

Liquid petroleum products — Vapour pressure —

**Part 2: Determination of absolute
pressure (AVP) between 40 °C
and 100 °C**

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National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Foreword

This document (EN 13016-2:2007) has been prepared by Technical Committee CEN/TC 19 “Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin”, the secretariat of which is held by NEN.

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of an identical text or by endorsement, at the latest by March 2008, and conflicting national standards shall be withdrawn at the latest by March 2008.

This document supersedes EN 13016-2:2000, which has been revised editorially to clarify a number of steps related to sampling, calibration of the pressure transducer and the procedure. No new precision evaluations have been carried out. The equation to calculate the absolute vapour pressure in the informative Annex A has been corrected.

EN 13016 consists of the following parts, under the general title *Liquid petroleum products - Vapour pressure*:

- *Part 1: Determination of air-saturated vapour pressure (ASVP) and calculated dry vapour pressure equivalent (DVPE).*
- *Part 2: Determination of absolute vapour pressure (AVP) between 40 °C and 100 °C.*

Part 1 is based on and developed in parallel with IP 394 [1] and ASTM D 5191 [2]. It describes a general determination method, whereas this part describes one at elevated temperatures.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard : Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

Vapour pressure is used as a classification criterion for the safe handling and carriage of petroleum products, feedstocks and components; it has a relationship to the potential for hydrocarbon emissions, under uncontrolled conditions, and thus is the subject of environmental scrutiny.

Vapour pressure limitations are often imposed to prevent pump cavitation during transfer operations.

Vapour pressure is one measure of the volatility characteristics of fuels used in many differing types of engines with large variations in operating temperatures. Fuels having a high vapour pressure may vaporize too readily in the fuel handling systems, resulting in decreased flow to the engine and possible stoppage by vapour lock. Conversely, fuels of low vapour pressure may not vaporize readily enough, resulting in difficult starting, slow warm-up and poor acceleration.

1 Scope

This European Standard specifies a method for the determination of absolute vapour pressure of liquid petroleum products at elevated temperatures.

The conditions used in the test described in this standard are a vapour to liquid ratio of 3:2 and an initial injection temperature of 37,8 °C or 30,0 °C.

The method described is suitable for testing air-saturated samples that exert an air-saturated vapour pressure of between 9 kPa and 500 kPa at temperatures between 40 °C and 100 °C.

This European Standard is applicable to fuels containing oxygenated compounds up to the limits stated in the relevant EC Directive 85/536/EEC [3].

NOTE 1 If a sample injection is into a test chamber which is raised to 37,8 °C and the vapour to liquid ratio is 4:1, the initial measurement corresponds with the measurement in Part 1 of this standard.

NOTE 2 For the purposes of this European Standard, the term “% (V/V)” is used to represent the volume fraction.

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

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.

EN ISO 3170, *Petroleum liquids - Manual sampling (ISO 3170:2004)*

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

3.1

air-saturated vapour pressure

total pressure

ASVP

observed pressure exerted in vacuo by the partial pressure of air-saturated petroleum products, components and feedstocks, in the absence of non-dissolved water, and the partial pressure of the dissolved air

3.2

absolute vapour pressure

AVP

air-saturated vapour pressure minus the partial pressure due to dissolved air in the liquid

4 Principle

A cooled air-saturated sample of known volume is introduced into an evacuated, temperature-controlled chamber in two equal test portions. The volume of the chamber is five times that of the sample introduced to provide a vapour-to-liquid ratio of 4:1. The vapour-to-liquid ratio following the second injection is 3:2. After

the injection of each test portion, the air-saturated vapour pressure is determined. The partial pressure of the dissolved air is calculated from the two resulting pressures.

The temperature of the test chamber is then increased to a specified value and the air-saturated vapour pressure determined. The absolute vapour pressure (AVP) is calculated by subtracting the partial pressure of the dissolved air in the liquid, which has been corrected for temperature and compensated for compressive effects and solubility, from the air-saturated vapour pressure. The procedure may be repeated at further test temperatures.

5 Reagents

Pentane, reagent grade 99,5 % (V/V) minimum purity.

6 Apparatus

6.1 Instrument

6.1.1 The instrument shall conform to the general requirements given in 6.1.2 to 6.1.6.

NOTE Full details of suitable instruments are not given because of differences in the way that the basic principles are applied by individual manufacturers.

