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Petroleum waxes — Determination of oil content

Cires de pétrole — Détermination de la teneur en huile

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2908 was drawn up by Technical Committee ISO/TC 28, *Petroleum products*, and circulated to the Member Bodies in March 1973.

It has been approved by the Member Bodies of the following countries :

Belgium	Iran	South Africa, Rep. of
Brazil	Israel	Sweden
Bulgaria	Mexico	Thailand
Canada	Netherlands	Turkey
Czechoslovakia	New Zealand	United Kingdom
France	Norway	U.S.A.
Germany	Poland	U.S.S.R.
Hungary	Portugal	
India	Romania	

No Member Body expressed disapproval of the document.

Petroleum waxes – Determination of oil content

1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard specifies a test procedure for the determination of oil in petroleum waxes having a congealing point of 30 °C or higher as determined by ISO 2207 and containing not more than 15 % of oil.

NOTE – Difficulties can arise with some waxes of oil contents greater than 5 % due to the limited solubility of oil in methyl ethyl ketone which can lead to the formation of two liquid phases. If this occurs, the method is not applicable to the material under test.

1.2 The oil content of a wax may have significant effects on several of its properties, such as strength, hardness, flexibility, scuff resistance, coefficient of friction, coefficient of expansion, melting point, and oil staining. The importance of these effects may be dependent upon the ultimate use of the wax.

2 REFERENCE

ISO 2207, *Petroleum waxes – Determination of congealing point*.¹⁾

3 PRINCIPLE

The sample is dissolved in methyl ethyl ketone, cooled to –32 °C to precipitate the wax, and filtered. The oil content of the filtrate is determined by evaporating the methyl ethyl ketone and weighing the residue.

4 SOLVENT

Methyl ethyl ketone, conforming to the following specification as determined by the specified or equivalent methods:

Property	Values	Methods ³⁾
Relative density 20/20 °C	0,805 to 0,807	2)
Colour	water white, 1,0 max.	IP 17 (B)
Distillation range	100 % between 78 and 81 °C	ASTM D 1078
Acidity	0,003 % (m/m) max. (expressed as acetic acid)	ASTM D 1613
Water content	not more than 0,3 % (m/m)	ASTM D 1364
Residue on evaporation	residue remaining after evaporation of 4 ml shall not exceed 0,1 mg	see procedure in 7.6
Refractive index at 20 °C	1,378 ± 0,002	ASTM D 1218

Store the solvent over anhydrous calcium sulphate (5 % (m/m) of the solvent).

Filter the solvent prior to use.

1) At present at the stage of draft.

2) A suitable method that is accurate to the third decimal place may be employed.

3) Substitution of the IP and ASTM method references by cross-references to appropriate International Standards will be made as soon as such International Standards become available.

5 APPARATUS

5.1 **Filter stick and assembly**, consisting of a 10 mm diameter sintered glass filter stick of porosity grade P16 (10 to 15 μm pore size index) as determined by the method given in the annex, provided with an air pressure inlet tube and delivery nozzle. It is provided with a ground glass joint to fit a 25 by 170 mm test tube. The dimensions for a suitable filtration assembly are shown in figure 1.

NOTE — A metallic filter stick may be employed if desired. A filter stick made of stainless steel and having a 12,7 mm disc of porosity grade P16 (10 to 15 μm pore size index) has been found to be satisfactory. The metallic apparatus is inserted into a 25 by 150 mm test tube and held in place by means of a cork.

