BRITISH STANDARD

BS EN
13132:2000
BS
2000-466:2001

Methods of test for petroleum and its products —

BS 2000-466: Liquid petroleum products — Unleaded petrol — Determination of organic oxygenate compounds and total organically bound oxygen content by gas chromatography using column switching

(Identical with IP 466/2001)

The European Standard EN 13132:2000 has the status of a British Standard

ICS 75.160.20

National foreword

This British Standard is the official English language version of EN 13132:2000.

The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum testing and terminology, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

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

The Institute of Petroleum publishes and sells all parts of BS 2000, and all BS EN petroleum test methods that would be part of BS 2000, both in its annual publication "Standard methods for analysis and testing of petroleum and related products and British Standard 2000 parts" and individually.

Further information is available from:

The Institute of Petroleum, 61 New Cavendish Street, London W1M 8AR. Tel: 020 7467 7100. Fax: 020 7255 1472.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled "International Standards Correspondence Index", or by using the "Find" facility of the BSI Standards Electronic Catalogue.

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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 21 and a back cover.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 13132

March 2000

ICS 75.160.20

English version

Liquid petroleum products - Unleaded petrol - Determination of organic oxygenate compounds and total organically bound oxygen content by gas chromatography using column switching

Produit pétroliers liquides - Essence sans plomb - Détermination des composés oxygénés organiques et de la teneur totale en oxygène organique par chromatographie en phase gazeuse avec commutation de colonnes

This European Standard was approved by CEN on 14 February 2000.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. 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.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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

Ref. No. EN 13132:2000 E

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 19, Petroleum products, lubricants and related products, the Secretariat of which is held by NNI.

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 September 2000, and conflicting national standards shall be withdrawn at the latest by September 2000.

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

In this standard, annex A is normative and annex B is informative.

1 Scope

This European Standard specifies a gas chromatographic method using column switching for the quantitative determination of individual organic oxygenate compounds in the range 0,17 % (*m/m*) to 15 % (*m/m)* and total organically bound oxygen up 3,7 % (*m/m*) in unleaded petrol having a final boiling point not greater than 220 °C.

NOTE 1 The final boiling point can be measured by using prEN ISO 3405:1998 [1)].

NOTE 2 Fur the purposes of this European Standard, the terms "% (*m/m*)" and "% (*V/V*)" are used to represent respectively the mass fraction and the volume fraction.

This European Standard is applicable to the determination of oxygen-containing compounds and total organically bound oxygen in unleaded petrol in line with the relevant EU Directives[2)].

WARNING The use of this standard can 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 determine the applicability of regulatory limitations prior to use.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN ISO 3170, *Petroleum liquids - Manual sampling (ISO 3170:1988, including Amendment 1:1998).*

EN ISO 3171, *Petroleum liquids - Automatic pipeline sampling (ISO 3171:1988).*

EN ISO 3675, *Crude petroleum and liquid petroleum products - Laboratory determination of density - Hydrometer method (ISO 3675:1998).*

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987).*

EN ISO 3838, *Crude petroleum and liquid or solid petroleum products - Determination of density or relative density - Capillary-stoppered pyknometer and graduated bicapillary pyknometer methods (ISO 3838:1983).*

EN ISO 12185, *Crude petroleum and petroleum products - Determination of density - Oscillating U-tube method (ISO 12185:1996)*.

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1) prEN ISO 34051998: Petroleum products - Determination of distillation characteristics at atmosphere pressure (ISO/DIS 3405:1998).

2) EU Directive 85/210/EEC, Council Directive on the approximation of the laws of the Member States concerning the lead content of petrol. EU Directive 85/536/EEC, Council Directive on crude-oil savings through the use of substitute fuel components in petrol.

3 Principle

Oxygen containing organic compounds are isolated from the sample using a first capillary column. In a second capillary column the oxygen containing organic compounds are separated, and detected individually using a flame ionization detector.

NOTE Guidance on the column switching technique is given in annex B.

4 Reagents and materials

Use only reagents of recognized analytical grade and water conforming to grade 3 of EN ISO 3696.

4.1 Carrier gas

Hydrogen, helium, or nitrogen, free of hydrocarbons.

WARNING Hydrogen is explosive when mixed with air at concentrations ranging approximately from 4 % (*V/V*) to 75 % (*V/V*). All joints and lines carrying hydrogen shall be made gas tight to prevent leakage of hydrogen into a confined space.

4.2 Reagents for the preparation of calibration samples

Reagents shall be not less than 99,0 % (*m/m*) pure.

Calibration samples may be combinations of the following reagents:

methanol	CH_3OH	methyl alcohol; MEOH;
ethanol	CH_3CH_2OH	ethyl alcohol; ETOH;
propan-1-ol	$CH_3CH_2CH_2OH$	propyl alcohol; NPA;
propan-2-ol	$(CH_3)_2CHOH$	iso-propyl alcohol; IPA;
butan-1-ol	$CH_3[CH_2]_3OH$	butyl alcohol; NBA;
butan-2-ol	$CH_3CH(OH)CH_2CH_3$	*sec*-butyl alcohol; SBA;
2-methylpropan-2-ol	$(CH_3)_3COH$	*tert*-butyl alcohol; TBA;
2-methylpropan-1-ol	$(CH_3)_2CHCH_2OH$	iso-butyl alcohol; IBA;
pentan-2-ol	$CH_3CH(OH)CH_2CH_2CH_3$	*sec*-amyl alcohol; SAA;
tert-butyl methyl ether	$(CH_3)_3CO\ CH_3$	methyl tertiary butyl ether; MTBE;
methyl *tert*-pentyl ether	$(CH_3)_2C(OCH_3)CH_2CH_3$	tertiary amyl methyl ether; TAME;
ethyl *tert*-pentyl ether	$(CH_3)_2C(OCH_2CH_3)CH_2CH_3$	ethyl tertiary amyl ether; ETAE;
acetone	$(CH_3)_2CO$	
butanone	$CH_3CH_2COCH_3$	methyl ethyl ketone; MEK
tert-butyl ethyl ether	$(CH_3)_3CO\ CH_2CH_3$	ethyl tertiary butyl ether; ETBE

