



SLOVENSKI STANDARD
SIST EN ISO 13757:1998
01-maj-1998

Utekočinjeni naftni plini - Določevanje oljnih ostankov - Visokotemperaturna metoda (ISO 13757:1996)

Liquefied petroleum gases - Determination of oily residues - High-temperature method (ISO 13757:1996)

Flüssiggase - Bestimmung der öligen Rückstände - Hochtemperaturmethode (ISO 13757:1996)

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Gaz de pétrole liquéfiés - Détermination des résidus huileux - Méthode a haute température (ISO 13757:1996)

[SIST EN ISO 13757:1998](#)

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Ta slovenski standard je istoveten z: EN ISO 13757:1996

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75.160.30 Plinska goriva Gaseous fuels

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EUROPEAN STANDARD

EN ISO 13757

NORME EUROPÉENNE

EUROPÄISCHE NORM

July 1996

ICS 75.160.00

Descriptors: see ISO document

English version

Liquefied petroleum gases - Determination of oily
residues - High-temperature method
(ISO 13757:1996)

Gaz de pétrole liquéfiés - Détermination des
résidus huileux - Méthode à haute température
(ISO 13757:1996)

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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

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EN ISO 13757:1996

Foreword

The text of the International Standard ISO 13757:1996 has been prepared by Technical Committee ISO/TC 28 "Petroleum products and lubricants" in collaboration with 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of an identical text or by endorsement, at the latest by January 1997, and conflicting national standards shall be withdrawn at the latest by January 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of 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 and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 13757:1996 was approved by CEN as a European Standard without any modification.

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INTERNATIONAL
STANDARD

ISO
13757

First edition
1996-07-15

**Liquefied petroleum gases —
Determination of oily residues —
High-temperature method**

iTeh STANDARD PREVIEW

(standards.iteh.ai)

*Gaz de pétrole liquéfiés — Détermination des résidus huileux — Méthode
à haute température*

[SIST EN ISO 13757:1998](#)

<https://standards.iteh.ai/catalog/standards/sist/3c59780a-e831-477b-bc51-5a58eca44ea3/sist-en-iso-13757-1998>



Reference number
ISO 13757:1996(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 voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 13757 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

Annex A of this International Standard is for information only.

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Liquefied petroleum gases — Determination of oily residues — High-temperature method

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International 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.

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1 Scope

This International Standard specifies a method for the determination of the residual matter in liquefied petroleum gases (LPG) that remains after evaporation at 105 °C. This material, termed "oily residues", represents those products that are deposited in vaporizers that are subject to a heat input greater than that of ambient evaporation.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

ISO 4257:1988, *Liquefied petroleum gases — Method of sampling*.

3 Definition

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For the purposes of this International Standard, the following definition applies.

3.1 liquefied petroleum gas: Hydrocarbon gas that can be stored and/or handled in the liquid phase under moderate conditions of pressure and at ambient temperature. It consists essentially of C₃ and C₄ alkanes or alkenes, or a mixture of these, contains generally less than 5 % by liquid volume of material of higher carbon number, and has a gauge vapour pressure not exceeding approximately 1 600 kPa at 40 °C.

4 Principle

A known mass of filtered LPG sample is concentrated by evaporation. The concentrate is transferred into an evaporation flask using dichloromethane (methylene chloride) and then evaporated by means of a rotary evaporator. The oily residue remaining after this evaporation is placed in an oven at 105 °C for 1 h, then cooled and weighed.

5 Reagents

5.1 Acetone, analytical grade.

5.2 Ammonium peroxodisulfate, $(\text{NH}_4)_2\text{S}_2\text{O}_8$, in sulfuric acid solution.

Prepare the necessary volume of solution by dissolving the appropriate quantity (8 g/dm^3) of ammonium peroxodisulfate in concentrated sulfuric acid (98 % minimum purity).

CAUTION — Sulfuric acid and ammonium peroxodisulfate are strong oxidizing agents and therefore contact with organic materials should be avoided. Wear goggles, gloves and safety apron, all made from appropriate materials, during all handling operations.

5.3 Solid carbon dioxide, for the cooling bath.

5.4 Dichloromethane, analytical grade, containing less than 4 mg/kg oily residue after evaporation at 105°C .

NOTE 1 If the use of dichloromethane poses problems in terms of health and any regulations, it may be possible to replace it by pentane of the same quality (containing less than 4 mg/kg of residues at 105°C). In this case the procedure is unchanged.

CAUTION — Dichloromethane is an irritant and poses risks to health if handled incorrectly. Extract the vapours while working under a fume cupboard.

5.5 Laboratory detergent, see annex A, reference [6].

5.6 Water, conforming to grade 3 of ISO 3696.

6 Apparatus

6.1 Sample cylinder, stainless steel, fitted with two stainless steel valves, the upper one fitted with a dip tube, maximum service pressure 3 MPa, minimum capacity $3,75 \text{ dm}^3$, containing the LPG whose oily residues are to be determined.

6.2 In-line filter support, stainless steel, for high pressure (see annex A, reference [1]).

6.3 Filter discs, plain white membrane, diameter 47 mm, made of a blend of cellulose esters, nominal pore dimension $0,8 \mu\text{m}$ (see annex A, reference [2]).

6.4 Cooling coil, stainless steel, made by coiling 5 m of tube of external diameter 3 mm and internal diameter 2 mm onto a mandrel of diameter approximately 50 mm and fitted with the necessary connections (see annex A, reference [3]).

