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भारतीय मानक मसौदा

20 प्रतिशत डाइमिथाइल ईथर (डीएमई) मिश्रित तरलीकृत पेट्रोलियम गैस (एलपीजी) - विशिष्ट

Draft Indian Standard

**20 PERCENT DIMETHYL ETHER (DME) BLENDED LIQUEFIED PETROLEUM GAS (LPG) —
SPECIFICATION**

(ICS 75.160.30;71.080.60)

Petroleum and their Related Products of Synthesis
or Biological Origin Sectional Committee, PCD 03

Last date for receipt of
comment is 23 September 2023

FOREWORD

(Formal clause to be added later)

Liquefied Petroleum Gas (LPG) is being used in India to cater to the energy needs of domestic, commercial and industrial sectors apart from use as an automotive fuel. The consumption of LPG in the country is expected to rise. This necessitates the use of alternate fuels to partially substitute LPG with fuel such as synthetically produced dimethyl ether (DME).

DME is a viable and clean alternative which can be blended with LPG. It is the simplest ether with oxygen connecting two methyl groups having no C-C bond. DME can be stored, transported and used in the same manner as LPG. DME can be blended with LPG upto 20 percent by weight and the blended fuel can be used for cooking in households and other applications.

Reference to various other overseas standards like ISO, ASTM, IP, to name a few, has been given as presently there is no Indian Standard available for them. Once the Indian Standards are formulated, the references will be modified accordingly.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1 SCOPE

1.1 This draft standard prescribes the requirements and methods of sampling and test for 20 percent Dimethyl Ether (DME) blended Liquefied Petroleum Gas (LPG) marked for household, commercial and industrial applications excluding automotive use in the country.

1.2 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 REFERENCES

The following Standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to

agreements based on this standard are advised to use the latest editions of the standards indicated below:

<i>IS No./International Standards</i>	<i>Title</i>
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods Part 1 Dangerous good (<i>first revision</i>)
IS 1447 (Part 2): 2013 / ISO 4257 : 2001	Methods of sampling of petroleum and its products Part 2 Sampling of liquefied petroleum gases (LPG) (<i>second revision</i>)
IS 1448	Methods of Test For Petroleum And Its Products
(Part 70) : 2018	Determination of residue in Liquefied Petroleum Gases (<i>first revision</i>)
(Part 71): 2004 / ISO 4256:1996	Liquefied Petroleum Gases - Determination of Gauge Vapour Pressure - LPG Method (<i>second revision</i>)
(Part 72) : 2023	Volatility of liquefied petroleum gases (<i>first revision</i>)
(Part 73): 2004 / ISO 8819 : 1993	Liquifiable Petroleum gases - Detection of hydrogen sulphide - Lead acetate method (<i>first revision</i>)
(Part 76) : 2019 / ISO 3993 : 1984	Liquified Petroleum Gases and Light Hydrocarbons — Determination of Density or Relative Density — Pressure Hydrometer Method (<i>first revision</i>)
(Part 151) : 2004/ ISO 7941 : 1998	Commercial propane and butane - analysis by gas chromatography
(Part 152) : 2004/ ISO 6251 : 1996	Liquefied petroleum gases — Corrosiveness to copper — Copper strips test
IS 2825 : 1969	Code for unfired pressure vessels
IS 3196 (Part 1) : 2013	Welded Low Carbon Steel Cylinders Exceeding 5 Litres Water Capacity for Low Pressure Liquefiable Gases — Part 1 Cylinders for liquefied Petroleum Gases (LPG) (<i>sixth revision</i>)
IS 4576 : 2021	Liquefied Petroleum Gases — Specification (<i>fourth revision</i>)
IS 4639 (Part 1) : 2000 / ISO 1998-1 : 1998	Petroleum industry - Terminology: Part 1 Raw materials and products (<i>first revision</i>)
IS 16704: 2018 / ISO 16861 : 2015	Petroleum Products – Fuels (Class F) — Specifications of Dimethyl Ether (DME)
ISO 8973 : 1997	Liquefied petroleum gases -- Calculation method for density and vapour pressure
EN 15470 : 2017	Liquefied petroleum gases. Determination of dissolved residues. High temperature gas chromatographic method
EN 15471: 2017	Liquefied petroleum gases. Determination of dissolved residues. High-temperature gravimetric method
ASTM D1267 : 2023	Standard Test Method for Gauge Vapor Pressure of Liquefied Petroleum (LP) Gases (LP-Gas Method)
ASTM D1657 : 2022	Standard Test Method for Density or Relative Density of Light Hydrocarbons by Pressure Hydrometer
ASTM D1838 : 2021	Standard Test Method for Copper Strip Corrosion by Liquefied Petroleum (LP) Gases
ASTM D2158 : 2021	Standard Test Method for Residues in Liquefied Petroleum (LP) Gases
ASTM D2420 : 2023	Standard Test Method for Hydrogen Sulfide in Liquefied Petroleum (LP) Gases (Lead Acetate Method)
ASTM D2421 :2021	Standard Practice for Interconversion of Analysis of C5 and Lighter Hydrocarbons to Gas-Volume, Liquid-Volume, or Mass Basis