Dimensions in millimetres

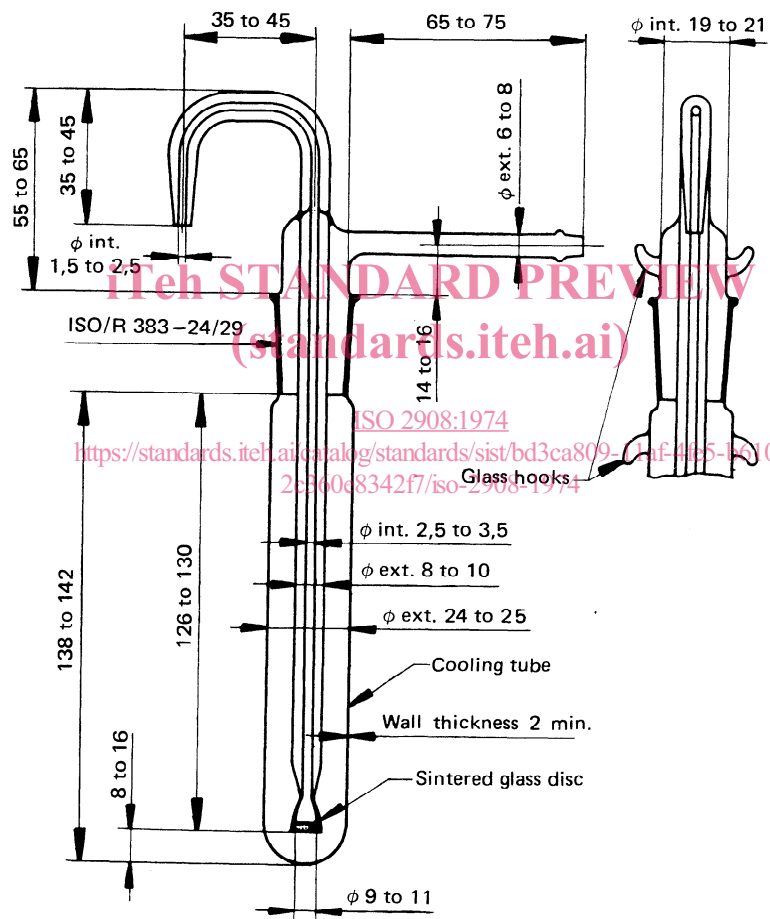


FIGURE 1 – Filter stick

5.2 Cooling bath, consisting of an insulated box with 25,4 mm holes in the centre to accommodate any desired number of test tubes. The bath may be filled with a suitable medium such as kerosine, and may be cooled by circulating a refrigerant through coils, or by using solid carbon dioxide. A suitable cooling bath to accommodate three test tubes is shown in figure 2.