6.5 Cooling bath, comprising a 3-dm^3 Dewar flask, three-quarters filled with an appropriate liquid, e.g. acetone cooled with solid carbon dioxide, to achieve a temperature below -60°C .

To obtain quickly a cooling bath at the correct temperature, introduce approximately 1 kg of solid carbon dioxide into a 3-dm^3 beaker and slowly pour in acetone. Transfer the cooled acetone into the Dewar flask containing approximately 200 g of solid carbon dioxide. Repeat the operation until the desired volume is obtained.

NOTE 2 Such a bath can maintain a temperature below -60°C for several hours.

6.6 Thermometer, capable of measuring a temperature of $-60^\circ\text{C} \pm 1^\circ\text{C}$.

6.7 Sampling lines, stainless steel, conforming to the specification given in 6.4. The capacity of the sampling unit (filter, cooling coil, lines) shall be less than 25 cm^3 .

6.8 Needle valves

6.8.1 One stainless steel spindle-needle valve with 3-mm fittings, permitting regulation of the flow (equipment of chromatographic quality) (see annex A, reference [4]).

6.8.2 One stainless steel V-needle valve with 3-mm fittings (see annex A, reference [4]).

6.9 Beaker, glass, graduated, of capacity 5 dm^3 , shallow form.

6.10 Water bath, comprising a metal tank measuring approximately $300 \text{ mm} \times 300 \text{ mm} \times 300 \text{ mm}$, with water inlet and outlet, capable of being maintained at a temperature of $15^\circ\text{C} \pm 5^\circ\text{C}$; or a **steam bath** fitted with an orifice of diameter between 58 mm and 62 mm.

6.11 Rotary evaporator, any model enabling the evaporation of at least 200 cm^3 of solvent, over a bath of softened water regulated at $65^\circ\text{C} \pm 5^\circ\text{C}$, from a ground-necked evaporation flask.

NOTE 3 A double-coil water condenser may suffice for apparatus located in a fume cupboard; otherwise, a solid carbon dioxide/acetone coolant is necessary (see annex A, reference [5]).

6.12 Evaporation flask, 200-cm³ glass round-bottom flask with a ground neck and fitted with a glass stopper, tare less than 130 g (including stopper).

6.13 Oven, capable of maintaining a temperature of 105 °C ± 5 °C.

6.14 Balances

6.14.1 For the sample cylinder, balance with a range of 10 kg minimum, accurate to within ± 1 g or better.

6.14.2 For the evaporation flask, balance with a range of 160 g minimum, accurate to within ± 0,1 mg or better.

6.15 Laboratory gloves, of fine PVC, resistant to dichloromethane and talc-free.

6.16 Desiccator, without desiccating agent.

7 Preparation of equipment

7.1 New glassware

Clean new glassware by immersing it in detergent (5.5) for a minimum of 12 h before use. In case of dispute or when abnormal results have been obtained, immerse the glassware in a solution of ammonium peroxodisulfate/sulfuric acid (5.2) for 12 h.

After cleaning with detergent or ammonium peroxodisulfate/sulfuric acid solution, remove the glassware and rinse it in tap water and then in grade 3 water (5.6), using gloves (6.15) or tongs. Continue to wear gloves when handling the glassware throughout the remainder of the test procedure. Dry the glassware in the oven (6.13) for 30 min. Leave to cool in a desiccator (6.16) for at least 2 h before use (take care that the stopper does not become stuck during cooling).

After cleaning the apparatus, carry out the tare by weighing the evaporation flask (with stopper) to the nearest 0,1 mg after having unstoppered it for 30 s.

7.2 Previously used glassware

Remove any traces of organic products from used glassware by washing it carefully with dichloromethane (5.4) using gloves. Rinse with acetone (5.1) and dry in the oven (6.13) for 30 min. Continue to wear gloves when handling the glassware throughout the remainder of the test procedure. Carefully remove the solvent vapours from the evaporation flask, either

by inverting the flask for approximately 10 s or by blowing nitrogen into it. Place the flask, fitted with its stopper, and the rest of the glassware in a desiccator (6.16) for at least 2 h before use (take care that the stopper does not become stuck during cooling).

If the glassware is not clean enough, or in case of dispute or when abnormal results have been obtained, clean the glassware as described in 7.1, using gloves, having first removed any organic products by rinsing with dichloromethane (5.4) followed by acetone (5.1).

After cleaning the apparatus, carry out the tare by weighing the evaporation flask (with stopper) to the nearest 0,1 mg after having unstoppered it for 30 s.

7.3 Sampling line

The sampling line shall be rinsed first with dichloromethane (5.4), and then with the LPG to be tested.

8 Test procedure

CAUTION — It is essential to work under an explosion-proof hood or in the open, taking all the necessary safety measures, in particular the need to earth equipment to eliminate the risks associated with static electricity.

8.1 Sampling procedure

Slowly shake the sample cylinder containing the LPG to be analysed in order to mix the contents, and then remove a mass of 800 g to 1 200 g of product, weighed to within ± 1 g, into a 5-dm³ shallow-form beaker in accordance with the procedure defined in 8.1.1 and 8.1.2.

8.1.1 Assemble the apparatus, as shown in figure 1, by connecting the lower valve of the sample cylinder to the sampling line comprising:

- the V-needle valve (3);
- the in-line filter (4);
- the cooling coil (6) immersed in the bath (5), the temperature of which (below – 60 °C) has been checked with a thermometer;
- the spindle-needle valve (7);
- a nozzle in the form of a flexible polytetrafluoroethylene (PTFE) tube (8) or, if not available, a stainless steel tube, enabling flow into the beaker (9).