4.3 Internal standards

Use one of the reagents listed in 4.2. If all of these reagents are likely to be present in the sample under test, use a different organic oxygenate compound of the same purity and similar volatility.

4.4 Petrol containing no organic oxygenate compounds, or n-heptane

Petrol which has been examined to ensure that it contains no organic oxygenate compounds detectable by this method, or n-heptane.

5 Apparatus

Usual laboratory apparatus and glassware, together with the following:

5.1 Gas chromatographic assembly

5.1.1 Gas chromatograph, provided with a means for column switching, equipped with a programmable oven temperature controller, or controllers in the case of dual oven gas chromatographs, and a flame ionization detector (FID).

NOTE It is recommended that a system constructed entirely of glass from the sample injection port up to the detector system is used since petrol may contain organic oxygenate compounds which can give rise to corrosion and changes of retention times in systems constructed using metals.

5.1.2 Two capillary columns

NOTE Recommended columns are described in annex B.

The columns shall be coated with a suitable phase so that the required resolution between the components and between the components and the matrix to be determined, shall be at least 1 after elution from the second column.

The resolution, R, between peaks A and B (see figure 1) shall be calculated as follows:

$$R = 1{,}18\,\frac{t'_B - t'_A}{w_A + w_B}$$

where:

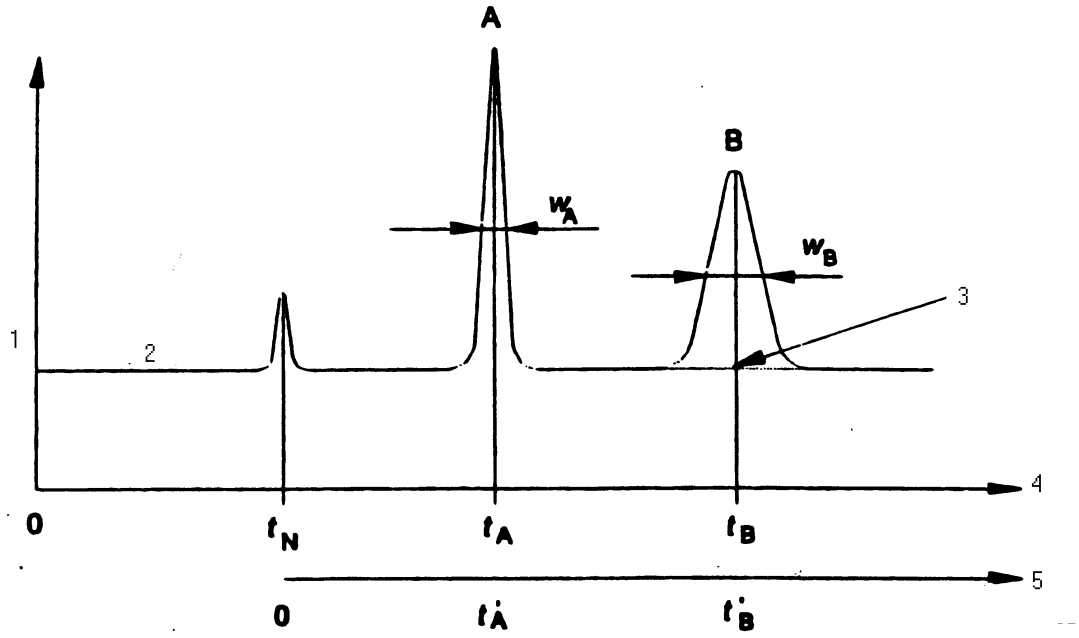
t'_A is the retention time of component A in s;

t'_B is the retention time of component B in s;

w_A is the peak width at half-height of component A in s;

w_B is the peak width at half-height of component B in s;

1,18 is a factor originating from a peak resolution equation.



Key

1 Start
2 Zero line
3 Baseline
4 Time axis
5 Time axis

NOTE t_N is the hold-up time zero of the column, i.e. the time taken for an inert component, such as methane, to travel through the column without chromatography taking place.

Figure 1 - Calculation of the resolution between peaks A and B

5.1.3 Device for the control of the flow of carrier gas

5.1.4 Recorder and/or integrator

An amplifier and potentiometric recording device, or an integrator or data processor systems, giving area values corresponding to the peak area.

5.2 Injection device

5.3 Test sample container, normally with a capacity of between 10 ml and 100 ml, fitted with a self-sealing rubber septum coated with polytetrafluoroethylene (PTFE).

6 Sampling

Unless otherwise specified in the commodity specification, samples shall be taken in accordance with EN ISO 3170 or EN ISO 3171, and/or in accordance with the requirements of national standards or regulations for the sampling of petrol.

7 Procedure

7.1 Setting up the apparatus

7.1.1 General

Prepare the equipment and set the test conditions in accordance with the manufacturer's instructions.