ASTM D 2598 : 2021	Standard Practice for Calculation of Certain Physical Properties of Liquefied Petroleum (LP) Gases from Compositional Analysis
ASTM D 3246 : 2015	Standard Test Method for Sulfur in Petroleum Gas by Oxidative Microcoulometry
ASTM D 5305 :2023	Standard Test Method for Determination of Ethyl Mercaptan in LP-Gas Vapor
ASTM D 6667 : 2021	Standard Test Method for Determination of Total Volatile Sulfur in Gaseous Hydrocarbons and Liquefied Petroleum Gases by Ultraviolet Fluorescence
ASTM D 6897 : 2016	Standard Test Method for Vapor Pressure of Liquefied Petroleum Gases (LPG) (Expansion Method)
IP 432 : 2000	Liquefied petroleum gases- calculation method for density and vapour pressure

3 TERMINOLOGY

For the purpose of this standard, the following definition shall apply.

3.1 Liquefied Petroleum Gas (LP Gas or LPG) — See IS 4639 (Part 1).

3.1.1 LP gases mainly consist of one or more of the following hydrocarbons:

- a) Propane (C₃H₈)
- b) Propylene (C₃H₆)
- c) n-butane (C₄H₁₀)
- d) Iso-butane (C₄H₁₀)
- e) Butylene (C₄H₈)

3.1.2 Small quantities of one or more of the following hydrocarbons may also be present:

- a) Ethane (C₂H₆)
- b) Ethylene (C₂H₄)
- c) Pentane (C₅H₁₂)
- d) Pentene (C₅H₁₀)

3.2 Di Methyl Ether (DME) — Colourless gas, synthetically produced from biomass or natural gas, capable of being used as alternate fuel

4 REQUIREMENTS

4.1 LPG shall meet the requirements as per IS 4576.

4.2 DME shall meet the requirements as per IS 16704.

4.3 20 percent DME blended LPG shall comply with the requirements given in Table 1 when tested according to appropriate methods given in col. 4 of Table 1.

4.4 Residue of LPG

Subject to agreement between the purchaser and the supplier, the material shall also pass the agreed limits for residue when tested according to IS 1448 (Part 70).

Table 1 Requirements for 20 percent DME blended LPG

(Clause 4.3)

Sl No.	Characteristics	Requirements	Method of Test, Ref to Part of IS 1448 /ISO/ASTM/Annex
(1)	(2)	(3)	(4)
(i)	Density ¹⁾ at 15°C, kg/m ³	Report	(Part 76) ⁹⁾ /D1657
(ii)	Vapour pressure ²⁾ at 40°C, kPa, gauge, <i>Max</i>	1050 ³⁾	(Part 71) ⁹⁾ /ISO 8973/D1267/D6897
(iii)	Composition, mass fraction of DME, percent, w/w, <i>Max</i>	20	Annex A
(iv)	Other Hydrocarbons, liquid volume percent ⁴⁾		
	a) C ₂ hydrocarbon	Report	
	b) C ₃ hydrocarbon	Report	(Part 151)
	c) C ₄ hydrocarbon	Report	
	d) C ₅ hydrocarbon and heavier, <i>Max</i>	1.0	
	OR		
	Volatility: Evaporation temperature in °C for 95 percent by volume at 760 mm Hg Pressure, <i>Max</i>	0.05	(Part 72) ⁹⁾ /D2158/EN 15470/EN 15471
(v)	Total volatile Sulphur ⁵⁾ , mg/kg (ppmw), <i>Max</i> ,	140	D3246/D6667 ⁹⁾
(vi)	Copper strip corrosion at 38°C for 1 hour	Not worse than No 1	(Part 152) ⁹⁾ /D 1838
(vii)	Hydrogen sulphide ⁶⁾	Pass	(Part 73) ⁹⁾ / D2420
(viii)	Free water content ^{7,8)} , ppm, <i>Max</i>	None	(Part 76) ⁹⁾ /D1657 (visual inspection using these equipment)
(ix)	Caustic test	Pass	See Note 10
(x)	Ethyl mercaptan ¹¹⁾ , ppm, <i>Min</i>	20	D5305