The instrument shall be installed, operated and maintained in accordance with the manufacturer's manual.

6.1.2 The system shall be configured to enable the test chamber to be evacuated and isolated, the sample to be drained, and the system flushed and purged as necessary.

6.1.3 The test chamber shall be vacuum-tight, with provision for the introduction of the sample, and shall be capable of containing between 5 ml and 15 ml of liquid and vapour with an accuracy of 1 %. The test chamber shall be capable of controlling the temperature of the sample to achieve the specified test temperature to within $\pm 0,1$ °C and of indicating the temperature to a resolution of at least 0,1 °C.

NOTE 1 The test chambers used in the instruments that generated the precision statements were constructed from either aluminum or stainless steel.

NOTE 2 Test chambers with capacities less than 5 ml or greater than 15 ml may be used but the precision of the method can be affected.

6.1.4 The apparatus shall be capable of measuring the vapour pressure of small samples of petroleum products, components and feedstocks over the pressure range 9,0 kPa to 500,0 kPa and at temperatures between 40 °C and 100 °C, by means of a pressure transducer, with an accuracy of 0,8 kPa and a resolution of 0,1 kPa.

6.1.5 If a vacuum pump is required for use with the instrument, it shall be capable of reducing the pressure in the test chamber to less than 0,01 kPa absolute.

6.1.6 If a vacuum-tight syringe or similar equipment is required for measuring or injecting the required volume of sample into the test chamber, it shall be sized appropriately to the required sample size with an accuracy of at least 1 %.

6.2 Cooling equipment, air or iced-water bath or a refrigerator, capable of cooling the samples to a temperature of between 0 °C and 1 °C.

NOTE A suitably safe refrigerator should be used with highly volatile petroleum products.

6.3 Barometer for calibration, capable of measuring atmospheric pressure within an accuracy of 0,1 kPa or better and calibrated and/or verified against an instrument certified by an authorized certification body.

6.4 Vacuum gauge for calibration, covering at least the range 0 kPa to 177 kPa, calibrated and/or verified against an instrument certified by an authorized certification body.

6.5 Pressure measuring device for the apparatus, having a minimum measuring range from 0 kPa to 500 kPa, calibrated and/or verified against an instrument certified by an authorized certification body.

7 Sampling

7.1 Due to the extreme sensitivity of vapour pressure measurements to losses through evaporation and the resulting changes in composition, the utmost precaution and the most meticulous care shall be taken in the drawing and handling of samples.

7.2 Samples shall be drawn in accordance with EN ISO 3170 and/or in accordance with the requirements of National Standards or regulations for the sampling of the product under test. However, the water displacement technique shall not be used.

NOTE Drawing samples using automatic techniques, such as those described in EN ISO 3171 [4], is not recommended unless the technique has been proven not to lose light ends from the product or component being sampled. Loss of light ends may effect the vapour pressure measurement.

7.3 For routine testing, the sample shall be supplied in a sealed container, constructed of suitable material, of either 1 l capacity or in a container of a different capacity with the same ullage requirement. For referee testing a 1 l sample container shall be used. The container shall be a minimum of 70 % (V/V) full of sample at the time of receipt.

NOTE The precision of the method can be different if container capacities differ from 1 l.

7.4 Samples shall be placed in a cool place as soon as possible after they have been obtained and held there until the test has been completed.

NOTE To protect the samples from excessive temperature prior to testing, it is recommended to store the samples in the cooling equipment described in clause 6.2.

7.5 Samples in leaking containers shall not be considered for testing, but shall be discarded and new samples obtained.

8 Sample preparation

8.1 The vapour pressure determination shall be the first test on a sample. For referee testing, only one test portion shall be taken from the container; for routine testing, it is permitted for further samples to be taken from the same container.

8.2 Before the sample container is opened, place it in the cooling equipment (6.2) and allow sufficient time for the container and contents to cool to between 0 °C and 1 °C.

NOTE Sufficient time to reach this temperature may be ensured by direct measurement of the temperature of a similar liquid in a similar container placed in the same bath at the same time as the sample.

8.3 With the sample at a temperature of 0 °C to 1 °C, remove the container from the cooling equipment and wipe dry with an absorbent material. Unseal the container (if it is not transparent) and examine the sample content.