7.1.2 Carrier gas

Adjust the pressure and flow rate of the carrier gas to levels such that the resolutions are in accordance with 5.1.2.

7.2 Calibration

Prepare the calibration sample by combining known masses of organic oxygenate compounds (4.2), with the internal standard (4.3), and diluting them to a known mass with the petrol or n-heptane (4.4).

NOTE The calibration sample should contain the same organic oxygenate compounds in similar proportions as present in the sample under test.

Inject a suitable quantity of the prepared calibration sample into the gas chromatograph such that the capacity of the columns and other components is not exceeded and the linearity of the detector is not impaired.

Determine and record the retention times, t_i, for all the components i to be evaluated. Calculate the calibration factor, f_i, for all the components i to be evaluated, using the equation:

$$f_i = \frac{m_i \times A_{st}}{A_i \times m_{st}}$$

where:

m_i is the mass, in grams, of component i in the calibration sample;

A_{st} is the peak area, in microvoltseconds or in square millimetres, of the internal standard;

A_i is the peak area, in microvoltseconds or in square millimetres, of component i;

m_{st} is the mass, in grams, of the internal standard in the calibration sample.

Record the calibration factor for each component.

7.3 Determination of density

Determine the density at 15 °C, ρ_S, of the sample in accordance with EN ISO 3675, EN ISO 3838 or EN ISO 12185 and record the result to the nearest 0,1 kg/m^3.

7.4 Preparation of test sample

Cool the sample to between 5 °C and 10 °C. Weigh, to the nearest 0,1 mg, the test sample container (5.3) and its rubber septum without sealing it.

Transfer a quantity of the internal standard (4.3) to the test sample container and weigh, to the nearest 0,1 mg, the test sample container with contents and septum, without sealing the sample container. The mass, m_{st}, in grams, of the internal standard shall amount to between 2 % (*m/m*) and 5 % (*m/m*) of the sample, m_s, but shall not be less than 0,050 g.

Transfer a quantity, normally between 5 ml and 100 ml, of the cooled sample to the test sample container and seal immediately with the septum. Weigh, to the nearest 0,1 mg, the test sample container and contents. Record the mass, m_s, in grams, of the test sample taken, to the nearest 0,1 mg.

Record the amount of internal standard in the prepared test sample as a percentage by mass. Mix the contents of the test sample container by shaking until homogeneous.

7.5 Introduction of test portion

Inject a suitable quantity of the prepared test sample (7.4) into the gas chromatograph. Ensure that the test portion size is such that the capacity of the columns and other components of the gas chromatograph is not exceeded and that the linearity of the detector is not impaired.

7.6 Examination of chromatogram

Examine the chromatogram and identify the components of the test portion by means of their retention times (see 7.2).

8 Calculation

8.1 Calculation of mass of each component in the test sample

Calculate the mass, m_i, in grams, of each component i of the test sample using the equation:

$$m_i = \frac{A_i \times f_i \times m_{st}}{A_{st}}$$

where:

A_i is the peak area, in microvoltseconds or in square millimetres, of component i;

f_i is the calibration factor for component i;

m_{st} is the mass, in grams, of the internal standard included in the test sample (7.4);

A_{st} is the peak area, in microvoltseconds or in square millimetres, of the internal standard.

8.2 Calculation of each component in the sample as a percentage by mass

Calculate as a percentage by mass, ω_i, each component i in the sample using the equation:

$$\omega_i = \frac{m_i}{m_s} \times 100$$

8.3 Calculation of each component in the sample as a percentage by volume

Calculate as a percentage by volume, ϕ_i, of each component i in the sample using the equation:

$$\phi_i = \frac{V_i}{V_s} \times 100$$

where:

V_i is the volume, in millilitres, of component i;

V_s is the volume, in millilitres, of the sample taken (7.4).

The volume, V_i, of component i is calculated from the mass of each component, the densities given in annex A and the density of the sample (7.3), using the general equation:

$$\text{volume} = \frac{\text{mass}}{\text{density}}$$

For component, i, this becomes

$$V_i = \frac{m_i \times 1\,000}{\rho_i}$$

where:

ρ_i is the density in kilograms per cubic metre at 15 °C of component i.

The volume, V_s, of the sample taken is calculated using the equation:

$$V_s = \frac{m_s \times 1\,000}{\rho_s}$$

8.4 Total organically bound oxygen

Calculate the total content of organically bound oxygen, Ω, as a percentage by mass, from the percentages by mass of the individual components, after identification, using the equation:

$$\Omega = \Sigma \frac{\omega_i \times 16{,}00}{W_i}$$

where:

W_i is the molecular mass of component i.

EXAMPLE If the sample has been determined to contain 2 % (*m/m*) methanol and 4 % (*m/m)* ethanol

then: ω_i for methanol is 2 % (*m/m*), W_i is 32,04;

and: ω_i for ethanol is 4 % (*m/m*), W_i is 46,07;

$$\Omega = \frac{2 \times 16{,}00}{32{,}04} + \frac{4 \times 16{,}00}{46{,}07} = 1{,}00 + 1{,}39 = 2{,}39\ \%\ (m/m)$$

9 Expression of results

Report the contents of each component to the nearest 0,1 % ((*m/m*) or % (*V/V*)).

Report the total content of organically bound oxygen to the nearest 0,01 % (*m/m*).

10 Precision

10.1 Repeatability

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 in table 1 and table 2 only in one case in twenty.

10.2 Reproducibility

The difference between two single and independent 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 in table 1 and table 2 only in one case in twenty.

