INTERNATIONAL STANDARD

ISO 4404-1

First edition
2001-12-01

Corrected version
2002-06-01

Petroleum and related products — Determination of the corrosion resistance of fire-resistant hydraulic fluids —

Part 1: Water-containing fluids

Pétrole et produits connexes — Détermination de la résistance à la corrosion de fluides hydrauliques difficilement inflammables —

Partie 1: Fluides contenant de l'eau

Reference number
ISO 4404-1:2001(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 4404 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 4404-1 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This first edition of ISO 4404-1, together with ISO 4404-2, cancels and replaces ISO 4404:1998.

ISO 4404 consists of the following parts, under the general title *Petroleum and related products — Determination of the corrosion resistance of fire-resistant hydraulic fluids*:

— *Part 1: Water-containing fluids*

— *Part 2: Non-aqueous fluids*

Annex A forms a normative part of this part of ISO 4404. Annexes B and C are for information only.

This corrected version of ISO 4404-1:2001 incorporates corrections to

— the Foreword (information regarding the document cancelled and replaced by this part of ISO 4404 has been added), and

— the second paragraph of clause 1 (“HED” has been replaced by “HFD”).

Introduction

Water-containing hydraulic fluids are used in hydraulic systems where fire-resistant fluids are required due to operating conditions. The corrosion resistance of such fluids has to be assessed in order to choose a suitable system design and prepare maintenance instructions. The method established by this part of ISO 4404 is based on CETOP R 48H[1)] and the 7th edition of the Luxembourg report[2)].

1) *Procedure for determining corrosion resistant characteristics of water based fire resistant fluids* (1984).

2) Safety and Health Commission for the Mining and other Extractive Industries, Doc. No. 4746/10/91 EN (for English version, FR for French version), April 1994, *Requirements and tests applicable to fire-resistant hydraulic fluids used for power transmissions and control (hydrostatic and hydrokinetic)*, available from the Commission of the European Communities, Directorate-General V, Unit V.F.4 “Extractive, Iron and Steel Industries”, Bâtiment Jean Monnet, C4/65, L-2920 Luxembourg.

Petroleum and related products — Determination of the corrosion resistance of fire-resistant hydraulic fluids —

Part 1:
Water-containing fluids

WARNING — The use of this part of ISO 4404 may involve hazardous materials, operations and equipment. This part of ISO 4404 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 4404 to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 4404 specifies a test method to determine the influence on metals of fire-resistant fluids in categories HFA, HFB and HFC, as classified in ISO 6743-4. It evaluates the corrosion protection provided by these fluids towards metal components used in hydraulic systems and installations.

A similar technique for fluids in category HFD is described in ISO 4404-2:—[3)].

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 4404. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 4404 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 209-1:1989, *Wrought aluminium and aluminium alloys — Chemical composition and forms of products — Part 1: Chemical composition*

ISO 426-1:1983, *Wrought copper-zinc alloys — Chemical composition and forms of wrought products — Part 1: Non-leaded and special copper-zinc alloys*

ISO 648:1977, *Laboratory glassware — One-mark pipettes*

ISO 752:1981, *Zinc ingots*

ISO 1337:1980, *Wrought coppers (having minimum copper contents of 99,85 %) — Chemical composition and forms of wrought products*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 3819:1985, *Laboratory glassware — Beakers*

3) To be published.

ISO 5598:1985, *Fluid power systems and components — Vocabulary*

ISO 6344-1:1998, *Coated abrasives — Grain size analysis — Part 1: Grain size distribution test*

ISO 6743-4:1999, *Lubricants, industrial oils and related products (class L) — Classification — Part 4: Family H (Hydraulic systems)*

EN 1179:—[4], *Zinc and zinc alloys — Primary zinc*

EN 10083-2:1991, *Quenched and tempered steels — Part 2: Technical delivery conditions for non-alloy steels*

DIN 12331:1988, *Laboratory glassware; beakers*

DIN 51346:1986, *Testing the stability of fire-resistant fluids*

3 Terms and definitions

For the purposes of this part of ISO 4404, the terms and definitions given in ISO 5598 apply.

