# SLOVENSKI STANDARD
# SIST EN 12:1996

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**Tekoči naftni proizvodi - Določanje parnega tlaka po Reidu - Mokra metoda**

Petroleum products - Determination of Reid vapour pressure - Wet method

Mineralölerzeugnisse - Bestimmung des Reid-Dampfdruckes - Nasses Verfahren

Produits pétroliers - Détermination de la pression vapeur Reid - Méthode humide



**Ta slovenski standard je istoveten z: EN 12:1993**

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English version

# Petroleum products - Determination of Reid vapour pressure - Wet method

Produits pétroliers - Détermination de la pression vapeur Reid - Méthode humide

Mineralölerzeugnisse - Bestimmung des Reid-Dampfdruckes - Nasses Verfahren



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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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**CEN**

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

## Foreword

This European Standard - Petroleum products - Determination of Reid vapour pressure - Wet method, was prepared by the technical committee CEN/TC 19 "Test methods and specifications for petroleum products", the secretariat of which is held by the Netherlands Standardization Institute (NNI).

In 1967 it was decided by CEN/TC 19 to draft a European method for the determination of the vapour pressure of petroleum products, based on ASTM D 323 (a Reid vapour pressure method, RVP).

PrEN 12 was subsequently drafted and submitted for preliminary vote, with a positive result in 1974, but was held in abeyance awaiting developments within ISO. The ISO standard giving the Reid method for the determination of the vapour pressure of petroleum products (ISO 3007) was published in 1974.

PrEN 12 combined an additional so called "dry" procedure to that stated in ISO 3007, to be applied in case of gasoline containing water soluble constituents. In order to establish the necessity and validity of this additional "dry" procedure for oxygenated fuels, prEN 12 was again held in abeyance.



With the publication of Directive 85/536/EEC on "crude oil saving through the use of oxygenated petrol", it became necessary to reconsider a "dry" procedure in the RVP method for petrol containing oxygenated compounds.



However, at the 15th and 16th meeting in 1986 and 1987 of CEN/TC 19, it was concluded that "a wet RVP method would be acceptable as European Standard for the determination of the vapour pressure of petrol containing oxygenates within the limits as given in Directive 85/536/EEC, Annex, Column A" (Resolution 13 of the 16th meeting).

Consequently a final version of prEN 12 was prepared, based on the work of WG 15. This standard is a modification of ISO 3007 (1986) but extended to take into account the application to petrol containing oxygenated compounds.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 1994, and conflicting national standards shall be withdrawn at the latest by March 1994.

In accordance with the CEN/CENELEC Internal Regulations, following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

## 1 Scope

This European Standard specifies a method for the determination of the absolute vapour pressure of volatile crude oil and volatile non-viscous petroleum products, with vapour pressures below 180 kPa.

This method is applicable to fuels containing oxygenated compounds up to the limits stated in Annex A, column A of this standard. The method is also applicable to fuels containing up to 2,8 % *m/m* total oxygen, if due to ethers only.

NOTE 1:
For higher oxygenate contents national standards may be applied until a European Standard has been developed.

NOTE 2:
Because the external atmospheric pressure is counteracted by the atmospheric pressure initially present in the air chamber, the "Reid vapour pressure" is approximately the absolute vapour pressure of the material at 37,8°C in kilopascals (bars). The "Reid vapour pressure" differs from the true vapour pressure of the sample owing to slight vaporisation and the presence of water vapour and air in the confined space.



## 2 Principle



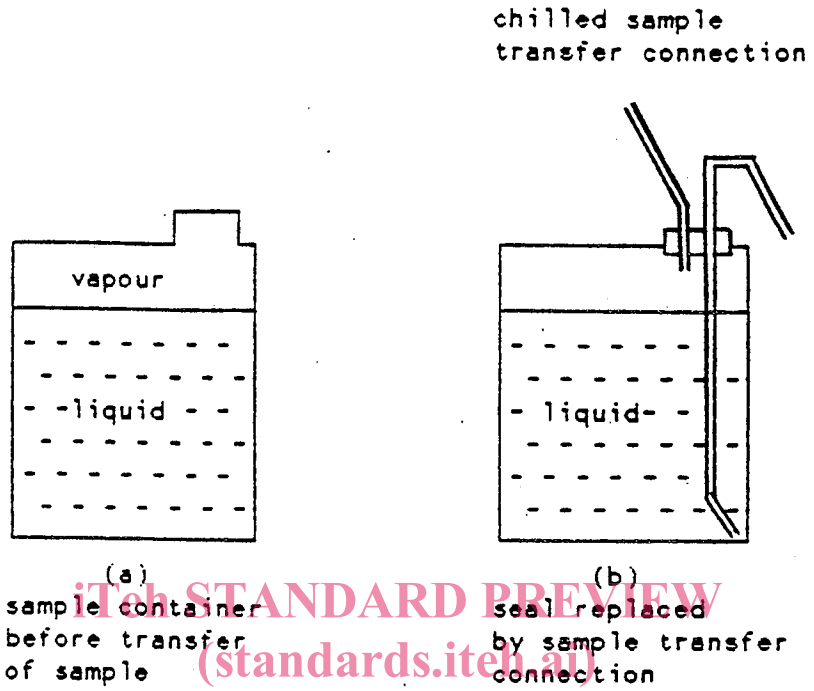
The liquid chamber of the vapour pressure apparatus is filled with the chilled sample and connected to the air chamber. The apparatus is immersed in a constant temperature bath (37,8°C ± 0,1°C) and is agitated until equilibrium is reached. The reading of the pressure indicator is the Reid vapour pressure.