Table 1 - Repeatability and reproducibility of the determination of organic oxygenate compounds

Organic oxygenates compounds % (*m/m)* or % (*V/V)*			Repeatability % (*m/m)* or % (*V/V)*	Reproducibility % (*m/m)* or % (*V/V)*
0,1	to	1,0	0,05	0,1
> 1,0	to	3,0	0,1	0,3
> 3,0	to	5,0	0,1	0,4
> 5,0	to	7,0	0,2	0,5
> 7,0	to	9,0	0,2	0,6
> 9,0	to	11,0	0,2	0,8
> 11,0	to	13,0	0,3	0,9
> 13,0	to	15,0	0,3	1,0

Table 2 - Repeatability and reproducibility of the determination of the total organically bound oxygen content

Total organically bound oxygen % (*m/m)*	Repeatability % (*m/m)*	Reproducibility % (*m/m)*
1,5 to 3,0	0,08	0,3

11 Test report

The report shall include at least the following information:

a) the type and identification of the product under test;

b) a reference to this European Standard;

c) the sampling procedure used (see clause 6);

d) the density of the sample (see 7.3);

e) the result of the test (see clause 9);

f) any deviation from the procedure described;

g) the date of test.

ANNEX A
(normative)

Densities at 15 °C of organic oxygen containing compounds.

Substance	Density at 15 °C, kg/m^3
Methanol, CH_3OH	795,8
Ethanol, CH_3CH_2OH	794,8
Propan-1-ol, $CH_3CH_2CH_2OH$	813,3
Propan-2-ol, $(CH_3)_2CHOH$	789,5
Butan-1-ol, $CH_3(CH_2)_3OH$	813,3
Butan-2-ol, $CH_3CH(OH)CH_2CH_3$	810,6
2-methylpropan-2-ol, $(CH_3)_3COH$	791,0
2-methylpropan-1-ol, $(CH_3)_2CHCH_2OH$	805,8
Pentan-1-ol, $CH_3(CH_2)_4OH$	818,5
Pentan-2-ol, $CH_3CH(OH)CH_2CH_2CH_3$	813,5
Pentan-3-ol, $CH_3CH_2CH(OH)CH_2CH_3$	824,6
2-methylbutan-1-ol, $C_2H_5CH(CH_3)CH_2OH$	823,5
3-methylbutan-1-ol, $CH_3CH(CH_3)C_2H_4OH$	816,3
2-methylbutan-2-ol, $(CH_3)_2C(OH)CH_2CH_3$	813,5
3-methylbutan-2-ol, $CH_3C(CH_3)CH(OH)CH_3$	822,8
Hexan-1-ol, $CH_3(CH_2)_5OH$	822,5
Hexan-2-ol, $CH_3(CH_2)_3CH(OH)CH_3$	818,2
Hexan-3-ol, $CH_3CH_2CH_2CH(OH)CH_2CH_3$	822,7
2-methylpentan-1-ol, $CH_3CH_2CH_2CH(CH_3)CH_2OH$	827,9
3-methylpentan-1-ol, $CH_3CH_2CH(CH_3)CH_2CH_2OH$	826,1
4-methylpentan-1-ol, $CH_3CH(CH_3)CH_2CH_2CH_2OH$	816,6
2-methylpentan-2-ol, $CH_3C(CH_3)C(OH)(CH_2)_3CH_3$	817,7
3-methylpentan-2-ol, $CH_3CH(OH)CH(CH_3)CH_2CH_3$	833,3
4-methylpentan-2-ol, $CH_3CH(OH)CH_2(CH_3)_2$	811,3
2-methylpentan-3-ol, $(CH_3)_2CHCH(OH)CH_2CH_3$	829,0
3-methylpentan-3-ol, $CH_3CH_2C(CH_3)(OH)CH_2CH_3$	828,9
2-ethylbutan-1-ol, $CH_3CH_2CH(CH_2OH)CH_2CH_3$	837,4
2,2-dimethylbutan-1-ol, $CH_3CH_2C(CH_3)_2CH_2OH$	832,6
2,3-dimethylbutan-2-ol, $CH(CH_3)CH(CH_3)CH_2OH$	826,9
3,3-dimethylbutan-2-ol, $C(CH_3)C_2H_4OH$	823,1