8.4 The sample content shall be 70 % (V/V) to 80 % (V/V) of the container capacity. Discard the sample if its volume is less than 70 % (V/V) of the container capacity. If the container is more than 80 % (V/V) full, pour out sufficient sample to bring the container contents within the 70 % (V/V) to 80 % (V/V) range. Under no circumstances shall any sample be returned to the container if it has been poured out previously. Reseal the container and return it to the cooling equipment (6.2).

8.5 To ensure that the sample is air-saturated, remove the container from the cooling equipment (6.2) when the sample is at a temperature of 0 °C to 1 °C. Wipe the container dry with absorbent material, unseal it momentarily taking care that no water enters, reseal it and shake vigorously. Return to the cooling equipment for a minimum of 2 min.

8.6 Repeat clause 8.5 two more times. Return the sample to the cooling equipment until commencing the test.

9 Preparation of apparatus

9.1 Prepare the instrument for operation in accordance with the manufacturer's instructions.

9.2 Prepare the test chamber, as required to avoid contamination of the test portion, according to the manufacturer's instructions. Visually determine from the instrument display that the evacuated test chamber pressure is stable and does not exceed 0,1 kPa. When the pressure is not stable, or exceeds this value, check that no traces of volatile components are present in the chamber from a previous sample or check the calibration of the transducer.

9.3 If a syringe (6.1.6) is used for injection of the test portion, cool it between 0 °C and 1 °C in an air bath or refrigerator before drawing in the sample. Avoid water contamination of the syringe reservoir by sealing the outlet of the syringe suitably during the cooling process.

10 Calibration of apparatus

10.1 Pressure transducer

10.1.1 Check the calibration of the transducer at 37,8 °C on at least a 6 month basis or when required as indicated from the quality control checks. The calibration of the transducer shall be checked using two reference points; zero pressure (< 0,1 kPa) and the ambient barometric pressure.

NOTE 1 A mercury column barometer is the most accurate and suitable device for calibrating the atmospheric pressure display of the apparatus, however, these barometers are either calibrated at 0 °C or are manufactured using the density of mercury as defined at 0 °C. This means that, when the barometer is used in the laboratory at ambient temperature, the reading of the barometer will be slightly high. The necessary correction varies with temperature and atmospheric pressure, for example, at 20 °C the correction is minus 0,33 kPa.

NOTE 2 Calibration at a third point, e.g. 350 kPa, using a calibrated gauge or a calibrated source of pressure, such as a dead-weight tester, should be carried out if this is recommended by the manufacturer.

10.1.2 Connect the calibrated vacuum gauge or pressure transducer (6.4) to the vacuum source in line with the test chamber. When the calibrated gauge or transducer registers a pressure less than 0,1 kPa, adjust the transducer control to zero or to the actual reading on the calibrated gauge or transducer as dictated by the instrument design and the manufacturer's instructions.

10.1.3 Open the test chamber to the atmosphere and observe the transducer reading. If the pressure reading is not the ambient barometric pressure, adjust the transducer span control until the appropriate reading is observed. Ensure that the instrument is set to display the total pressure and not a calculated or corrected value.

10.1.4 Repeat 10.1.2 and 10.1.3 until the zero and barometric pressures read to within $\pm 0,1$ kPa without further adjustment.

NOTE Some instruments automate the calibration procedure and carry out these repeat operations automatically.

10.2 Temperature measuring device

Check the calibration of the temperature measuring device used to monitor the temperature of the sample in the test chamber, against a calibrated temperature device (6.5) at least every 6 months or when needed as indicated from the quality control checks. The temperature readings shall agree to within $\pm 0,1$ °C at the test temperature. When a calibrated liquid-in-glass thermometer is used to check the calibration of the temperature-measuring device, ensure that a thermometer with the correct immersion depth is used or apply appropriate emergent stem corrections.

11 Verification of apparatus

Check the performance of the instrument each day it is in use, or at a frequency determined by statistical quality control analysis data, by running a quality control check sample of pentane (5.1). Treat the pentane quality control check sample in the same manner as a sample (see clauses 8 and 12). Determine the absolute vapour pressure (AVP) at 80 °C, if this differs from 367 kPa by more than ± 2 kPa, check the instrument calibration (see clause 10).

