INTERNATIONAL STANDARD

ISO 9114

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Crude petroleum — Determination of water content by hydride reaction — Field method

Pétrole brut — Dosage de l'eau par la méthode à l'hydrure — Méthode de terrain

Reference number
ISO 9114:1997(E)

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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.

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.

International Standard ISO 9114 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*, Subcommittee SC 6*, Bulk cargo transfer, accountability, inspection and reconciliation*.

Annex A forms an integral part of this International Standard.

Crude petroleum — Determination of water content by hydride reaction — Field method

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

1 Scope

This International Standard specifies a field method for the determination of water in crude petroleum, at concentrations ranging from 0,05 %(*V/V*) to 2 %(*V/V*). It is applicable where due to circumstances the laboratory methods cannot be used. This method is not intended for custody transfer but could be so used if prior agreement from all parties is obtained.

NOTE — For the purposes of this International Standard, the expression "%(*V/V*)" is used to represent the volume fraction.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3170:1988, *Petroleum liquids — Manual sampling.*

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling.*

ISO 3733:1976, *Petroleum products and bituminous materials — Determination of water — Distillation method.*

ISO 3734:1997, *Petroleum products — Determination of water and sediment in residual fuel oils — Centrifuge method.*

3 Principle

After homogenizing the crude petroleum with a mixer an aliquiot is transferred to the reaction flask where calcium hydride reacts with the water contained therein according to:

$$CaH_2 + 2H_2O \rightarrow Ca(OH)_2 + 2H_2 \uparrow$$

The quantity of hydrogen released is proportional to the amount of water present, and is measured volumetrically enabling the percentage by volume of water in the sample to be calculated.

4 Reagents and materials

4.1 Capsules, each containing 0,6 g to 0,7 g calcium hydride in powder form.

4.2 Light aromatic-free mineral oil (medicinal type), used as balancing liquid surplus in the gas burette and reservoir [density at 15 °C: between 830 kg/m^3 and 850 kg/m^3; viscosity: between 14 mm^2/s and 30 mm^2/s (14 cSt and 30 cSt) at 20 °C].

4.3 Wetting agent (e.g. sodium dioctyl sulfosuccinate, technical).

4.4 Kerosine, clear and bright.

4.5 Acetone.

4.6 Heptane.

4.7 Water-free silicone grease.

5 Apparatus

The apparatus is illustrated in figure 1 and consists of the following parts:

5.1 Reaction flask: a 100 ml vessel graduated at volumes corresponding to 10 ml, 20 ml and 40 ml.

5.2 Tap R_1, consisting of a stopper impervious to hydrocarbons, fitting the flask and through which a PTFE device is fitted, consisting of two channels, one of which ends in a two-way tap, whilst the other links the flask with the gas burette (figure 2).

5.3 Gas burette, graduated from 0 % to 1,0 % H_2O in 0,01 % by volume scale units, the upper opening of which is fitted with a tap R_2 linked to tap R_1 by a flexible plastic tube, impervious to hydrocarbons.

The 1 % graduation mark corresponds to the volume of gas that under the reference conditions (20 °C and 101,325 kPa) would be released by the calcium hydride reaction on 20 ml of a sample containing 1,0 % water.

Tap R_2 has two positions (figure 3):

— position "A" in which the gases from reaction flask and burette can be released to the atmosphere;

— position "B" in which the contents are isolated from the surrounding atmosphere.

5.4 Reservoir, linked to the base of the gas burette by a flexible plastic tube, impervious to hydrocarbons and filled with a liquid (4.2) to balance the pressures inside the apparatus.

