INTERNATIONAL STANDARD

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Petroleum and related products — Determination of the ageing behaviour of inhibited oils and fluids — TOST test —

Part 3: Anhydrous procedure for synthetic hydraulic fluids

Pétrole et produits connexes — Détermination du comportement au vieillissement des fluides et huiles inhibés — Essai TOST —

Partie 3: Méthode anhydre pour les fluides hydrauliques synthétiques

Reference number
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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 2.

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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 4263-3 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

ISO 4263 consists of the following parts, under the general title *Petroleum and related products — Determination of the ageing behaviour of inhibited oils and fluids — TOST test*:

— *Part 1: Procedure for mineral oils*

— *Part 2: Procedure for category HFC hydraulic fluids*

— *Part 3: Anhydrous procedure for synthetic hydraulic fluids*

— *Part 4: Procedure for industrial gear oils*

Petroleum and related products — Determination of the ageing behaviour of inhibited oils and fluids — TOST test —

Part 3: Anhydrous procedure for synthetic hydraulic fluids

WARNING — The use of this part of ISO 4263 may involve hazardous materials, operations and equipment. This part of ISO 4263 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 4263 to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 4263 specifies a method for the determination of the ageing behaviour of synthetic hydraulic fluids of categories HFDR, HFDU, HEES and HEPG as defined e.g. in ISO 12922[4] and ISO 15380[5]. The ageing is accelerated by the presence of oxygen and metal catalysts at elevated temperature, and the degradation of the fluid is followed by changes in acid number. Other parts of ISO 4263 specify similar procedures for the determination of ageing behaviour of mineral oils and specified categories of fire-resistant fluids used in hydraulic and other applications.

NOTE Other signs of fluid deterioration, such as the formation of insoluble sludge, catalyst coil corrosion or decrease in viscosity, can occur, which indicate oxidation of the fluid, but are not reflected in the calculated oxidation lifetime. The correlation of these occurrences with field service is under investigation.

This test method can be used to compare the oxidation stability of fluids that are not prone to contamination with water. However, because of the large number of individual field-service applications, the correlation between the results of this test and actual service performance can vary markedly, and is best judged on experience.

The precision of this test method for synthetic hydraulic fluids is not known because interlaboratory data are not available. This method might not be suitable for use in specifications or in the event of disputed results as long as these data are not available. However, precision for inhibited turbine oils is given in Clause 11 for guidance as an indication of the precision that could be obtained for synthetic hydraulic fluids.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3170:2004, *Petroleum liquids — Manual sampling*

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

ISO 7537:1997, *Petroleum products — Determination of acid number — Semi-micro colour-indicator titration method*

3 Principle

A test portion is reacted, in the absence of light, at 95 °C with oxygen and a steel and copper catalyst coil. Small aliquots of the fluid are withdrawn at regular intervals and the acid number is measured (see the Note in Clause 1). The test is continued until an acid number increase of 2,0 mg of potassium hydroxide (KOH) per gram of test portion is reached and the number of hours is recorded as the oxidation life. For some requirements, the test may be discontinued at a fixed number of hours (e.g. 500 h or 1 000 h) when the value of the acid number is still below 2,0 mg of KOH per gram of test portion.

4 Reagents and materials

4.1 Water, unless otherwise specified, in accordance with the requirements of grade 2 of ISO 3696:1987. Potable water means tap water, unless normal piped supplies are contaminated with particulate or highly soluble mineral content.

4.2 Heptane (C_7H_{16}), of minimum purity 99,75 %.

4.3 Acetone (CH_3COCH_3), of general purpose reagent grade (GPR).

4.4 Propan-2-ol ($CH_3CHOHCH_3$), of general purpose reagent grade (GPR).

4.5 Oxygen, of minimum purity 99,5 %, supplied through a pressure-regulation system adequate to maintain the specified flow rate throughout the test duration.

Supply from an oxygen cylinder should be via a two-stage regulation system and a needle valve to improve the consistency of gas-flow regulation.

WARNING — Use oxygen only with equipment validated for oxygen service. Do not allow oil or grease to come into contact with oxygen and clean and inspect all regulators, gauges and control equipment. Check the oxygen-supply system regularly for leaks. If a leak is suspected, turn off immediately and seek qualified assistance.

4.6 Cleaning solutions

4.6.1 Strong oxidizing acid solution

The reference strong oxidizing cleaning solution on which precision was based, is chromosulfuric acid (see the following warning), but alternative non-chromium containing solutions, such as ammonium persulfate in concentrated sulfuric acid (8 g/l) have been found to give satisfactory cleanliness. A 10 % solution of three parts of hydrochloric acid (1 mol/l) and one part of orthophosphoric acid (concentrated GPR grade) removes iron oxide deposits.

