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**Komercialni propan in butan - Analiza s plinsko kromatografijo (ISO 7941:1988)**

Commercial propane and butane - Analysis by gas chromatography (ISO 7941:1988)

Handelsübliches Propan und Butan - Gaschromatographische Analyse (ISO 7941:1988)

Propanes et butanes commerciaux - Analyse par chromatographie en phase gazeuse  
(ISO 7941:1988)

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**Ta slovenski standard je istoveten z: EN 27941:1993**

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**ICS:**

75.160.30

Plinska goriva

Gaseous fuels

**SIST EN 27941:1998**

**en**

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EUROPEAN STANDARD

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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

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## Foreword

This European Standard is the endorsement of ISO 7941:1988. Endorsement of ISO 7941 was recommended by Technical Committee CEN/TC 19 "Methods of test and specifications for petroleum products" under whose competence this European Standard will henceforth fall.

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of an identical text or by endorsement, at the latest by April 1994, and conflicting national standards shall be withdrawn at the latest by April 1994.

The standard was approved and in accordance with the CEN/CENELEC Internal Regulations, the 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, United Kingdom.

## Endorsement notice

The text of the International Standard ISO 7941:1988 was approved by CEN as a European Standard without any modification.

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# INTERNATIONAL STANDARD

ISO  
7941

First edition  
1988-08-01



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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION  
ORGANISATION INTERNATIONALE DE NORMALISATION  
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

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## Commercial propane and butane — Analysis by gas chromatography

*Propanes et butanes commerciaux — Analyse par chromatographie en phase gazeuse*

**(standards.iteh.ai)**

SIST EN 27941:1998

<https://standards.iteh.ai/catalog/standards/sist/930fdd4-8aaf-417a-9a44-92068a9bf3b4/sist-en-27941-1998>

**ISO 7941 : 1988 (E)****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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 7941 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

# Commercial propane and butane — Analysis by gas chromatography

**WARNING: Safety precautions — When testing LPG it is essential to observe suitable safety precautions and any regulations applicable to installations, apparatus and storage. Particular attention shall be given to the following.**

- a) LPG can cause serious burns from the cold, and the liquid should not be allowed to contact the skin. When sampling LPG, goggles and gloves must be worn.
- b) Discharge of LPG can give rise to static electricity and it is essential to connect containers to "earth" prior to and during discharge.

If hydrogen is used as a carrier gas, special safety precautions shall be taken. More particularly, the hydrogen line shall be carefully tested for leaks, especially in the oven.

## 1 Scope and field of application

This International Standard specifies a gas chromatographic method for the quantitative determination of hydrocarbons in liquefied petroleum gas (LPG), excluding components whose concentrations are below 0,1 % (*m/m*). It is applicable to the analysis of propane, butane and their commercial mixtures, which may include saturated and unsaturated C<sub>2</sub>, C<sub>3</sub>, C<sub>4</sub> and C<sub>5</sub> hydrocarbons. It does not apply to "on-line" chromatography.

## 2 References

ISO 565, *Test sieves — Woven metal wire cloth, perforated plate and electroformed sheet — Nominal sizes of openings.*

ISO 4257, *Liquefied petroleum gases — Method of sampling.*<sup>1)</sup>

## 3 Principle

Physical separation by gas chromatography. Identification of the components by passing a standard reference mixture or pure hydrocarbons through the column, or by comparison with relative retention volumes of typical chromatograms. Calculation of concentrations of components by measuring peak areas and applying correction factors.

## 4 Definitions

**4.1 correction factor:** A factor applied to account for the fact that equal amounts of different components produce unequal signals in the detector.

**4.2 peak:** The portion of the chromatogram recording the detector response while a component is eluted from the column.

**4.2.1 peak area:** The area bounded by the peak and the baseline.

**4.2.2 peak height:** The distance between the peak maximum and the baseline.

**4.2.3 peak width:** The segment of the baseline intercepted by the tangents drawn at the inflection point of each side of the peak.

The **peak width at half height** is the segment of a line drawn parallel to the baseline at half the peak height which is intercepted by the peak sides.