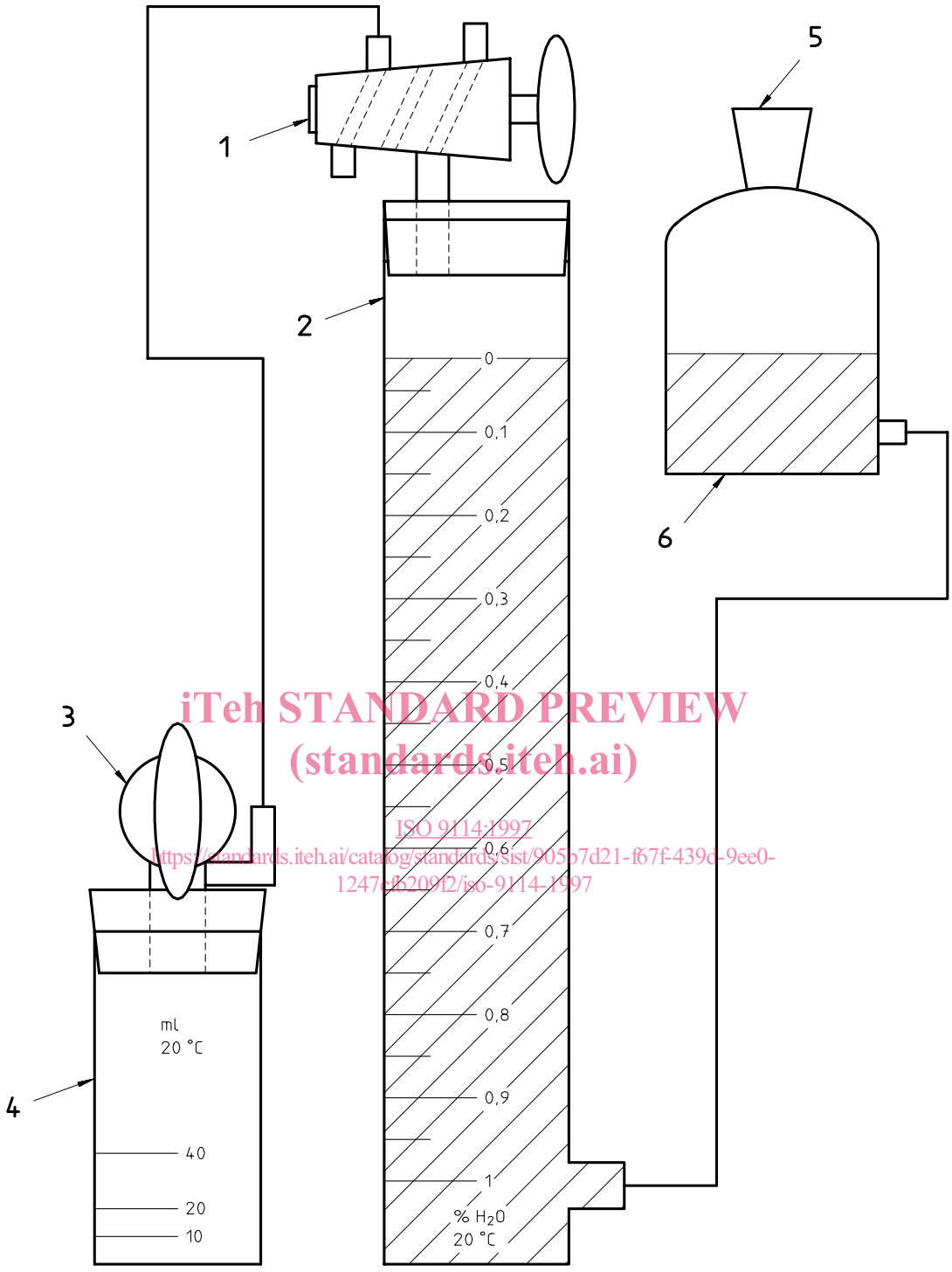
The height of the reservoir shall be adjustable in order to obtain the required balance of liquid.

The stopper of the reservoir has two positions (figure 4):

— position "α" (open): the "working" position in which the stopper is open to the surrounding atmosphere,

— position "β" (closed): in which the flask is isolated from the atmosphere (storage and carrying position).