4 Principle

Test strips of various metals and metal pairs are partially submerged in the test fluid at a specific temperature for a specific period. The change in mass of each test strip, its surface appearance, and the change in the appearance of the fluid during the test are determined.

5 Reagents and materials

5.1 **Acetone**, analytical grade.

5.2 **Heptane**, analytical grade.

5.3 **Water**, conforming to at least grade 2 of ISO 3696.

5.4 **Metallic salts**, listed in Table A.1 in normative annex A, analytical grade[5].

6 Apparatus

Usual laboratory apparatus and glassware, together with the following:

6.1 **Glass beakers (ten required),** of capacity 400 ml, height approximately 135 mm, without a spout, conforming to ISO 3819 (see Figure 1).

6.2 **Glass beaker,** type H 1000, conforming to DIN 12331 (of capacity 1 000 ml).

6.3 **Pipette,** complying with ISO 648, class A.

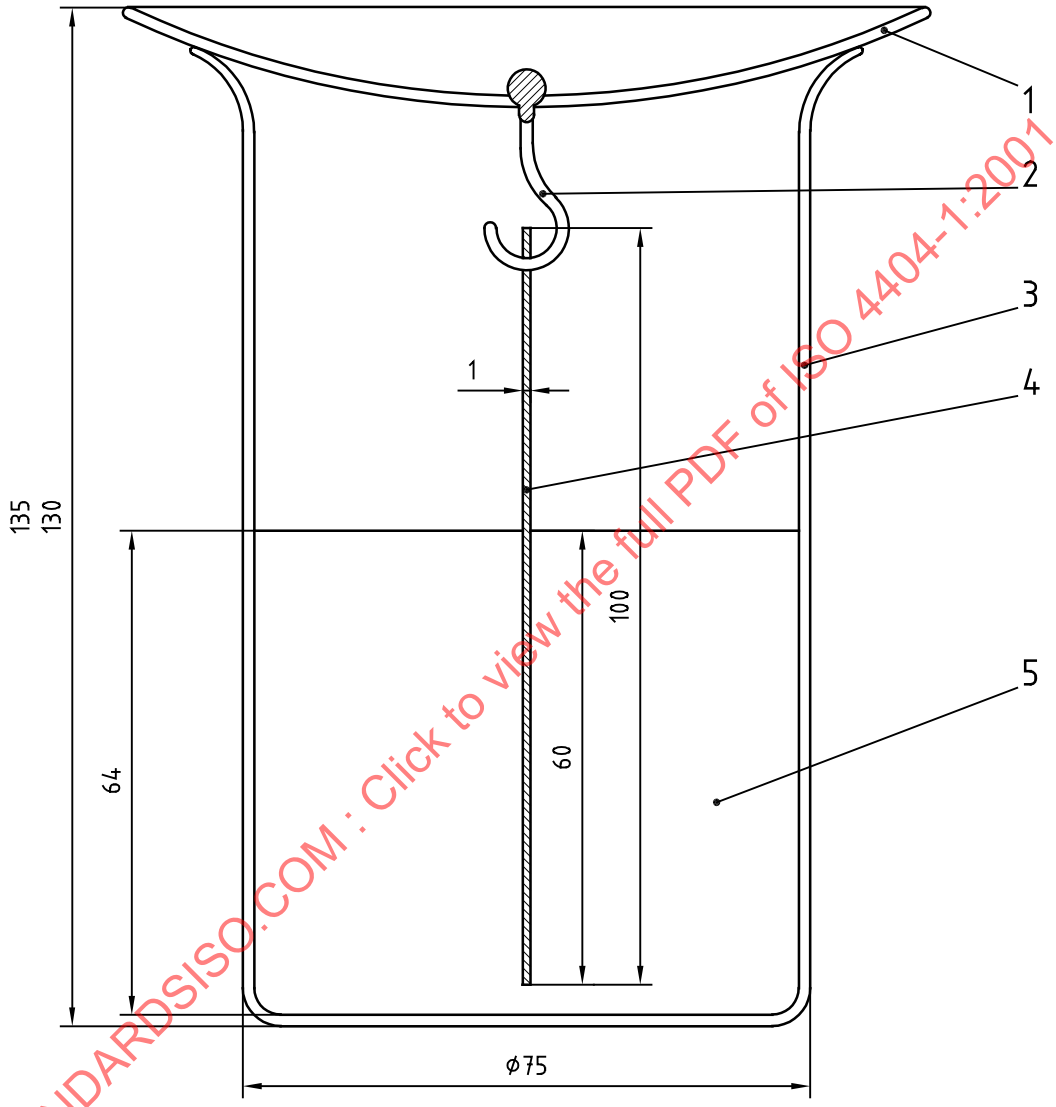
4) To be published. (Revision of EN 1179:1995)