If the baseline is seen to be sloping from the horizontal, both measurements are of the projection of these segments onto the horizontal axis.

**4.2.4 peak resolution:** The extent to which the peaks of two components overlap or are separated. It is expressed by means of the equation in 6.3.3. Values below 1 imply overlapping; values above 1 imply separation of the components.

## 4.3 Retention

**4.3.1 adjusted retention time [or volume]:** The time elapsed [or the volume of gas emerged from the column] between the moment of elution of unretained components (e.g. air or methane) and the moment of elution of the component in question, both referring to peak maxima.

<sup>1)</sup> To be published.

## ISO 7941 : 1988 (E)

When a flame ionization detector is used, the air peak time may be calculated from uncorrected retention times of three consecutive normal paraffins as follows:

$$t_0 = \frac{t_1 t_3 - t_2^2}{t_1 + t_3 - 2t_2}$$

where

- $t_0$  is the retention time for the unretained component;
- $t_1$  is the retention time for component 1;
- $t_2$  is the retention time for component 2;
- $t_3$  is the retention time for component 3.

**4.3.2 relative retention:** The ratio of the adjusted retention time [or volume] of a component to that of a standard reference component.

**4.4 internal normalization technique:** The technique by which the concentration of a component is found by comparing its corrected peak area (the product of its peak area and correction factor) with the sum of the corrected peak areas of all components.

## 5 Materials

### 5.1 Carrier gas

Hydrogen (see **warning** on page 1), helium or nitrogen, free of hydrocarbons, oxygen and water impurities.

### 5.2 Reference gases

Pure gases or a mixture of gases with certified compositions, boiling in the LPG range.

## 6 Apparatus

### 6.1 General

Apparatus for gas phase chromatography, or chromatograph, containing the following main elements and satisfying the requirements defined in 6.2 to 6.7:

- a) device for the control of the flow of carrier gas;
- b) injection device (see 6.2);
- c) oven with suitable column or columns;
- d) detector (see 6.4);
- e) recorder and, generally, integrator or computer (see 6.5).

### 6.2 Injection device

A liquid sample valve capable of delivering a liquid test portion of 0,5 to 1  $\mu$ l, or a gaseous sample valve capable of delivering a gaseous test portion of up to 0,5 ml.

### 6.3 Column

The types of column described in this clause have been found suitable and are recommended. Other columns may be used provided that the resolution performance quoted in 6.3.3 is achieved and provided that the relative retentions of other hydrocarbons are well known.

#### 6.3.1 Column material

The column should be made from glass, copper, stainless steel or aluminium tubing and have the following dimensions and form.

##### 6.3.1.1 Dimensions

- a) For commercial propane, 8 m of di-*n*-butyl maleate packing + 3 m of  $\beta\beta'$ -oxy-dipropionitrile packing.
- b) For commercial butane, 8 m of di-*n*-butyl maleate.
- c) For both applications, alternatively 6 m of sebaconitrile (1,8-dicyano-octane) packing.

Tubing with an internal diameter between 2 mm and 5 mm is recommended. The external diameter of the tubing should be appropriate to the chromatograph.

##### 6.3.1.2 Form

Any suitable coil shape that will fit into the oven without acute bends.

### 6.3.2 Packing

#### 6.3.2.1 Solid support

Chromosorb P<sup>1)</sup>, acid washed and sieved to obtain the portion between 180  $\mu$ m and 250  $\mu$ m (see ISO 565).

#### 6.3.2.2 Stationary phase

Chemical identity:

- di-*n*-butyl maleate and  $\beta\beta'$ -oxy-dipropionitrile [see 6.3.1.1 a) and 6.3.1.1 b)]
- sebaconitrile (1,8-dicyano-octane) [6.3.1.1 c)]

1) Chromosorb P is the trade-name of a commercially available product. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.



Level of loading:

- 25 g of stationary phase per 75 g of support for columns 6.3.1.1 a) and 6.3.1.1 b)
- 20 g of stationary phase per 80 g of support for column 6.3.1.1 c).

Solvent:

- pentane for columns 6.3.1.1 a) and 6.3.1.1 b)
- dichloromethane or toluene for column 6.3.1.1 c).