**WARNING: This standard calls for the use of substances and/or procedures that may be injurious to health if adequate precautions are not taken. It refers only to the technical suitability and in no way absolves the user from statutory obligations relating to health and safety at any stage.**

## 3 Apparatus

The construction of the required apparatus is described in annex B. This method is based upon the use of pressure gauges or a pressurized manometer (annex C). Other pressure indicators may also be used.

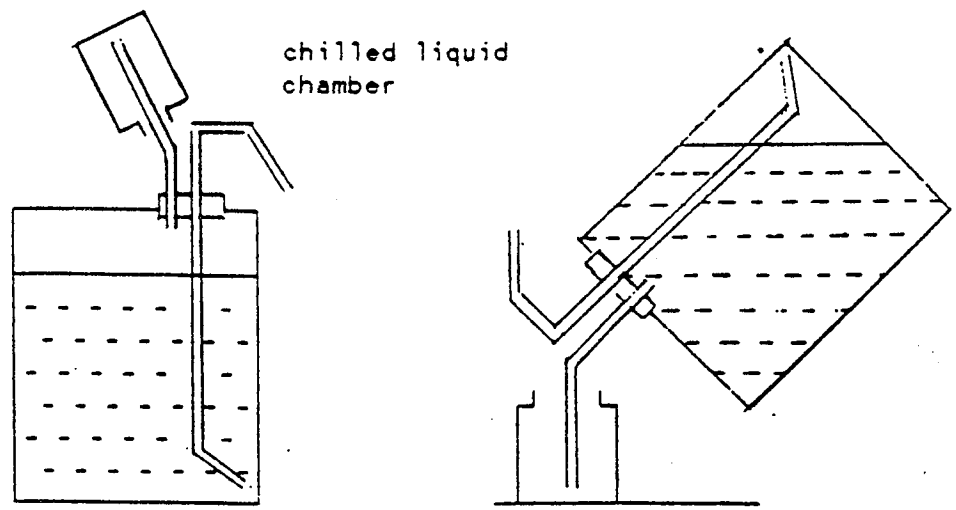
Automatic equipment may be used provided it gives equivalent accuracy and precision as the manual method. In case of dispute the manual method is the reference method.




(a)
sample container
before transfer
of sample

(b)
seal replaced
by sample transfer
connection






(c)
liquid chamber
placed over liquid
delivery tube

(d)
position of system
for sample transfer

Figure 1: Method of transferring the sample to the liquid chamber from open type containers

sample till the container contents are within the 70 % to 80 % range. After the correct liquid content has been assured, reseal the container, shake it vigorously and return it to the cooling bath or refrigerator. Repeat this procedure of unsealing momentarily, resealing, shaking vigoursly, and returning to the bath or refrigerator three times at intervals of not less than 2 min to ensure complete air saturation.

## 5.2 Preparation of the liquid chamber

Put the open liquid chamber and the sample transfer connection in the cooling bath or refrigerator for a sufficient time (at least 10 min) to allow the chamber and connection to reach the bath temperature (0°C to 1°C), ensuring no ingress of water to prevent phase separation.

## 5.3 Preparation of the air chamber

### 5.3.1 *General*

Use the procedure as described in 5.3.2 or 5.3.3 (see note 3).

NOTE 3:
In the preparation of the air chamber, the 37,8°C procedure is preferred but the ambient temperature procedure gives equivalent results.



### 5.3.2 *37,8°C Procedure*

After purging and rinsing the air chamber and pressure gauge in accordance with 6.6 connect the gauge to the air chamber. Immerse the air chamber to at least 25 mm above its top in the water bath maintained at 37,8°C ± 0,1°C for not less than 10 min, just before coupling it to the liquid chamber. Do not remove the air chamber from the bath until the liquid chamber has been filled with the sample as described in 6.2.