12 Procedure

12.1 Set the temperature of the test chamber, T_c , to $37,8$ °C $\pm 0,1$ °C or $30,0$ °C $\pm 0,1$ °C for the injection of the first test portion according to the manufacturer's instructions.

12.2 Remove the sample (see 4) from the cooling equipment (6.2), dry the exterior of the container with absorbent material, uncap, and insert a syringe (see 9.3). Draw a bubble-free test portion to provide a vapour-to-liquid ratio of 4:1 (see clause 4) and deliver it to the test chamber as rapidly as possible. Reseal the container. The total time between opening the cooled sample container and injection of the test portion into the sealed test chamber shall not exceed 1 min.

12.3 Obtain an absolute vapour pressure result by following the manufacturer's instructions for operation of the instrument.

12.4 Record the pressure indicator reading from the instrument to the nearest 0,1 kPa. For instruments that do not automatically record a stable pressure value, manually record the pressure indicator reading every $60 \text{ s} \pm 5 \text{ s}$ to the nearest 0,1 kPa. When three successive readings agree within 0,1 kPa, record the mean to the nearest 0,1 kPa as $ASVP_1$.

12.5 With the first test portion remaining in the test chamber at $37,8$ °C or $30,0$ °C, follow 12.2 to inject a second test portion into the chamber of the same volume as initially injected. The resulting vapour to liquid ratio shall be 3:2.

12.6 Follow 12.4 to obtain a stable pressure indicator reading. When three successive readings agree with 0,1 kPa, record the mean to the nearest 0,1 kPa as $ASVP_2$.

12.7 Allow the temperature of the test chamber to reach the desired test temperature (between 40 °C and 100 °C) within $\pm 0,1$ °C. After a minimum of 3 min, when the pressure has stabilized, record the resulting vapour pressure as $ASVP_T$.

This procedure may be repeated at further test temperatures.

12.8 After drawing the second test portion and injecting it into the instrument for analysis, check the remaining sample for phase separation.

If the sample is in a non-transparent container, shake the sample thoroughly and immediately pour a portion of the remaining sample into a glass container and observe for evidence of phase separation. If the sample is already in a glass container, this observation can be made prior to sample transfer.

If the sample is not clear and bright or if a second phase is observed, discard the test results and the sample.

13 Calculation

NOTE The derivation of the equations used to calculate *AVP* is given in Annex A.

13.1 Calculate the partial pressure of the air, PP_c , after the test portions have been injected into the test chamber, using the following equation:

$$PP_c = 1,7 (ASVP_2 - ASVP_1) \quad (1)$$

where

$ASVP_1$ is the air-saturated vapour pressure of the first test portion, in kilopascals;

$ASVP_2$ is the air-saturated vapour pressure after injection of the second test portion, in kilopascals.

13.2 Calculate the partial pressure of the air at the test temperature, PP_T , using the following equation:

$$PP_T = \frac{PP_c(273,2 + T)}{(273,2 + T_c)} \quad (2)$$

where

T_c is the temperature of the test chamber when the test portion is injected;

T is the test temperature in degrees Celsius.

EXAMPLE Using conditions of a sample injection temperature of 37,8 °C and test temperatures of 80 °C and 100 °C, the air corrections would be $PP_{80} = 1,136 \times PP_{37,8}$ and $PP_{100} = 1,2 \times PP_{37,8}$.

13.3 Calculate the absolute vapour pressure, AVP_T , at temperature T , using the following equation:

$$AVP_T = ASVP_T - PP_T \quad (3)$$

where

$ASVP_T$ is the air-saturated vapour pressure at temperature T , in kilopascals;

PP_T is the partial pressure of the air at temperature T , in kilopascals.

EXAMPLE Using conditions of a sample injection temperature of 37,8 °C and test temperatures of 80 °C and 100 °C, absolute vapour pressures would be $AVP_{80} = ASVP_{80} - 1,931 \times (ASVP_2 - ASVP_1)$ and $AVP_{100} = ASVP_{100} - 2,04 \times (ASVP_2 - ASVP_1)$.

14 Expression of results

Report the *AVP* of the sample to the nearest 0,1 kPa.