Substance	Density at 15 °C, kg/m^3
Heptan-1-ol, $CH_3(CH_2)_6OH$	825,9
Heptan-2-ol, $CH_3(CH_2)_4CH(OH)CH_3$	821,7
Heptan-3-ol, $CH_3(CH_2)_3CH(OH)CH_2CH_3$	825,2
Heptan-4-ol, $CH_3CH_2CH_2CH(OH)CH_2CH_2CH_3$	822,8
2-methylhexan-2-ol, $(CH_3)C(OH)CH_3(CH_2)_3CH_3$	818,3
2-methylhexan-3-ol, $(CH_3)_2CHCH(OH)CH_2CH_2CH_3$	827,9
3-methylhexan-3-ol, $CH_3CH_2C(CH_3)(OH)CH_2CH_2CH_3$	828,9
3-ethylpentan-3-ol, $(CH_3CH_2)_3COH$	848,2
2,4-dimethylpentan-3-ol, $(CH_3)_2CHCH(OH)(CH_3)_2$	835,1
Octan-1-ol, $CH_3(CH_2)_7OH$	828,8
Octan-2-ol, $CH_3(CH_2)_5CHOHCH_3$	824,0
Octan-3-ol, $CH_3(CH_2)_4CHOHCH_2CH_3$	824,5
Octan-4-ol, $CH_3(CH_2)_3CHOHCH_2CH_2CH_3$	823,5
2-methylheptan-1-ol, $CH_3(CH_2)_4CH(CH_3)CH_2OH$	805,7
3-methylheptan-1-ol, $CH_3(CH_2)_3CH(CH_3)CH_2CH_2OH$	791,8
4-methylheptan-1-ol, $CH_3CH_2CH_2CH(CH_3)(CH_2)_2CH_2OH$	813,7
5-methylheptan-1-ol, $CH_3CH_2CH(CH_3)(CH_2)_3CH_2OH$	822,3
6-methyheptan-1-ol, $CH_3CH(CH_3)(CH_2)_4CH_2OH$	824,4
2-methylheptan-2-ol, $CH_3(CH_2)_4C(CH_3)(OH)CH_3$	811,0
3-methylheptan-2-ol, $CH_3(CH_2)_3CH(CH_3)CH(OH)CH_3$	793,8
4-methylheptan-2-ol, $CH_3CH(OH)CH_2CH(CH_3)CH_2CH_2CH_3$	806,2
5-methylheptan-2-ol, $CH_3CH_2CH(CH_3)CH_2CH_2CH(OH)CH_3$	817,0
6-methylheptan-2-ol, $(CH_3)_2CH(CH_2)_3CH(OH)CH_3$	810,7
2-methylheptan-3-ol, $CH_3(CH_2)_3CH(OH)CH(CH_3)_2$	828,6
3-methylheptan-3-ol, $CH_3(CH_2)_3C(OH)(CH_3)CH_2CH_3$	833,3
4-methylheptan-3-ol, $CH_3(CH_2)_2CH(CH_3)CH(OH)CH_2CH_3$	803,1
5-methylheptan-3-ol, $CH_3CH_2CH(CH_3)CH_2CH(OH)CH_2CH_3$	822,0
6-methylheptan-3-ol, $CH_3CH_2CH(OH)CH_2CH_2CH(CH_3)_2$	784,9
2-methylheptan-4-ol, $(CH_3)_2CHCH_2CH(OH)CH_2CH_2CH_3$	817,2
3-methylheptan-4-ol, $CH_3CH_2CH(CH_3)CH(OH)CH_2CH_2CH_3$	841,2
4-methylheptan-4-ol, $CH_3(CH_2)_2C(OH)(CH_3)CH_2CH_2CH_3$	828,0
2-ethylhexan-1-ol, $CH_3(CH_2)_3CH(CH_2CH_3)CH_2OH$	835,3
3-ethylhexan-3-ol, $CH_3(CH_2)_2C(OH)(CH_2CH_3)CH_2CH_3$	841,7
Nonan-1-ol, $CH_3(CH_2)_8OH$	831,7

Substance	Density at 15 °C, kg/m^3
Nonan-2-ol, $CH_3(CH_2)_6CH(OH)CH_3$	826,7
Nonan-3-ol, $CH_3(CH_2)_5CH(OH)CH_2CH_3$	830,2
2-methyloctan-2-ol, $CH_3(CH_2)_5C(OH)(CH_3)_2$	821,5
2-mehtyloctan-3-ol, $CH_3(CH_2)_4CH(OH)CH(CH_3)_2$	833,0
3-methyloctan-3-ol, $CH_3(CH_2)_4C(OH)(CH_3)CH_2CH_3$	836,7
4-methyloctan-4-ol, $CH_3(CH_2)_3C(OH)(CH_3)(CH_2)CH_3$	832,3
Decan-2-ol, $CH_3(CH_2)_7CH(OH)CH_3$	829,0
Decan-3-ol, $CH_3(CH_2)_6CH(OH)CH_2CH_3$	831,0
Decan-4-ol, $CH_3(CH_2)_5CH(OH)(CH_2)_2CH_3$	828,7
Decan-5-ol, $CH_3(CH_2)_4CH(OH)(CH_2)_3CH_3$	828,8
2-methylnonan-1-ol, $CH_3(CH_2)_6CH(CH_3)CH_2OH$	839,2
2-methylnonan-3-ol, $CH_3(CH_2)_5CH(OH)CH(CH_3)_2$	832,0
tert-butyl methyl ether, $(CH_3)_3COCH_3$	745,3
methyl *tert*-pentyl ether, $(CH_3)_2C(OCH_3)CH_2CH_3$	775,2
tert-butyl ethyl ether, $(CH_3)_3COCH_2CH_3$	745,6
ethyl *tert*-pentyl ether, $(CH_3)_2C(OCH_2CH_3)CH_2CH_3$	774,9
methyl propyl ether, $CH_3O\ CH_2CH_2CH_3$	730,2
isopropyl methyl ether, $(CH_3)_2CHOCH_3$	720,5
Diethyl ether, $CH_3CH_2OCH_2CH_3$	719,2
butyl methyl ether, $CH_3O(CH_2)_3CH_3$	749,2
isobutyl methyl ether, $(CH_3)_2CHCH_2OCH_3$	737,5
but-2-yl methyl ether, $CH_3CH_2CH(CH_3)OCH_3$	746,7
ethyl propyl ether, $CH_2CH_2OCH_2CH_2CH_3$	741,2
ethyl isopropyl ether, $(CH_3)_2CHOCH_2CH_3$	728,1
methyl pentyl ether, $CH_3O(CH_2)_4CH_3$	764,2
methyl isopentyl ether, $CH_3CH_2(CH_3)_2COCH_3$	758,4
butyl ethyl ether, $CH_3(CH_2)_3OCH_2CH_3$	754,3
ethyl isobutyl ether, $(CH_3)_2CHCH_2OCH_2CH_3$	744,2
sec-butyl ethyl ether, $(CH_3)(CH_3CH_2)COCH_2CH_3$	748,2
Dipropyl ether, $CH_3CH_2CH_2OCH_2CH_2CH_3$	751,6
isopropyl propyl ether, $(CH_3)_2CHOCH_2CH_2CH_3$	742,5
Diisopropyl ether, $(CH_3)_2CHOCH(CH_3)_2$	729,2
hexyl methyl ether, $CH_3O(CH_2)_5CH_3$	774,9
ethyl pentyl ether, $CH_3CH_2O(CH_2)_4CH_3$	765,9