5) For a specification of the reagents, see for example, national pharmacopoeias.

6.4 Watch-glasses (ten required), for covering the beakers (6.1), with a hole in the centre for suspending glass hooks (see Figures 1 and 2).

6.5 Glass hooks, allowing free suspension of the test strips in the beaker and formed in such a way that the hole in the watch-glass will be closed by the suspension device (see Figures 1 and 2).

Dimensions in millimetres

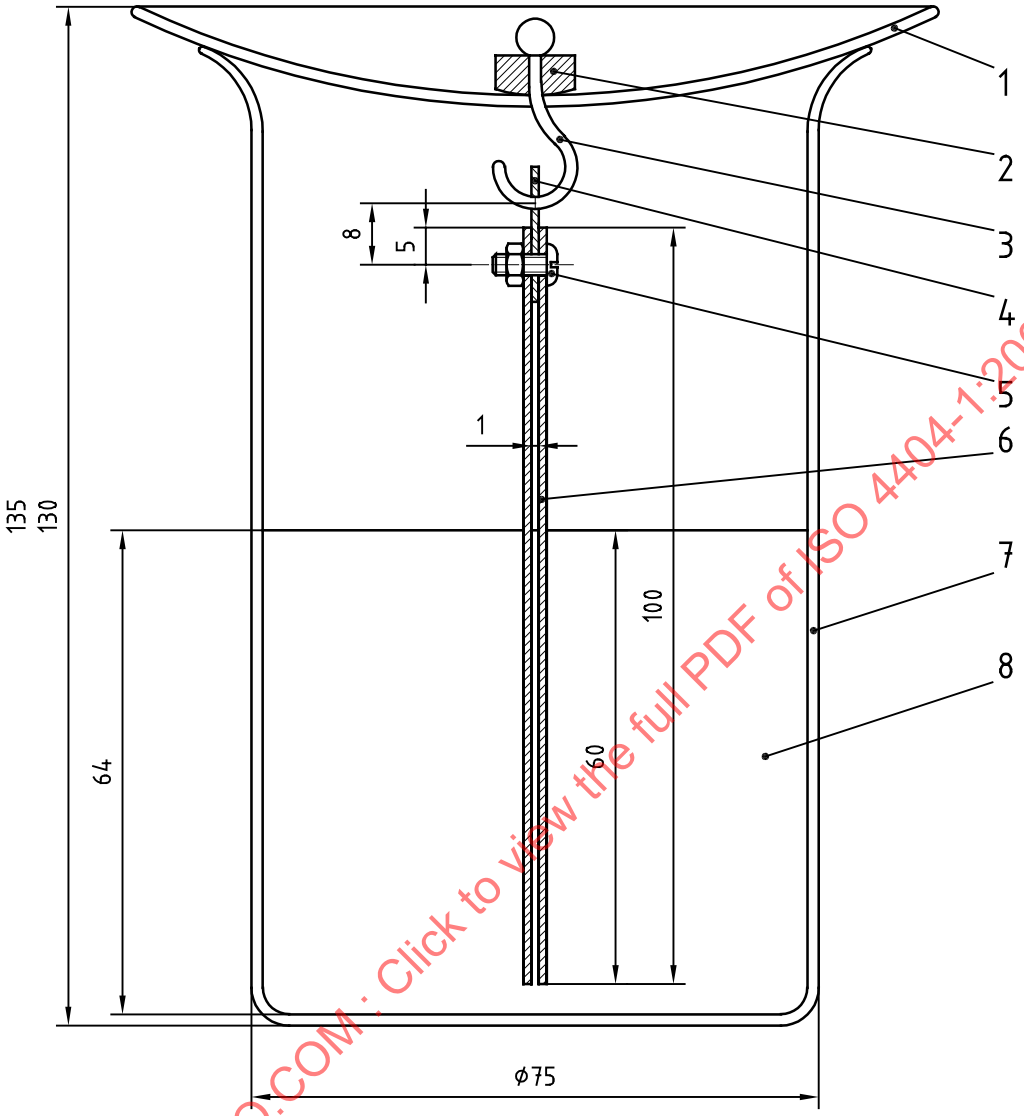


Key

1 Watch-glass
2 Glass hook
3 Beaker without a spout
4 Test strip
5 Test fluid

Figure 1 — Assembly for a single test strip

Dimensions in millimetres



Key

1 Watch-glass
2 Shim
3 Glass hook
4 Nylon spacer (15 × 10 × 1)
5 Nylon bolt and nut, diameter 4
6 Pair of test strips
7 Beaker without a spout
8 Test fluid

Figure 2 — Assembly for a pair of test strips

6.6 Heating bath or oven, thermostatically controlled and capable of maintaining the test fluids at 35 °C ± 1 °C. If a heating bath is used, it shall be equipped to give adequate stirring to ensure even temperature distribution.

6.7 Analytical balance, accurate to 0,000 2 g.

6.8 Abrasive paper, corundum/haematite/quartz (commercially known as “emery”), of various grades of fineness, including P 120, P 400 and P 600 grit designation according to ISO 6344-1.

6.9 Cotton wool.

6.10 Tweezers, suitable for handling the test strips.

6.11 Grinding wheel (fine), rotating at approximately 1 400 r/min.

6.12 Test strips, of the materials listed in Table 1, measuring 100 mm × 20 mm × 1 mm and having a hole of 4 mm diameter at one end for suspending on the glass hook (6.5).

NOTE 1 Test strips measuring 100 mm × 20 mm × 2 mm may also be used.