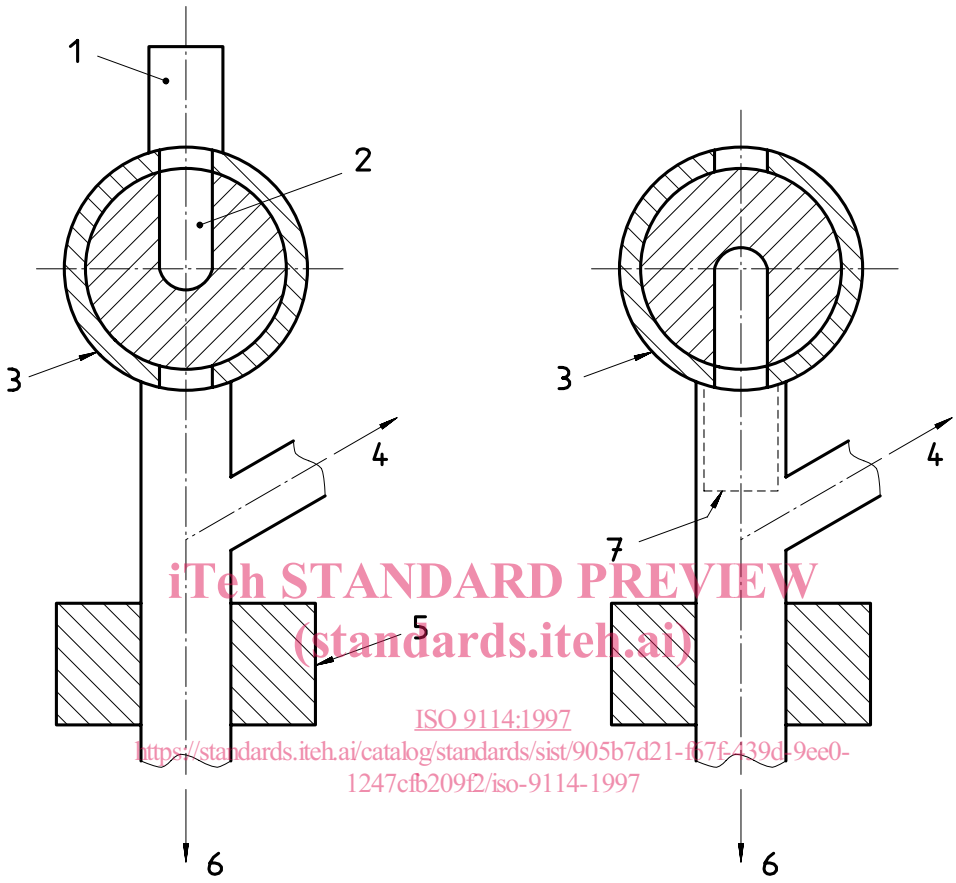
For convenience, the apparatus should be designed to fit compactly into a portable case or box.



Key

1 Tap R
2 Gas burette
3 Tap R_1
4 Reaction flask
5 Plug with two positions
6 Reservoir

Figure 1 — Diagram of the apparatus



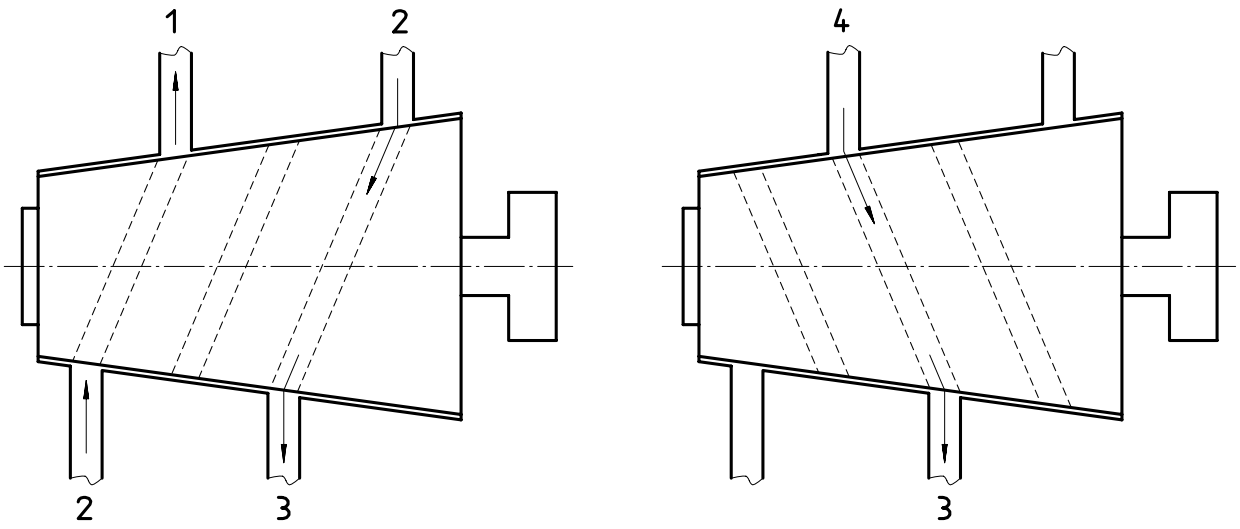
Position a for the lever
Introducing capsules
in the tap