WARNING — Chromosulfuric acid is a health hazard. It is toxic, a recognized carcinogen as it contains Cr(VI) compounds, highly corrosive and potentially hazardous in contact with organic materials. When using chromosulfuric acid cleaning solution, eye protection and protective clothing are essential. Never pipette the cleaning solution by mouth. After use, do not pour cleaning solution down the drain, but neutralize it with great care owing to the concentrated sulfuric acid present, and dispose of it in accordance with standard procedures for toxic laboratory waste (chromium is highly dangerous to the environment).

Strongly oxidizing acid cleaning solutions that are chromium-free are also highly corrosive and potentially hazardous in contact with organic materials, but do not contain chromium which has special disposal problems.

4.6.2 Surfactant cleaning fluid

A proprietary strong surfactant cleaning fluid is a preferred alternative.

4.6.3 Laboratory detergent

The detergent shall be water soluble.

4.7 Catalyst wires

4.7.1 Low-metalloid steel wire, of diameter 1,60 mm ± 0,05 mm, made of carbon steel, soft bright annealed and free from rust.

4.7.2 Copper wire, of diameter 1,63 mm ± 0,05 mm, made of either electrolytic copper wire of 99,9 % minimum purity or soft copper wire of an equivalent grade.

4.8 Abrasive cloth, made of silicon carbide of 150 µm (100-grit) with cloth backing, or an equivalent grade of abrasive cloth.