NOTE 2 This test may be performed with any other material (metal and/or alloy) used in hydraulic systems, provided that the dimensions of the test strips are observed as specified in this subclause.

6.13 Spacer, of nylon, rectangular, 15 mm × 10 mm × 1 mm, with two holes of 5 mm diameter, for the glass hook (6.5) and bolt (6.14) (see Figure 2).

6.14 Bolt and nut, of nylon, diameter 4 mm, length 15 mm (see Figure 2).

Table 1 — Specifications of standard test materials for test strips

Metal	Composition % (*m/m*)	References	
		ISO (grade)	EN (grade)
Steel	Fe 98,58 to 98,98 C 0,35 to 0,45 Mn 0,5 to 0,8 Si max. 0,10 S max. 0,035 P max. 0,035	—	10083-2 (C45)
Copper	Cu 99,9 (electrolyte copper)	1337 (Cu ETP or Cu FRHC)	—
Brass	Cu 65 Zn 35	426-1 (Cu Zn 35)	—
Zinc	Zn 99,5 (pure zinc)	752 (99,5)	1179 (Z4)
Aluminium	Al 99,5 (pure aluminium)	209-1 (Al 99,5/1050A)	—
NOTE For standards that are equivalent to those referenced in this table, see informative annex B.			

7 Producing the test fluid from an HFA type fluid concentrate

Mix the concentrate and the test water No. 1, No. 2 or No. 3, whose characteristics and preparation are specified in annex A, in the proportion recommended by the supplier of the concentrate.

The type of test water used, chosen from annex A, shall be of the highest hardness still able to form a stable emulsion, i.e. an emulsion which satisfies the ratings 1A and 1R of DIN 51346.

Measure 600 ml of the test water into a glass beaker (6.2). With constant stirring, add the required amount of the concentrate in doses using the pipette (6.3) within 10 min. After the whole concentrate has been added, stirring shall continue for 5 min more, and then 250 ml of the test fluid shall be measured immediately into the prepared beakers (see 8.1.5).

Start the test on the day of mixing.