Substance	Density at 15 °C, kg/m^3
ethyl isopentyl ether, $CH_3CH_2OCH_2CH_2CH(CH_3)_2$	761,3
butyl propyl ether, $CH_3(CH_2)_2O(CH_2)_3CH_3$	763,3
isobutyl propyl ether, $(CH_3)_2CHCH_2OCH_2CH_2CH_3$	753,3
sec-butyl propyl ether, $(CH_3)(CH_3CH_2)CHOCH_2CH_2CH_3$	759,4
tert-butyl propyl ether, $(CH_3)_3COCH_2CH_2CH_3$	758,2
butyl isopropyl ether, $(CH_3)_2CHO(CH_2)_3CH_3$	755,4
isobutyl isopropyl ether, $(CH_3)_2CHCH_2OCH(CH_3)_2$	744,6
sec-butyl isopropyl ether, $(CH_3)(CH_3CH_2)CHOCH(CH_3)_2$	749,0
tert-butyl isopropyl ether, $(CH_3)_3COCH(CH_3)_2$	746,0
heptyl methyl ether, $CH_3O(CH_2)_6CH_3$	783,8
ethyl hexyl ether, $CH_3CH_2O(CH_2)_5CH_3$	777,7
pentyl propyl ether, $CH_3CH_2CH_2O(CH_2)_4CH_3$	774,0
isopentyl propyl ether, $CH_3CH_2CH_2O(CH_2)_2CH(CH_3)_2$	768,7
isopropyl pentyl ether, $(CH_3)_2CHO(CH_2)_4CH_3$	768,1
isopentyl isopropyl ether, $(CH_3)_2CHO(CH_2)_2CH(CH_3)_2$	763,4
Dibutyl ether, $CH_3(CH_2)_3O(CH_2)_3CH_3$	772,5
butyl isobutyl ether, $(CH_3)_2CHCH_2O(CH_2)_3CH_3$	764,0
butyl *sec*-butyl ether, $(CH_3)(CH_2H_5)CHO(CH_2)_3CH_3$	769,6
butyl *tert*-butyl ether, $(CH_2)_3CO(CH_2)_3CH_3$	767,2
diisobutyl ether, $(CH_3)_2CHCH_2OCH_2CH(CH_3)_2$	754,1
sec-butyl isobutyl ether, $(CH_3)(CH_3CH_2)CHOCH_2CH(CH_3)_2$	759,8
tert-butyl isobutyl ether, $(CH_3)_3COCH_2CH(CH_3)_2$	757,4
di-*sec*-butyl ether, $CH_3CH_2CH(CH_3)OCH(CH_3)CH_2CH_3$	767,5
di-*tert*-butyl ether, $(CH_3)_3COC(CH_3)_3$	766,2
sec-butyl tert-butyl ether, $(CH_3)(CH_3CH_2)CHO(CH_3)_3$	766,9
methyl octyl ether, $CH_3O(CH_2)_7CH_3$	790,9
ethyl hexyl ether, $CH_3CH_2O[CH_2]_6CH_3$	783,8
hexylpropyl ether, $CH_3CH_2CH_2O(CH_2)_5CH_3$	781,3
hexyl isopropyl ether, $(CH_3)_2CHO(CH_2)_5CH_3$	775,9
butyl pentyl ether, $CH_3(CH_2)_3O(CH_2)_4CH_3$	780,4
butyl 2-methyl butyl ether, $(CH_3)(CH_3CH_2)CHCH_2OCH_2CH_2CH_2CH_3$	775,8
isobutyl pentyl ether, $(CH_3)_2CHCH_2O(CH_2)_4CH_3$	774,0
isobutyl isopentyl ether, $(CH_3)_2CHCH_2O(CH_2)_2CH(CH_3)_2$	787,7
sec-butyl pentyl ether, $CH_3CH_2CH(CH_3)O(CH_2)_4CH_3$	777,2

