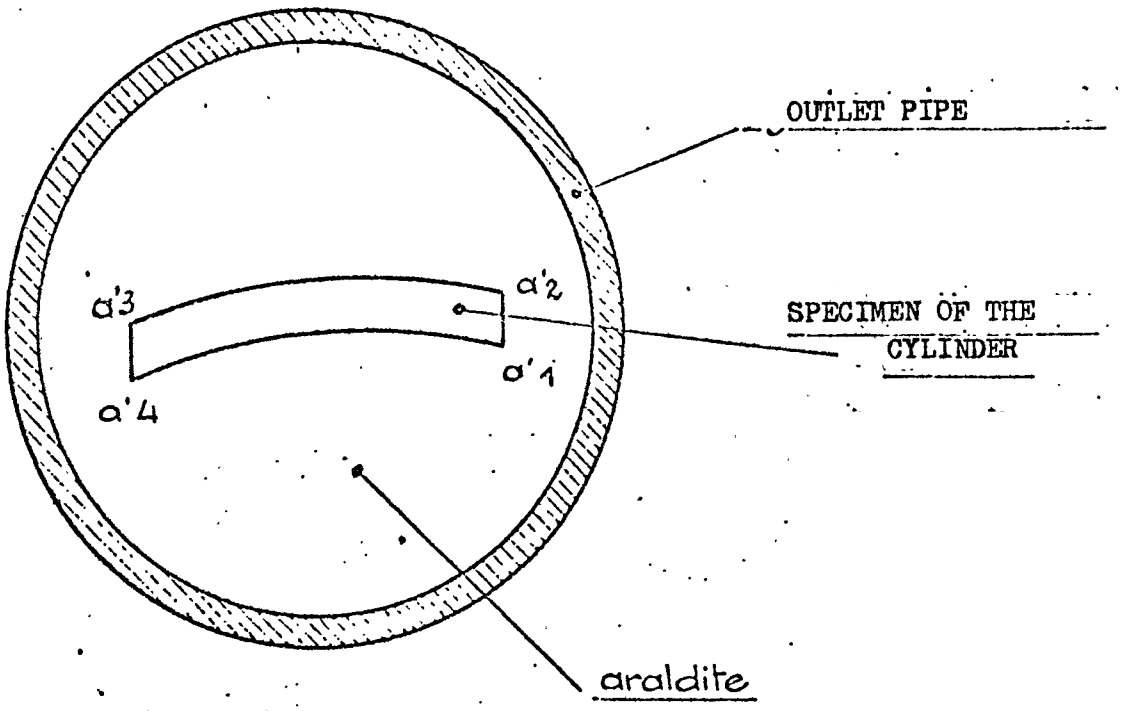

Fig. 3

5. Interpretation of micrographic examination

It is verified that intercrystalline corrosion is superficial, i.e., that its depth does not exceed three grains perpendicularly to the face examined.
However, it is permitted to exceed these values locally provided that they occur in not more than four fields of examination at x300 magnification.

II. TESTS TO EVALUATE REDUCTION IN WALL THICKNESS AS A RESULT OF CORROSION BY WEATHER

The examination of corrosion behaviour must be completed by a test on finished cylinders and/or specimens from finished cylinders to expose them to the most corrosive weather likely to occur during the utilization of the cylinders.

This test should permit the evaluation of any reduction in wall cylinders, during the cylinder's average life.

The value so obtained must not exceed 0.2 mm.
However, a reduction in thickness exceeding 0.2 mm is allowed provided that an equivalent extra thickness is taken into consideration when the wall thickness of the cylinder resistant shell is calculated.

A N N E X III

EEC APPROVAL CERTIFICATE

Inspection authority:

Application of Council Directive N° ..... of .....

Date:

Designation of cylinders:

EEC approval code N° .....
Capacity:.....

Manufacturer's name and address:

The undersigned hereby declares that he has checked that the verifications, tests and inspections, laid down in Point 4.2 of Annex I to the Council Directive N° ..... of .....have been carried out succesfully.

cylinders of the accepted design { -are / -are not } subject to EEC inspection

General remarks: the drawing which was the subject of approval is annexed hereto.

Signed and certified this ..... day of .....
at .....

.....
(signature and capacity)

A N N E X IV

EEC INSPECTION CERTIFICATE

Inspection authority:

Application of Council Directive N° ..... of .....
EEC approval code N° .....
EEC inspection code N° .....
Capacity: .....
Production batch N° .....

Date:
Designation of cylinders:

Name and address of the manufacturer:

The undersigned hereby declares that he has checked that the verifications, tests and inspections prescribed in Annex I, Point 5.2, of the Council Directive N° ..... of ..... have been carried out successfully.

Special remarks:

General remarks:

Signed and certified this ..... day of .....
at .....

.....
(signature and capacity)