Dimensions in millimetres

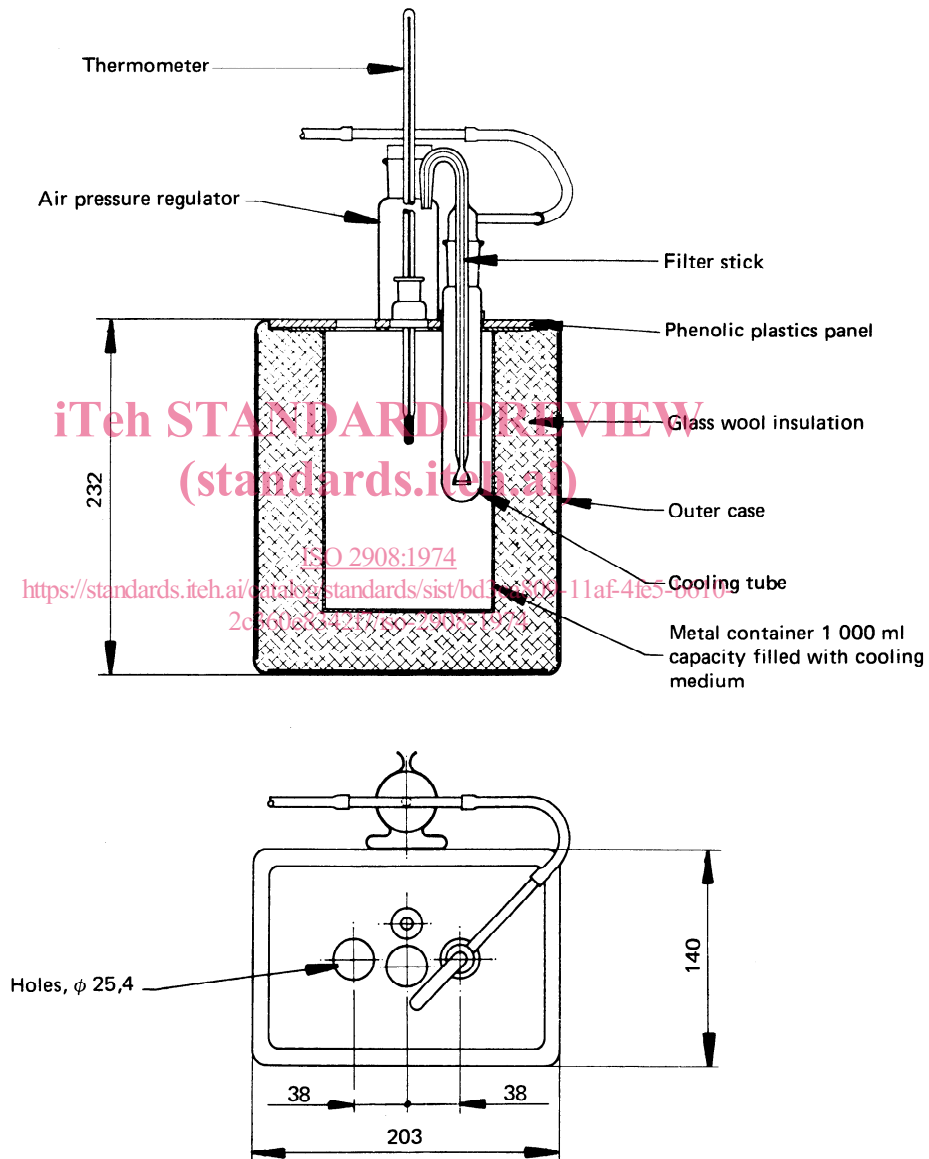


FIGURE 2 – Cooling bath

5.3 **Dropper pipette**, provided with a rubber bulb, and calibrated to deliver $1 \pm 0,05$ g of molten wax.

5.4 **Transfer pipette**, calibrated to deliver $15 \pm 0,06$ ml.

5.5 **Air pressure regulator**, designed to supply air to the filtration assembly (5.1) at the volume and pressure required to give an even flow of filtrate.

Either a conventional pressure-reducing valve or a mercury bubbler-type regulator has been found satisfactory. The latter type, illustrated in figure 3, consists of a 250 ml glass cylinder and a T-tube held in the cylinder by means of a rubber stopper grooved at the sides to permit the escape of excess air. The volume and pressure of the air supplied to the filtration assembly is regulated by the depth to which the T-tube is immersed in mercury at the bottom of the cylinder. Absorbent cotton placed in the space above the mercury prevents the loss of mercury by splattering. The air pressure regulator is connected to the filter stick and assembly by means of rubber tubing.

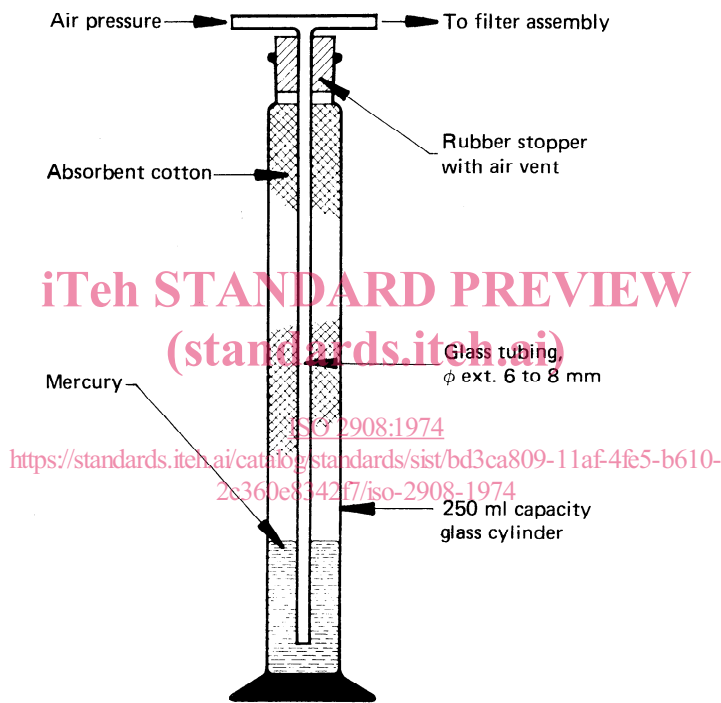


FIGURE 3 – Air pressure regulator

5.6 **Thermometer**, partial immersion type, conforming to the following specification :