4.9 Absorbent cotton

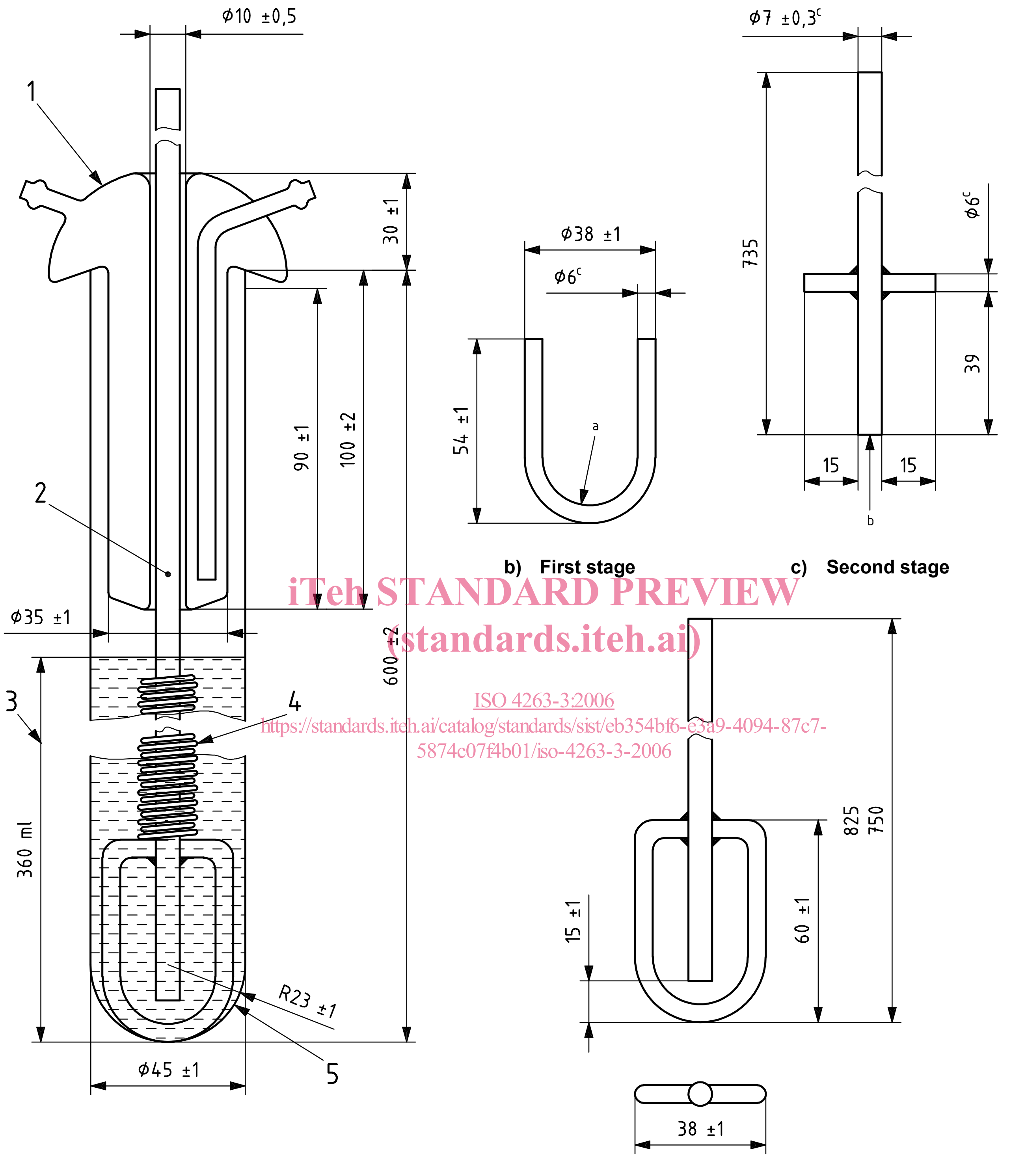
5 Apparatus

5.1 Oxidation cell, consisting of a large test tube of borosilicate glass with a graduation mark to indicate a volume of 300 ml ± 1 ml at 20 °C. A mushroom condenser and oxygen-delivery tube, also of borosilicate glass, fit into the test tube. The design and dimensions shall be as illustrated in Figure 1.

5.2 Heating bath, consisting of a thermostatically controlled bath capable of maintaining the hydraulic fluid test portion in the oxidation cell at 95,0 °C ± 0,2 °C. It shall be large enough to hold the required number of oxidation cells (5.1) immersed in the heat transfer medium to a depth of 355 mm ± 10 mm. It shall be constructed to ensure that light is excluded from the test portions during the test. If a fluid bath is used, it shall be fitted with a suitable stirring system to provide a uniform temperature throughout the bath. If the fluid bath is fitted with a top, the total length of the oxidation cell within the bath shall be 390 mm ± 10 mm. If a metal-block bath is used, the heaters shall be distributed so as to produce a uniform temperature throughout the bath, and the holes in the block shall have a minimum diameter of 50 mm and a depth, including any insulating cover, of 390 mm ± 10 mm.

5.3 Flowmeter, capable of measuring 3,0 l/h with an accuracy of ± 0,1 l/h.

Dimensions in millimetres (unless otherwise indicated)



a) Apparatus

d) Final stage

Key

1 glass condenser
2 oxygen-delivery tube
3 fluid sample
4 catalyst coil
5 radius of bottom cell

[a] Bend over mandrel of diameter 26.
[b] Grind end of tube.
[c] External diameter.

Figure 1 — Oxidation cell

5.4 Temperature-measurement devices

5.4.1 Heating bath. The temperature in liquid heating baths shall be measured by either a liquid-in-glass thermometer meeting the requirements of the specification given in Annex A, or an equivalent temperature-measurement system readable to ± 0,1 °C and graduated in 0,1 °C increments. For metal-block heating baths, a temperature-measurement system, with possibly more than one device of the same readability and accuracy, is required.

5.4.2 Oxidation cell. The temperature in the oxidation cell shall be measured by either a liquid-in-glass thermometer meeting the requirements of the specification given in Annex A, or an equivalent temperature-measurement system readable to ± 0,1 °C and calibrated to better than ± 0,1 °C.

5.4.3 Thermometer bracket. If a liquid-in-glass thermometer is used in the oxidation cell, it shall be suspended by means of a bracket as illustrated in Figure 2. The thermometer is held in the bracket by either two fluoro-elastomer O-rings of approximately 5 mm diameter, or by the use of thin stainless steel wire.

5.5 Wire-coiling mandrel, as illustrated in Figure 3, is used to produce the double spiral of copper and steel wire. The mandrel is included in a suitable winding device.

5.6 Oxygen-supply tube, flexible polyvinylchloride (PVC) tubing of approximately 6,4 mm inside diameter and 1,5 mm wall thickness, is required to deliver oxygen to the oxidation cell.

5.7 Aliquot-removal devices. Depending on the size and frequency of removal of aliquots of the test portion for analysis, a selection of devices are required. Glass syringes, fitted with Luer connectors and stainless steel needles, or long pipettes fitted with suitable pipette fillers, are suitable. These may be inserted via a sampling tube (5.9) fitted through the condenser. Aliquot sizes will generally be in the range of 2 ml to 10 ml, and the devices shall be capable of removing the required aliquot ± 0,2 ml.