Position b for the lever
Capsule drops into
the reaction flask

Key

1 Lever to turn tap R_1 by 180°
2 Opening for capsules
3 Tap
4 To gas burette
5 Plug to close the reaction flask
6 To reaction flask
7 Tap lever

Figure 2 — Tap R_1 (plug and device with two channels)



A

Position open to air

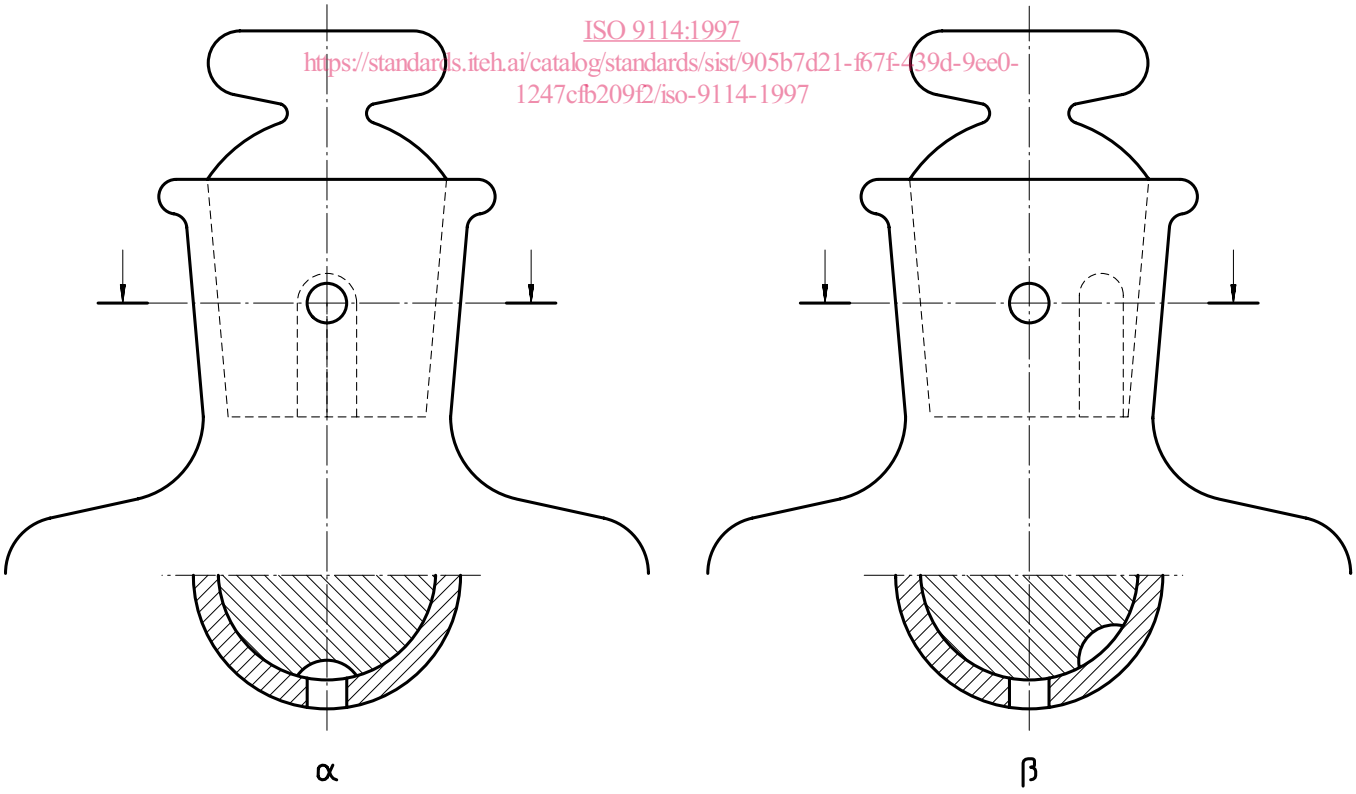
B

Position closed to air

Key

1 Towards tap R_1 and reaction flask
2 Air
3 Towards gas burette
4 From tap R_1 and reaction flask

Figure 3 — Diagram of tap R_2 in its two positions



α

Position open to air

β

Position closed to air

Figure 4 — Diagram of reservoir plug in its two positions