Range	– 37 to 21 °C
Immersion	76 mm
Graduation at each	0,5 °C
Longer lines at each	1 and 5 °C
Figured at each	5 °C
Scale error not to exceed	0,2 °C
Expansion chamber permitting heating to	105 °C
Overall length	350 to 355 mm
Stem diameter	7,0 to 8,0 mm
Bulb shape	cylindrical
Bulb length	15 to 20 mm
Bulb diameter	6,0 to 7,0 mm
Length of graduated portion	105 to 140 mm
Distance from bottom of bulb to – 37 °C line	170 to 185 mm
Top finish	plain
Mean temperature of medium surrounding exposed column during testing	21 °C

5.7 Weighing bottles, conical in shape and glass-stoppered, having a capacity of 15 ml.

5.8 Evaporation assembly, consisting of an evaporating cabinet and connections, essentially as illustrated in figure 4, and capable of maintaining a temperature of $35 \pm 1^\circ\text{C}$ around the evaporation flasks.

Construct the jets with an inside diameter of $4 \pm 0,2$ mm for delivering a stream of clean, dry air vertically downward into the weighing bottle. Support each jet so that the tip is 15 ± 5 mm above the surface of the liquid at the start of the evaporation. Supply air at the rate of 2 to 3 l/min per jet, purified by passage through a tube of 10 mm bore packed loosely to a height of 200 mm with absorbent cotton. Periodically check the cleanliness of the air by evaporating 4 ml of methyl ethyl ketone by the procedure specified in 7.6. When the residue does not exceed 0,1 mg, the evaporation equipment is operating satisfactorily.