### 5.3.3 *Ambient temperature procedure*

Adjust the air chamber to ambient or other temperature, which may be determined with an accuracy of at least 0,5°C in the following manner: After purging and rinsing the air chamber in accordance with 6.6 connect the pressure gauge to the air chamber. Insert the thermometer into the chamber, supporting it by means of a loosely fitting (not airtight) stopper in the opening of the air chamber.

Adjust the position of the thermometer so that it is aligned as closely as possible with the axis of the air chamber and with the thermometer bulb located in the air chamber, about 230 mm from the opening. Leave the thermometer in position until the temperature reading has remained constant within 0,5°C for a period of 5 min or more just before coupling the air chamber to the liquid chamber. At this time, record the thermometer reading as the "initial air temperature".

# 6 Procedure using a pressure gauge

## 6.1 General

Clause 6 applies to the manual procedure. If automatic equipment is used follow the manufacturers instructions.

## 6.2 Sample transfer

With everything in readiness, remove the chilled sample container from the bath, uncap it and insert the chilled transfer connection and air tube (see figure 1). Take the empty chilled liquid chamber and place it over the sample delivery tube of the transfer connection. Invert the entire system rapidly so that the liquid chamber is finally in an upright position with the delivery tube extending to within 6 mm of the bottom of the liquid chamber. Fill the liquid chamber to overflowing. Lightly tap the liquid chamber against the work bench to ensure that the sample is free of air bubbles. If any sample is displaced, refill the chamber to overflowing.

## 6.3 Assembly of apparatus



Without delay and as quickly as possible, attach the air chamber to the liquid chamber. Do not make more than 10 s to complete the assembly of the apparatus after filling the liquid chamber, using the following sequence of operations.



6.3.1 Add additional sample to the liquid chamber to fill to overflowing.

6.3.2 Remove the air chamber from the 37,8°C water bath (see 5.3.2).

6.3.3 If the "ambient air procedure" is used (see 5.3.3), record the initial temperature of the air chamber.

## 6.4 Introduction of the apparatus into the bath

Turn the assembled vapour pressure apparatus upside down to allow the sample in the liquid chamber to run into the air chamber and shake vigorously in a direction parallel to the length of the apparatus. Immerse the assembled apparatus in the bath, maintained at 37,8°C ± 0,1°C, in an inclined position so that the connection of the liquid and the air chamber is below the water level and may be observed closely for leaks. If no leaks are observed, immerse the apparatus to at least 25 mm above the top of the air chamber. Observe the apparatus throughout the test for leaks. If a leak is detected at any time during the test, the sample shall be discarded and the test recommenced with a fresh sample.

NOTE 4:
Liquid leaks are more difficult to detect than vapour leaks because the much-used coupling device is normally in the liquid section of the apparatus; give this particular attention.

### 6.5 Measurement of vapour pressure

After the assembled vapour pressure apparatus has been immersed in the bath for 5 min, observe the reading. Tap the pressure gauge lightly. Withdraw the apparatus from the bath, invert it, shake it vigorously in a direction parallel to the length of the apparatus and replace it in the bath in the shortest possible time to avoid cooling the apparatus. To ensure equilibrium conditions, repeat this agitation and pressure reading at least 5 times, at intervals of not less than 2 min and until the last 2 consecutive pressure readings are identical. This sequence of operations normally requires 20 min to 30 min. Read the final pressure to the nearest 0,25 kPa for pressure gauges with intermediate graduations of 0,5 kPa and to the nearest 0,5 kPa for gauges with graduations of 1,0 kPa. Remove the gauge and check its indicated value against that of the mercury manometer (B.6). If necessary, correct the gauge reading. Record this value as the Reid vapour pressure if the 37,8°C procedure has been used or as "final gauge reading" if the ambient air procedure has been used (for further calculation see clause 9).

### 6.6 Preparation of the apparatus for the next test



Disconnect the air chamber, liquid chamber and pressure gauge. Remove trapped fluid from the Bourdon gauge in the following manner: hold the gauge in the palms of the hands with the right hand on the face side and the threaded connection of the gauge forward. Extend the arms forward and upward at an angle of 45 degrees with the coupling of the gauge pointing in the same direction. Swing the arms downwards through an arc of about 135 degrees so that the centrifugal force aids gravity in removing the trapped liquid. Repeat this operation 3 times to expel all liquid. Purge the pressure gauge by directing a small jet of air into it's Bourdon tube for at least 5 min.