8 Procedure

8.1 Preparation for the test

8.1.1 Before polishing the test strips, carefully remove any burrs from the edges of the strips (6.12) using the grinding wheel (6.11).

8.1.2 Polish the test strips with suitable grades of emery paper (6.8), e.g. in the order of P 120, P 400 and finally P 600, until a smooth surface is achieved.

8.1.3 Subsequently, handle test strips exclusively with tweezers (6.10). Rub the test strips with dry cotton wool (6.9) and then with cotton wool soaked in heptane (5.2).

8.1.4 When all traces of solvent have evaporated, weigh the test strips and record their mass to the nearest 1 mg.

8.1.5 Fill each of the ten beakers (6.1) with 250 ml of the homogenized test fluid.

8.1.6 In five of these beakers, suspend single test strips of steel, copper, zinc, aluminium and brass on glass hooks (6.5) in such a way that approximately 60 mm of their entire length is immersed in the test fluid (see Figure 1).

NOTE These single test strips may also be suspended with the spacer (6.13) and the bolt (6.14).

8.1.7 In another four of the beakers (6.1), immerse the following pairs of test strips:

a) steel and zinc;

b) copper and zinc;

c) zinc and aluminium;

d) aluminium and steel.

8.1.8 Suspend these pairs of test strips on glass hooks (6.5) in such a way that 60 mm of their length is immersed in the test fluid and so that, by means of the spacer (6.13), the distance between the strip surfaces is 1 mm.

8.1.9 Do not suspend a test strip in the remaining tenth beaker (6.1) because it is used to evaluate the changes in the fluid during the test.

8.1.10 Cover all the beakers with a watch-glass (6.4) to minimize test-fluid evaporation losses.

8.2 Test

8.2.1 Place all ten beakers in the bath or oven (6.6). Maintain the temperature of the test fluid at 35 °C ± 1 °C.

8.2.2 Leave all ten beakers undisturbed in the bath or oven for a period of 672 h ± 2 h (equivalent to 28 days).

8.3 Assessment

8.3.1 After completion of the test, visually inspect the test strips and the test fluid under normal light (approximately 646 lx) and assess the following:

a) test strips:

— general appearance;

— deposits formed;

b) test fluid:

— colour;

— appearance;

— deposits.

Handle the test strips only with the tweezers (6.10).

Rate the appearance of metal strips according to Table 2.

Table 2 — Rating system for metal strips

Rating	Description
0	no effect
1	slight colour change or oxidation of less than 20 % of the surface
2	strong colour change
3	deposits or oxidation on more than 20 % of the surface
4	corrosion or pitting
5	other effects to be specified

Rate the effects on the fluids according to Table 3.

Table 3 — Rating system for the fluids

Rating	Description
0	no effect
1	deposits
2	separation, e.g. surface accumulation of oil, or distinct phase separation
3	cloudiness of an initially clear fluid
4	colour change
5	other effects to be specified

8.3.2 After the visual assessments, clean the test strips by immersing them in beakers containing solvent, and moving the strips in the solvent until all the test fluid is removed.

Use the following fluid/solvent combination:

a) test fluids HFA and HFB: first heptane (5.2), then acetone (5.1);

b) test fluids HFC: first water (5.3), then acetone (5.1).

NOTE Some HFA fluids may require a wash in water prior to heptane and acetone.

If, after the immersion, test fluid is still visible on the surface of the test strip, re-immerse the strip in the solvents. If necessary, repeat this procedure.

After immersion in acetone, dry the test strips, still handled with tweezers, in air until all traces of acetone are evaporated.

After the test strips have been cleaned and dried, weigh each one and record their mass to the nearest 1 mg.

9 Expression of results

9.1 Report the results of the visual inspection (see 8.3.1).

9.2 Report the difference in mass of each test strip used, indicating increases during the test by "+" and decreases during the test by "–" between the masses recorded in 8.1.4 and 8.3.2.

NOTE For interpretation of the results, see the explanatory comments in informative annex C.

10 Precision

The precision of this method has not yet been established.

11 Test report

The test report shall contain at least the following information:

a) a reference to this part of ISO 4404;

b) the type and complete identification of the product tested;

c) the results of the test (see clause 9);

d) the type of test water used (see clause 7);

e) any deviation, by agreement or otherwise, from the procedure specified;

f) the date of the test.