Substance	Density at 15 °C, kg/m^3
sec-butyl isopentyl ether, $CH_3CH_2CH(CH_3)O(CH_2)_2CH(CH_3)_2$	772,9
tert-butyl pentyl ether, $(CH_3)_3CO(CH_2)_4CH_3$	775,1
tert-butyl isopentyl ether, $(CH_3)CO(CH_2)_2CH(CH_3)_2$	770,5
methyl nonyl ether, $CH_3O(CH_2)_8CH_3$	796,6
ethyl nonyl ether, $CH_3CH_2O(CH_2)_8CH_3$	790,2
heptyl propyl ether, $CH_3(CH_2)_2O(CH_2)_6CH_3$	787,8
heptyl isopropyl ether, $(CH_3)_2CHO(CH_2)_6CH_3$	781,7
butyl hexyl ether, $CH_3(CH_2)_3O(CH_2)_5CH_3$	787,0
hexyl isobutyl ether, $(CH_3)_2CHCH_2O(CH_2)_5CH_3$	779,3
sec-butyl hexyl ether, $CH_3CH_2CH(CH_3)O(CH_2)_5CH_3$	783,9
dipentyl ether, $CH_3(CH_2)_4O(CH_2)_4CH_3$	787,0
2-methylbutyl pentyl ether, $(CH_3)(CH_3CH_2)CHCH_2OCH_2CH_2CH_2CH_2CH_3$	783,1
isopentyl 2-methylbutyl ether, $(CH_3)(CH_3CH_2)CHCH_2OCH_2CH_2(CH_3)_2$	779,4
decyl methyl ether, $CH_3O(CH_2)_9CH_3$	801,5
ethyl nonyl ether, $CH_3CH_2O(CH_2)_8CH_3$	795,6
octyl propyl ether, $CH_3CH_2CH_2O(CH_2)_7CH_3$	793,9
isopropyl octyl ether, $(CH_3)_2CHO(CH_2)_7CH_3$	787,9
butyl heptyl ether, $CH_3(CH_2)_3O(CH_2)_6CH_3$	792,8
hexyl pentyl ether, $CH_3(CH_2)_4O(CH_2)_5CH_3$	792,3
decyl ethyl ether, $CH_3CH_2O(CH_2)_9CH_3$	800,2
nonyl propyl ether, $CH_3(CH_2)_2O(CH_2)_8CH_3$	798,6
butyl octyl ether, $CH_3(CH_2)_3O(CH_2)_7CH_3$	797,5
heptyl pentyl ether, $CH_3(CH_2)_4O(CH_2)_6CH_3$	797,4
dihexyl ether, $CH_3(CH_2)_5O(CH_2)_5CH_3$	800,0
Acetone, $(CH_3)_2CO$	795,8
Butanone, $CH_3CH_2COCH_3$	810,0

ANNEX B
(informative)

Guidance on the column switching technique

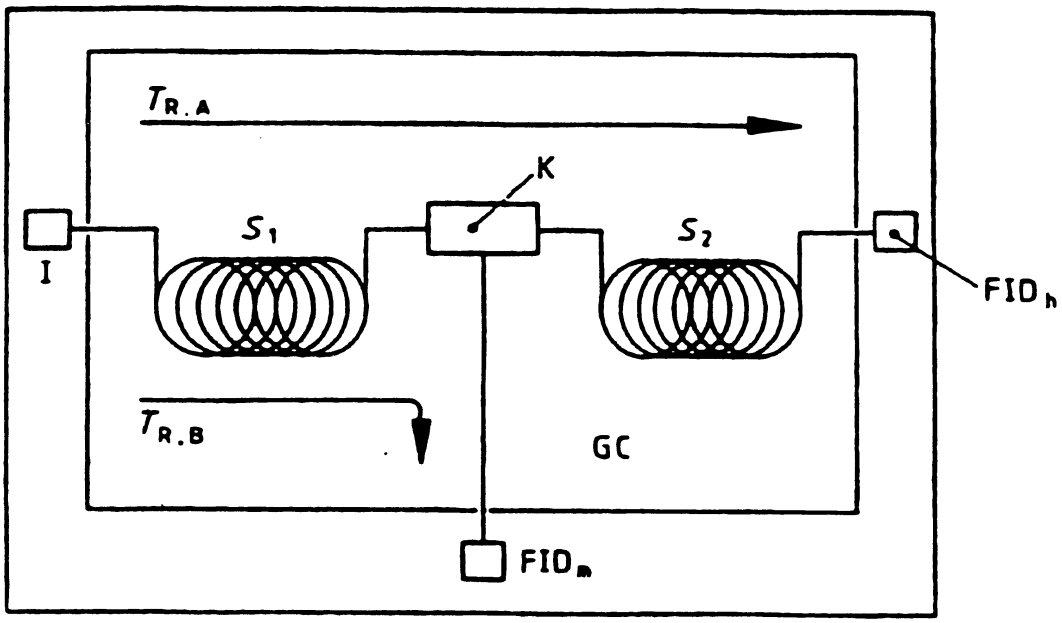
B.1 Introduction

Column switching gas chromatography is used to increase the separating power of a gas chromatography system by the further separation of unresolved components using additional columns (multidimensional chromatograph).

Valves suitable for column switching have a low switching volume in relation to peak volumes and do not show any interaction with the sample. As a substitute for valves, the gas flow through the columns may be altered by changing the pressure (Deans or off-line switching). For example, figure A.1 illustrates how the valveless flow switching technique functions. The central point of the system is a coupler through which the carrier gas flows and which can be pneumatically switched. Flow into the coupler can be selected freely in terms of amount and direction. The sample can be directed from column 1 into column 2 or towards special detectors without affecting the shape of the peak.

NOTE The same basic principle is used for the determination of benzene in petrol, described in EN 12177[3)].

3) EN 12177: Liquid petroleum products - Unleaded petrol - Determination of benzene content by gas chromatography.



Key

GC	Gas Chromatograph with a column oven
I	injector with splitting
S_1	capillary separation column (first column)
S_2	capillary separation column (main column)
K	coupler in accordance with Deans method
$T_{R,A}$	carrier gas flow in straight setting
$T_{R,B}$	carrier gas flow in heart-cut setting
FID	FID monitor detector
FID_h^m	FID main detector

Figure B.1 - Illustration of pneumatic separation flow switches via coupler in accordance with Deans method

B.2 General apparatus parameters for the flow switching technique

The following apparatus parameters have been found to be suitable. When similar apparatus is used, deviations from the data obtained can occur. In each case this should be optimized in accordance with the manufacturer's instructions.