Thoroughly purge the air chamber of residual sample by filling it with warm water (above 32°C) and allowing it to drain. Repeat this purging at least 5 times. After thoroughly removing the previous sample from the liquid chamber, dry the liquid chamber and place it in the cooling bath or refrigerator for the next test. In the case of crude oil it is necessary to wash all the equipment with a volatile solvent, preferably toluene, after each test.

NOTE 5:
If the purging of the air chamber is done in a bath, be sure to avoid small and unnoticeable films of floating sample by keeping the bottom and top openings of the chambers closed as they pass through the surface of the water.

## 7 Procedure for using the prepressurized mercury manometer
**(For vapour pressures below 100 kPa only)**

### 7.1 Sample transfer

Transfer the sample as described in 6.2.

## 7.2 Assembly of the apparatus

Assemble the apparatue as described in 6.3 and annex C. Check that the needle valve on the air chember is tightly closed, attach the manometer hose to the top of the air chember and follow the sequence of operations as described in 6.3.

## 7.3 Introduction of the apparatus into the bath

Introduce the apparatus into the bath as described in 6.4.

## 7.4 Prepressurizing the manometer

After the assembled vapour pressure apparatus has been immersed in the bath and checked for leaks as described in 6.4, prepressurize the manometer and flexible hose to the expected vapour pressure of the sample (see note 6), and record this value as the "initial manometer setting". While the sample is being brought to equilibrium as described in 6.5, observe the manometer occasionally to check for leaks in the manometer assembly. Any change from the "initial manometer setting" indicates a leak and the vapour pressure apparatus should be detached and connected to an alternative manometer.



NOTE 6:
For the purpose of prepressurizing and to avoid the necessity for rerunning determinations, knowledge of the expected vapour pressure is very helpful. An indication of the expected vapour pressure level should be given, where possible, on the sample identifiaction of the label. It is also helpful to maintain a list of current vapour pressures of samples being tested on a routine basis.



## 7.5 Measurement of the vapour pressure

After the apparatue has been immersed in the bath for 5 min and if no leaks have been observed, carefully withdraw the apparatus from the bath.
Without opening the needle valve, invert the apparatus, shake it vigorously along its length and replace it in the bath in the shortest possible time to avoid cooling the apparatus. Repeat this withdrawal and shaking operation after a further 5 min, again replacing the apparatus in the bath in the shortest possible time. After not less than 2 min, open the valve and record the manometer reading. Close the valve. Repeat this operation at intervals of not less than 2 min until the last 2 consecutive readings are constant. These operations normally require 20 min to 30 min. Read the final manometer pressure to the nearest 1 kPa and record the value as the "constant manometer reading" of the sample under test.

## 7.6 Appraisal of the observations

For accurate results, the "constant manometer reading" shall be within 10 kPa of the "initial manometer setting". If the difference is less than this amount proceed as described in clause 9. If the difference is greater, make a second determination using the first result as an approximation for prepressurizing the manometer. Repeat until the

difference is within the prescribed limits of 10 kPa, each time using a fresh sample.

### 7.7 Preparation of the apparatus for the next test

Disconnect the manometer hose, the air chamber and the liquid chamber. Remove the connector and valve assembly from the air chamber and, with the valve open, purge with air for at least 5 min. Purge the air chamber by filling it with warm water and draining at least 5 times. After removing the previous sample from the liquid chamber, purge the chamber with cold water, dry the liquid chamber and place it in the cooling bath or refrigerator for the next test. In the case of crude oil, it is necessary to wash all the equipment with a volatile solvent, preferably toluene, after each test.

## 8 Precautions

Gross errors are liable to be made in vapour pressure measurements if the prescribed procedure is not followed carefully. The following list emphasises the importance of strict adherence to the precautions given in the procedure.



### 8.1 Checking the pressure gauges

Check all pressure gauges regularly in order to ensure the precision of results. Ensure that gauges are in a vertical position before reading them.



### 8.2 Air saturation of the sample

Open and close the sample container after the contents have reached the temperature of 0°C to 1°C. Shake the container vigorously to ensure equilibrium of the sample with the air in the container. Repeat this procedure three times.

### 8.3 Checking for leaks

Check the apparatus before and during each test for liquid and vapour leaks (see B.1.5 and note 4).

### 8.4 Sampling

Because initial sampling and the handling of samples can greatly affect the final results, take great care to avoid losses through evaporation and slight changes in composition (see 4 and 6.2). In no case shall any part of the Reid apparatus be used as the sample container prior to actually conducting the test.

### 8.5 Purging the apparatus

Thoroughly purge the pressure gauge, the liquid chamber and the air