15 Precision

NOTE The precision data were based upon results obtained in 1991¹⁾ by 17 laboratories from 6 samples in the range 170 kPa to 420 kPa at temperatures of 80 °C and 100 °C. Laboratory Grabner CCA/VP and Setavap Analyser 8000 instruments were used to determine the precision.

15.1 Repeatability, r

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the values given below only in one case in twenty.

$$r = 0,0077X + 2,4 \quad (4)$$

where

X is the average of the results in kilopascals being compared.

15.2 Reproducibility, R

The difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the values given below only in one case in twenty.

$$R = 12,5$$

16 Test report

The test report shall contain at least the following information:

- a) type and identification of the product under test;
- b) reference to this European Standard, (EN 13016-2:2007);
- c) sampling procedure used and size of sample container (see clause 7);
- d) result of the test and the test temperature (see clause 14);
- e) any deviation from the procedure described;
- f) date of the test.

1) The results of this test programme are filed by the EI, London under Test Method IP 406. The report is DIN 0051 439 Cooperative RR-test DIN 0051 848, Munchen, 25.10.1991.

Annex A (informative)

Derivation of equations used to calculate AVP

A.1 Theory

The measured air-saturated vapour pressure is the sum of the absolute vapour pressure of the liquid and the partial pressure of air that has come out of the solution.

A.2 Derivation of equation

The air-saturated vapour pressure after the first installment of sample, $ASVP_1$, may be expressed using the following equation:

$$ASVP_1 = AVP + PP_{v,1} \quad (A.1)$$

where

AVP is the absolute vapour pressure of the liquid, in kilopascals,

$PP_{v,1}$ is the partial pressure of the volume of air in the first test portion, in kilopascals.

The air-saturated vapour pressure after the second installment of sample, $ASVP_2$, may be expressed using the following equation:

$$ASVP_2 = AVP + PP_{v,1} + PP_{v,2} \quad (A.2)$$

where

$PP_{v,2}$ is the partial pressure of the volume of air in the second test portion, in kilopascals.

Therefore,

$$ASVP_2 - ASVP_1 = PP_{v,2} \quad (A.3)$$

or

$$2(ASVP_2 - ASVP_1) = PP_{v,1} + PP_{v,2} = PP \quad (A.4)$$

where

PP is the partial pressure of the air, in kilopascals.

However, this equation does not take account of compressive effects and the solubility of air in the liquid, when the second sample installment is injected. If these factors are taken account of, then the following equation applies:

$$PP_c = 2(ASVP_2 - ASVP_1) \left[1 - \left\{ \frac{(1 - k/100)V}{V_c} \right\} \right] \quad (A.5)$$

where

PP_c is the partial pressure of the air at injection temperature T_c , in kilopascals;

$V = V_1 = V_2$ is the volume of each installment of sample, in milliliters;

V_c is the volume of chamber, in milliliters;

k is the solubility of air in the sample;

T_c is the temperature of the test chamber when the test portion is injected, in degrees Celsius.

Using the standard conditions of $k = 25$ and $V = V_c/5$ the equation becomes:

$$PP_c = 1,7(ASVP_2 - ASVP_1) \quad (A.6)$$

A.3 Air correction at test temperature

The partial pressure of the air at the test temperatures, PP_T , may be determined using equation (A.6) and the gas laws, as follows:

$$PP_T = \frac{PP_c(273,2 + T)}{(273,2 + T_c)} \quad (A.7)$$

where

T_c is the temperature of the test chamber when the test portion is injected, in degrees Celsius;

T is the test temperature, in degrees Celsius.

A.4 Calculation

As stated in A.1:

$$ASVP_T = AVP_T + PP_T \quad (A.8)$$

where

AVP_T is the absolute vapour pressure at temperature T , in kilopascals;

$ASVP_T$ is the air-saturated vapour pressure at temperature T , in kilopascals;

PP_T is the partial pressure of the air at temperature T , in kilopascals.

Bibliography

- [1] IP 394, *Determination of air-saturated vapour pressure (ASVP)*.
- [2] ASTM D 5191, *Test method for vapor pressure of petroleum products (Mini Method)*.
- [3] EC Directive 85/536/EEC, *Council Directive on crude-oil savings through the use of substitute fuel components in petrol*.
- [4] EN ISO 3171, *Petroleum liquids – Automatic pipeline sampling (ISO 3171:1988)*.

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