Procedure for coating:

- Dissolve 25 g [or 20 g for column 6.3.1.1 c)] of the stationary phase in a quantity of solvent such that the 75 g [or 80 g for column 6.3.1.1 c)] of support are covered entirely by the solution.
- Cover the solid support with the solution and stir the mixture with a clean glass rod until excess solvent has evaporated or been absorbed. Transfer the mixture to a rotary evaporator and remove the remaining solvent so that the packing becomes dry and free-running.
- Screen the support thus prepared gently, and preserve the 180  $\mu\text{m}$  to 250  $\mu\text{m}$  fraction.

### 6.3.2.3 Method of packing

Use a method of packing that allows reproducible columns to be prepared. The flow of packing into the column may be assisted by applying a vacuum to the column outlet, and regular packing ensured by tapping or by applying gentle vibration to the column.

### 6.3.2.4 Column conditioning

The column should be maintained at a temperature of 40 °C for 5 h with the carrier gas flowing but with the detector disconnected. The column outlet should be disconnected from the detector.

### 6.3.3 Resolution in the recommended experimental conditions

The following resolution should be obtained between propane and propene in commercial propane and between propene and isobutane in commercial butane (see figure 1):

$$R_{AB} = 2 \frac{[d'_{R(B)} - d'_{R(A)}]}{w_A + w_B} \geq 1,5$$

where

A and B are the components propane and propene or propene and isobutane respectively;

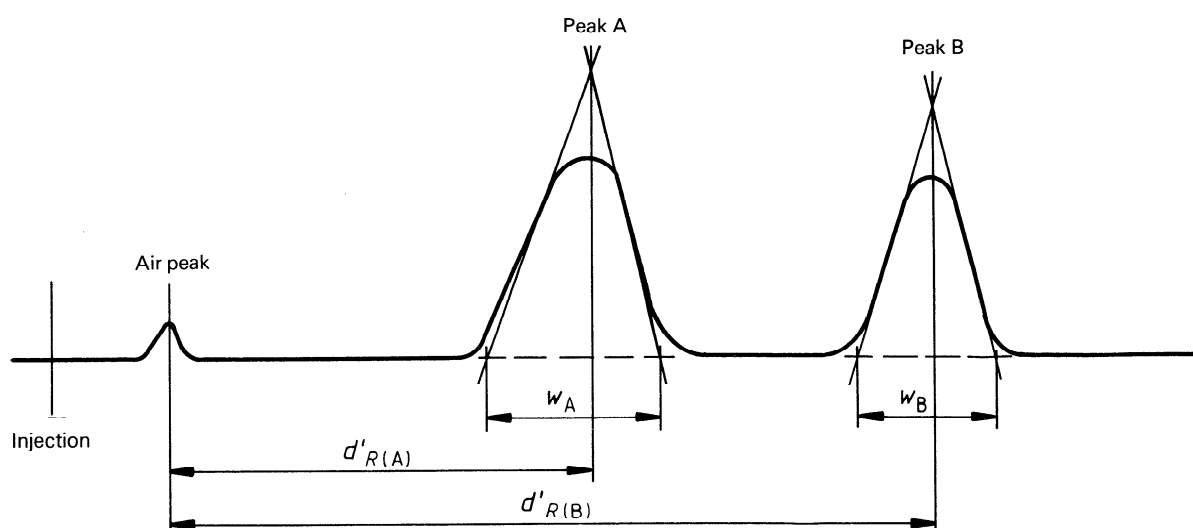
$R_{AB}$  is the resolution for the two peaks A and B;

$d'_{R(A)}$  and  $d'_{R(B)}$  are the adjusted retention times for components A and B respectively, the times being expressed as chart distances in millimetres;

$w_A$  and  $w_B$  are the peak widths of components A and B respectively.

### 6.4 Detector

The detector may be a thermal conductivity type (hot-wire type or thermistor type) or a flame ionization type. The system should be capable of detecting 0,1 % concentration of any



NOTE — The diagram shows an air peak, but this would not be seen with a flame ionization detector.

Figure 1 — Measurements for determination of resolution