5.8 Aliquot containers. Small, dark glass vials of 5 ml to 10 ml capacity, fitted with close-fitting polyethylene caps, are required.

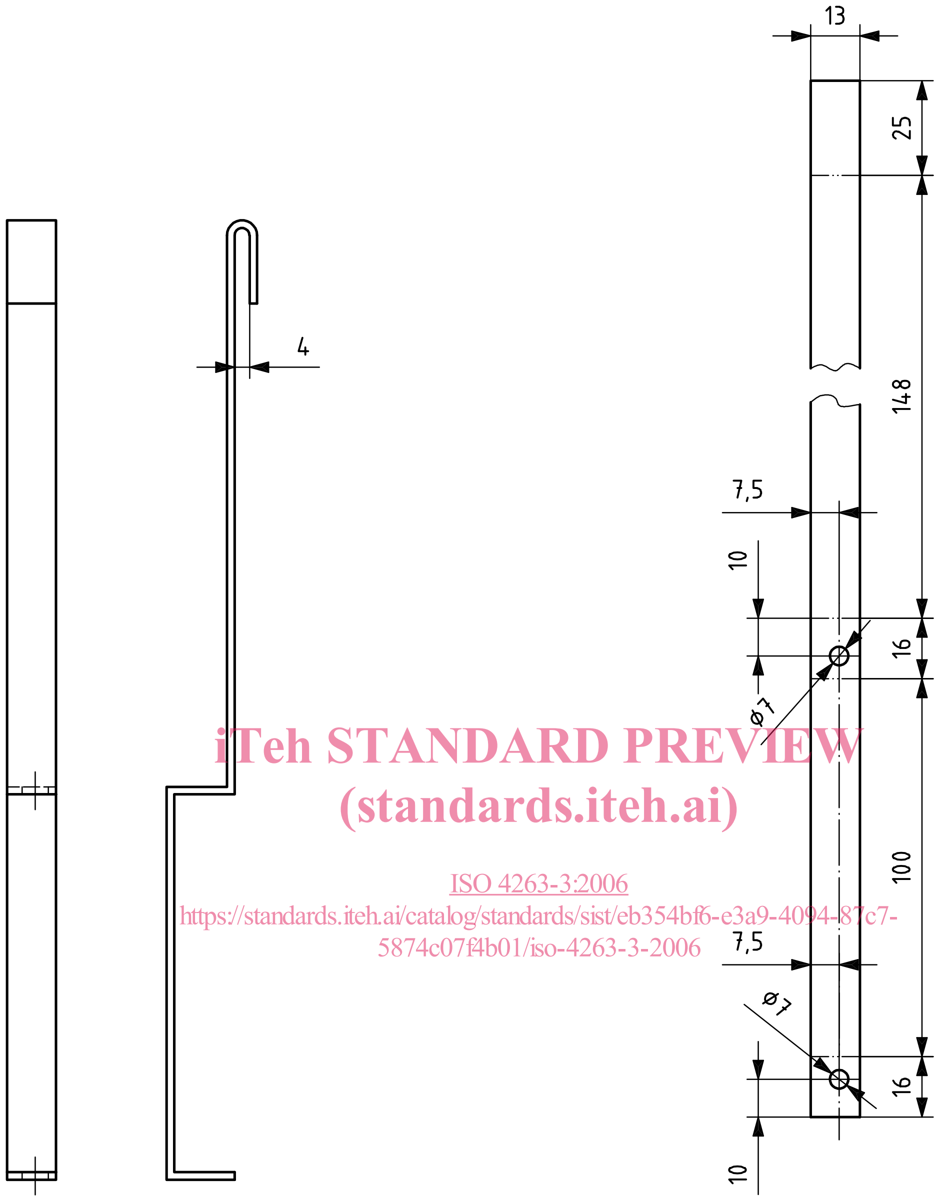
5.9 Sampling tube. Stainless steel tubing, 2,11 mm in outside diameter, 1,60 mm in inside diameter, 610 mm ± 2 mm long, with one end finished at 90° and the other end fitted with an optional female Luer connector (if using syringes as aliquot-removal devices (5.7)). The optional connector is preferably of elastomeric material such as poly(fluorovinyl chloride) to provide a good seal with the syringe.

5.10 Stopper, for the optional Luer fitting of the sampling tube (5.9), made of polytetrafluoroethylene (PTFE) or poly(fluorovinyl chloride).

5.11 Sampling tube holder, for supporting the sampling tube (5.9), made of methyl methacrylate resin, having the dimensions shown in Figure 4.