Dimensions in millimetres

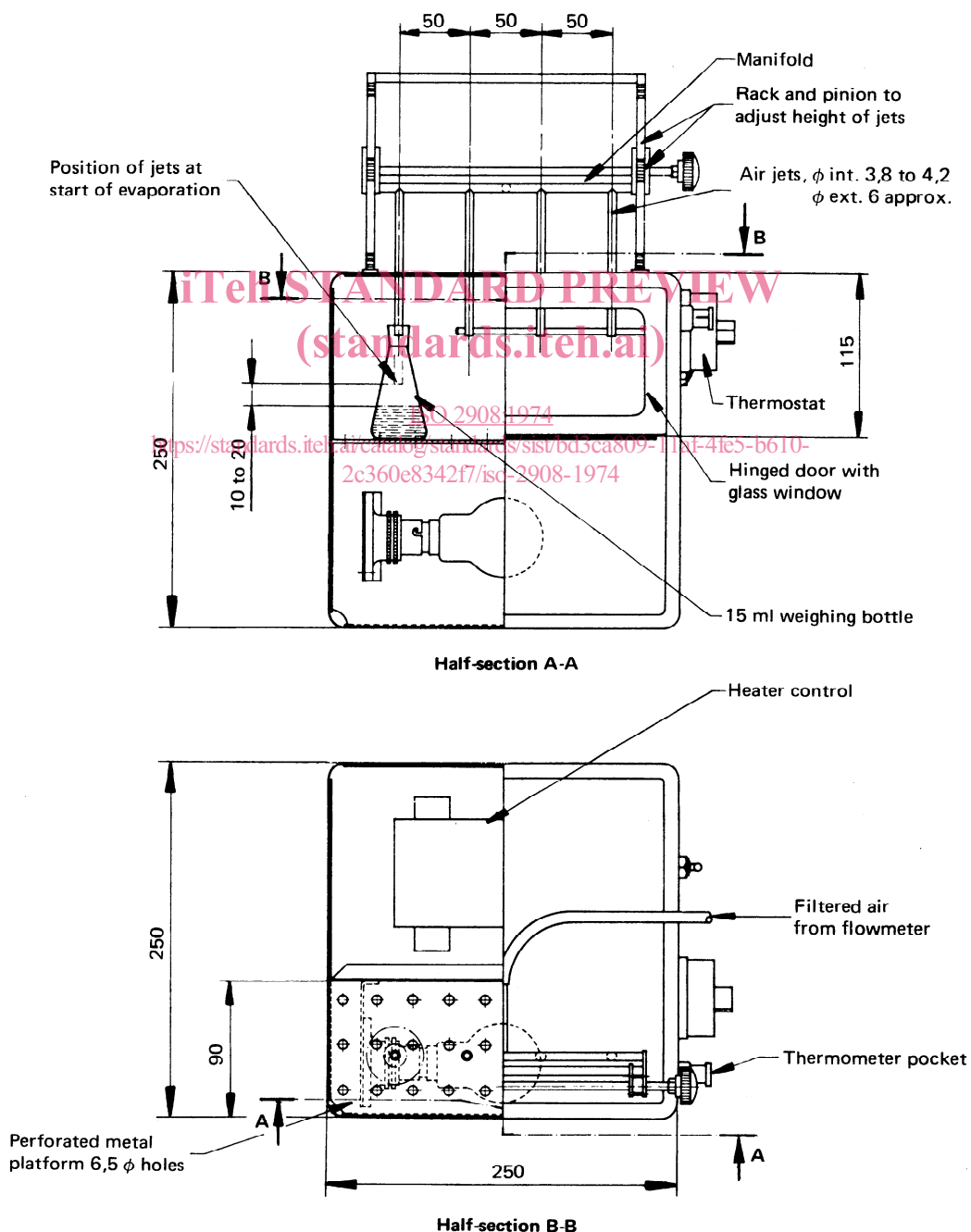


FIGURE 4 – Evaporation assembly

5.9 Analytical balance, accurate to 0,1 mg. In the case of a scale and pointer balance, the sensitivity should be adjusted so that 0,1 mg will deflect the pointer one half division on the pointer scale.

5.10 Wire stirrer, consisting of a piece of stiff iron or nickel-chromium alloy wire 0,9 mm in diameter and 250 mm long. A 10 mm diameter loop is formed at each end, and the loop at the bottom end is bent so that the plane of the loop is perpendicular to the wire.

6 SAMPLE

If the sample of wax is 1 kg or less, obtain a representative portion by melting the entire sample and stirring thoroughly. For samples over 1 kg, exercise special care to ensure that a truly representative portion is obtained, bearing in mind that the oil may not be distributed uniformly throughout the sample, and that mechanical operations may express some of the oil.

7 PROCEDURE

7.1 Melt a representative portion of the sample in a beaker, using a water bath or oven maintained at 70 to 100 °C. As soon as the wax is completely melted, thoroughly mix by stirring. Preheat the dropper pipette in order to prevent the solidification of wax in the tip, and withdraw a 1 g portion of the sample as soon as possible after the wax has melted. Hold the pipette in a vertical position, and carefully transfer its contents into a clean, dry test tube previously weighed to the nearest 1 mg (see note). Swirl the test tube so as to coat the bottom evenly with wax. This permits more rapid solution later. Allow the test tube to cool, and weigh to the nearest 1 mg.

NOTE — The mass of a test tube which is cleaned by means of solvents will not vary to a significant extent. Therefore, a tare mass may be obtained and used repeatedly.

7.2 Pipette 15 ml of methyl ethyl ketone into the test tube and place the latter just up to the level of its contents in a hot water or steam bath. Heat the solvent-wax mixture, stirring up and down with the wire stirrer, until a homogeneous solution is obtained. Exercise care to avoid loss of solvent by prolonged boiling.

NOTE — Wax samples with very high melting points may not form clear solutions. Stir until the undissolved material is well dispersed as a fine cloud.