Annex A
(normative)

Preparation of test waters

Prepare at least 2,5 litres of test water for each test using suitable amounts of salts as given in Table A.1.

Table A.1 — Composition of test waters

Salts to be added	Concentration mg/l	Ion concentration mg/l		Hardness
Test water No. 1				
Sodium hydrogen carbonate $NaHCO_3$	90	24,6 Na^+	65,4 HCO_3^-	= 87 mg/kg temporary hardness as $Ca(HCO_3)_2$
Magnesium chloride hexahydrate $MgCl_2 \cdot 6H_2O$	145	17,3 Mg^{2+}	50,6 Cl^-	= 71,3 mg/kg permanent hardness as $CaCO_3$
Calcium sulfate dihydrate $CaSO_4 \cdot 2H_2O$	220	51,3 Ca^{2+}	122,8 SO_4^{2-}	= 128,1 mg/kg permanent hardness as $CaCO_3$
Sodium sulfate Na_2SO_4	40	13,0 Na^+	27,0 SO_4^{2-}	
Sodium chloride NaCl	245	96,3 Na^+	148,7 Cl^-	
Sodium nitrate $NaNO_3$	27	7,3 Na^+	19,7 NO_3^-	
Test water No. 2				
Sodium hydrogen carbonate $NaHCO_3$	255	69,8 Na^+	185,5 HCO_3^-	= 245,9 mg/kg temporary hardness as $Ca(HCO_3)_2$
Magnesium chloride hexahydrate $MgCl_2 \cdot 6H_2O$	420	250,2 Mg^{2+}	146,6 Cl^-	= 206,8 mg/kg permanent hardness as $CaCO_3$
Calcium sulfate dihydrate $CaSO_4 \cdot 2H_2O$	260	60,6 Ca^{2+}	145,1 SO_4^{2-}	= 151,3 mg/kg permanent hardness as $CaCO_3$
Sodium chloride NaCl	90	35,4 Na^+	54,6 Cl^-	
Sodium nitrate $NaNO_3$	27	7,3 Na^+	19,7 NO_3^-	
Test water No. 3				
Magnesium sulfate heptahydrate $MgSO_4 \cdot 7H_2O$	924	91,2 Mg^{2+}	360,1 SO_4^{2-}	= 375,5 mg/kg permanent hardness as $CaCO_3$
Calcium sulfate dihydrate $CaSO_4 \cdot 2H_2O$	645	150,4 Ca^{2+}	360,0 SO_4^{2-}	= 375,4 mg/kg permanent hardness as $CaCO_3$
Sodium chloride NaCl	330	130,0 Na^+	200,0 Cl^-	

First, dissolve the calcium sulfate in the water (5.3) using an appropriate calibrated flask.

NOTE Calcium sulfate is hard to dissolve and may require two days of stirring. It is generally delivered as a fine powder. If not, it can be helpful to grind it.

After the dissolution of calcium sulfate has been completed, dissolve the other metallic salts in turn.

During the agitation, the flask shall be kept stoppered.

The shelf-life of the test water is 24 h maximum.

Table A.2 — Characteristics of test waters

Test-water characteristics	Units	Test waters			Comments
		No. 1	No. 2	No. 3	
Total hardness ($Ca^{2+} + Mg^{2+}$)	mg/kg mg/kg	199,4 111,8	358,1 200	750,9 421	$CaCO_3$ equivalent CaO equivalent
Temporary hardness (alkalinity) (HCO_3^- acid capacity)	mg/kg	87	245,9	—	$Ca(HCO_3)_2$ equivalent
Mg^{2+} ions	mg/kg	17	50	91	
Ca^{2+} ions	mg/kg	51	61	150	
SO_4^{2-} ions	mg/kg	150	145	720	
NO_3^- ions	mg/kg	20	20	—	
Cl^- ions	mg/kg	199	201	200	
Na^+ ions	mg/kg	141	113	130	
Alkaline earth	mmol/l	1,99	3,58	7,5	$Ca^{2+} + Mg^{2+}$