5.12 Sampling tube spacer, for positioning the end of the sampling tube (5.9) above the sampling tube holder (5.11), made of plastic tubing of poly(vinyl chloride), polyethylene, polypropylene, or polytetrafluoroethylene, having an inside diameter of approximately 3 mm and 51 mm ± 1 mm in length.

Dimensions in millimetres



a) **Bracket (finished)**

b) **Development of bracket**

Material:18/8 (0,792 mm) stainless steel

Figure 2 — Thermometer bracket

Dimensions in millimetres
(unless otherwise indicated)

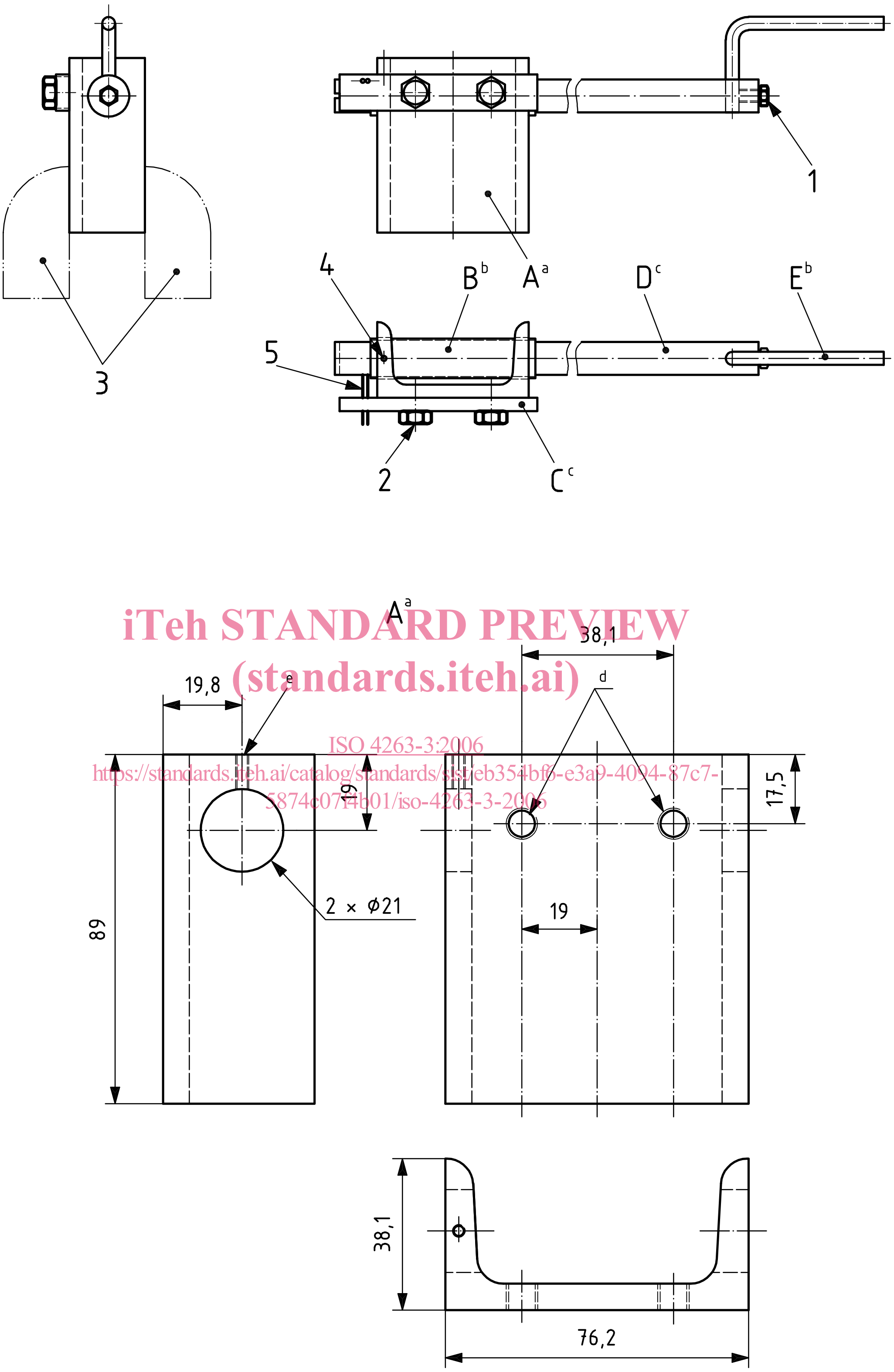


Figure 3 — Catalyst coil mandrel