7.3 Plunge the test tube into an 800 ml beaker of ice-water and continue to stir until the contents are cold. To ensure complete cooling, the test tube should be immersed in the ice-water for a minimum of 10 min. Remove the stirrer. Remove the test tube from the ice bath, wipe dry on the outside with a cloth, and weigh to the nearest 0,1 g.

NOTE — During this operation the loss of solvent through vaporization shall be less than 1 %. The mass of the solvent is, therefore, practically a constant, and, after a few samples are weighed, this mass, approximately 11,9 g, can be used as a constant factor.

7.4 Insert the thermometer into the test tube and place the test tube containing the wax-solvent slurry in the cooling bath, which is maintained at $-34,5 \pm 1$ °C. During this chilling operation it is important that stirring by means of the thermometer be almost continuous, in order to maintain a slurry of uniform consistency as the wax precipitates. Do not allow the wax to set on the walls of the cooling vessel and do not permit any lumps of wax crystals to form. Continue stirring until the temperature reaches $-31,7 \pm 0,3$ °C.

7.5 Immerse in the mixture the clean, dry filter stick, which has previously been cooled by placing it in a test tube and holding at $-34,5 \pm 1$ °C in the cooling bath for a minimum of 10 min. Seat the ground glass joint of the filter so as to make an airtight seal. Place an unstoppered weighing bottle, previously weighed together with the glass stopper to the nearest 0,1 mg, under the delivery nozzle of the filtration assembly.

NOTE — Take every precaution to ensure the accuracy of the weighing of the stoppered weighing bottle. Prior to determining this mass, rinse the clean, dry, weighing bottle and stopper with methyl ethyl ketone, wipe dry on the outside with a cloth, and place in the evaporation assembly to dry for about 5 min. Then remove the weighing bottle and stopper. Place near the balance, and allow to stand for 10 min prior to weighing. Stopper the bottle during this cooling period. Once the weighing bottle and stopper have been dried in the evaporation assembly, lift only with forceps. Take care to remove and replace the glass stopper with a light touch.

7.6 Apply air pressure to the filtration assembly, and immediately collect about 4 ml of filtrate in the weighing bottle. Release the air pressure to permit the liquid to drain back slowly from the delivery nozzle. Remove the weighing bottle immediately, and stopper and weigh to the nearest 10 mg without waiting for it to come to room temperature. Unstopper the weighing bottle and place it under one of the jets in the evaporation assembly maintained at 35 ± 1 °C, with the air jet centred inside the neck, and the tip 15 ± 5 mm above the surface of the liquid. After the solvent has evaporated, which usually takes less than 30 min, remove the bottle, stopper, and place near the balance. Allow to stand for 10 min and weigh to the nearest 0,1 mg. Repeat the evaporation procedure, using a 5 min evaporation period instead of 30 min until the loss between successive weighings is not over 0,2 mg.

8 EXPRESSION OF RESULTS

Calculate the amount of oil in the wax, as a percentage by mass, as follows :

$$\frac{100 m_1 m_3}{m_2 m_4} - 0,15$$

where

m_1 is the mass, in grams, of oil residue;

m_2 is the mass, in grams, of wax test portion;

m_3 is the mass, in grams, of solvent obtained by subtracting mass of test tube plus wax sample (see 7.1) from mass of test tube and contents (see 7.3);

m_4 is the mass, in grams, of solvent evaporated obtained by subtracting mass of weighing bottle plus oil residue from mass of weighing bottle plus filtrate (see 7.6);

0,15 is the average factor correcting for the solubility of wax in the solvent at $-31,7^{\circ}\text{C}$.

9 PRECISION

The following criteria shall be used for judging the acceptability of results (95 % confidence level) :

9.1 Duplicate results by the same operator shall be considered suspect if they differ by more than $0,06 + 8\%$ of the mean.

9.2 The results submitted by each of two laboratories shall be considered suspect if the two results differ by more than $0,2 + 11\%$ of the mean.

10 TEST REPORT

Report the result as oil content and make reference to this International Standard. If the result is negative, report as zero.

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