Apparatus:	gas chromatograph with Deans switching;
Detector:	flame ionization detector;
Temperature of injection block:	150 °C;
Carrier gas:	nitrogen;
Split:	1 to 80;
Injection amount:	0,5 µl;
Oven 1:	40 °C;
Temperature programme:	40 °C for 6 min, then increasing at 5 °C/min to 120 °C;
Column 1:	50 m x 0,25 mm internal diameter, fused silica coated with 0,4 µm tris-cyanoethoxy-propane (TCEP);
Oven 2: (Optional):	40 °C;
Temperage programme:	40 °C for 9 min, then increasing at 5 °C/min to 120 °C;
Column 2:	25 m x 0,25 mm internal diameter, fused silica coated with 0,4 µm methyl silicone[4)].

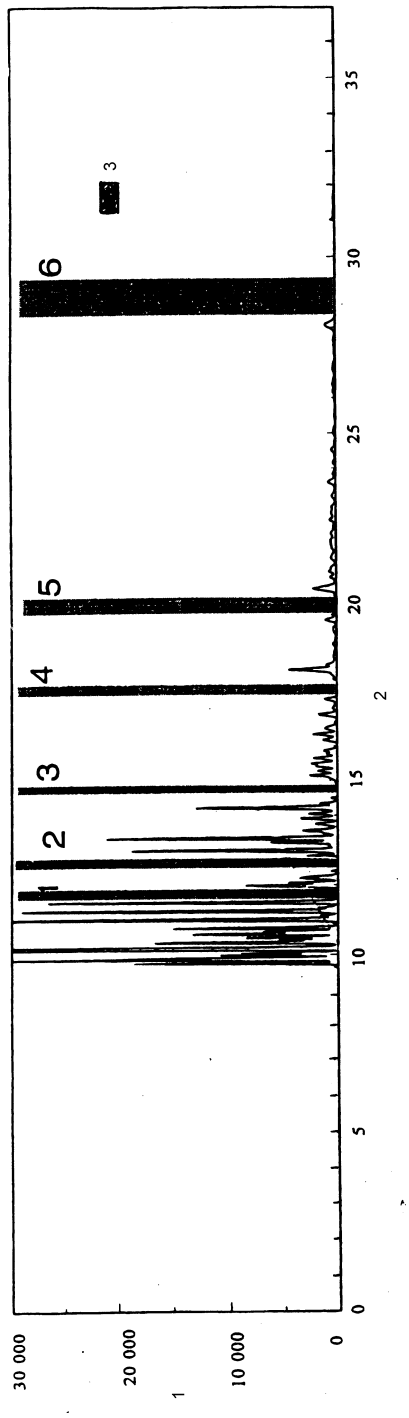
B.3 Gas chromatograms

Figure B.2 and B.3 are examples of gas chromatograms obtained from the determination of oxygenates in petrol using the column switching technique. The retention times for individual components may be determined using appropriate reference materials.

Figure B.2 illustrates the separation from the first column.

Figure B.3 illustrates the separation from the second column.

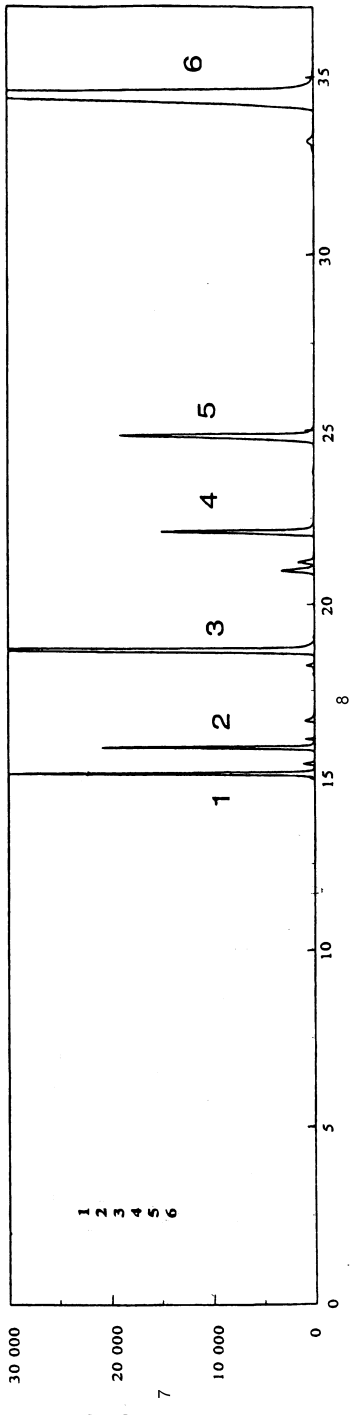
[4)] An example of a suitable methyl silicone product commercially available is OV1. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN of this product.



Key

1 peak area (mm²)
2 time (mins)
3 windows (cuts)

Figure B.2 - Typical FID chromatogram of petrol



Key

1 MTBE
2 Methanol
3 2-methylpropan-2-ol
4 Benzene
5 Standard
6 Toluene
7 Peak area (mm²)
8 time (mins)

Figure B.3 - Chromatogram of organic oxygenate compounds in petrol determined using column switching

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