NOTES

- Density/Relative Density may also be determined by analyzing the gas by Gas Chromatograph and using composition and Density factor data as per ISO 8973/ASTM D2598/IP 432.
- Vapour pressure may be determined at any other temperature and converted to 40 °C / 65 °C by means of suitable vapour pressure-temperature graph. The same can also be determined by analyzing the gas by Gas Chromatograph and using composition and vapour pressure data as per ISO 8973/ASTM D2598/IP 432.
- Each consignment of the product shall be designated by its maximum vapor pressure in kPa at 40 °C and shall not exceed 16.87 kg/sq.cm at 65 °C for transportation through road tank trucks. Further, if purchaser and the supplier agreed, the minimum vapor pressure of that mixture shall be not lower than 200 kPa gauge compared to the designated maximum vapor pressures and in any case the minimum for the mixture shall be not lower than 520 kPa at 40°C.
- Molar composition of LPG determined by Gas Chromatographic analysis can be converted into Liquid-Volume using the Standard Practice as per ASTD D2421.
- Total sulphur limits in these specifications do include sulphur compounds used for stanching purposes.
- 'Pass' the test indicates Hydrogen Sulphide not more than 5 ppm.
Hydrogen Sulphide can also be determined with the following procedure:
Take 5 ml of lead acetate solution (prepared in IPA) + 25 ml LPG (liquid) in measuring cylinder and shake well. If black precipitation is not seen then H₂S test Passes.
- Free water: Water promotes rust on internal surface of steel storage tanks and iron piping systems. Water can also cause odourant to fade and can block small openings. LPG shall not contain any free water at 0 °C and shall be determined by visual inspection of sample using equipment on which density is determined. Subject to agreement between purchaser and supplier, limit, temperature and method for

water content determination may be changed for specific applications like shipment of refrigerated LPG etc.

8. Free water content can also be determined with the following procedure:

Take 100 ml of liquid in dry graduated tube and allow to vaporize the LPG. When about 2 ml to 3 ml residue is left, shake and pour the residue on whatman filter paper. Allow the residue on filter paper to evaporate; if wet spot not seen, free water is absent.

9. In case of dispute, this method shall be the referee method.

10. For caustic test, following procedure shall be adopted:

2 ml LPG (liq) + 5 ml demineralized water are mixed in a tube and a drop of phenolphthalein is added. Caustic test passes if it doesn't turn into pink.

11. Product shall contain minimum 20 ppm ethyl mercaptan at first dispatching location to ensure the detection of odour.

(a) To detect the odour, the following procedures may be adopted:

5 ml Doctor Solution + 8 ml iso-octane + pinch of sulphur powder in 25 ml stoppered cylinder. Shake and add 2 ml LPG (Aq). Shake slowly by releasing pressure.

Odour is adequate, if solution turns yellowish brown.

(b) Subject to agreement between the purchaser and the supplier, requirement of dosing ethyl mercaptan in LPG may be dispensed with for certain applications.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in suitable cylinders / containers as agreed to between the purchaser and the supplier and subject to the requirements prescribed by statutory authorities. (For cylinders, *see* IS 3196 (Part 1) and for comments, *see* IS 2825).

5.2 Marking

The cylinders/containers shall be marked as prescribed by statutory authorities from time to time. They shall bear the labels marked with the following information:

- a) Name and type of the material;
- b) Mass in kg of the material in the container;
- c) Maximum vapor pressure; and
- d) Manufacturer's name and trade mark, if any

5.2.1 Each cylinder /container shall also be marked with the caution label 'FLAMMABLE' together with the corresponding symbol for labeling dangerous goods [*see* Fig.18 of IS 1260 (Part 1)].

5.2.2 BIS Certification Marking

5.2.2.1 The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the BIS Act, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

6 SAMPLING

6.1 Representative samples of DME blended liquefied petroleum gas shall be drawn as prescribed in IS 1447 (Part 2).

6.2 Tests on vapor pressure shall be conducted on individual samples and the rest of the tests shall be conducted on composite samples.

ANNEX A

[Table 1, Sl No. (iii)]

DETERMINATION OF MASS FRACTION OF DIMETHYL ETHER (DME) IN DME BLENDED LPG

1.1 Outline of the method

DME-LPG blended sample is analyzed via gas sampling valves by gas chromatography and compared to corresponding components separated under identical operating conditions from pure DME reference standards. The chromatogram of the sample is interpreted by comparing peak retention times and areas with those obtained for the pure DME reference standard.

1.2 Apparatus

1.2.1 Gas Chromatograph (GC)

Any gas chromatographic instrument provided with a linear temperature programmable column oven. Multi-step column oven temperature programming is required, consisting of an initial hold time, an initial temperature program followed by an isothermal temperature hold and another programmed temperature rise. The temperature control must be capable of obtaining a retention time repeatability of 0.05 min throughout the scope of this analysis. A flame ionization detector (FID) having sensitivity of 0.5 percent (mole) or less for the DME compound is strongly recommended.

1.2.2 Data Acquisition

Any commercial integrator or computerized data acquisition system may be used for display of the chromatographic detector signal and peak area integration. The device should be capable of calibration and reporting of the final response corrected results.

1.2.3 Sample Introduction

For gas sampling, a six-port gas sampling valve (GSV) with a 250 µl fixed sampling loop may be provided. This valve shall be contained in a heated enclosure and operated at a temperature above the boiling point of the highest boiling component in the sample.

1.3 Gas Controls

The GC shall be provided with suitable facilities for delivery and control of carrier gas and the detector gases. This will consist of the appropriate tank and downstream regulators and supply tubing as well as the mass or pressure controls for the precise regulation of the instrument operation.

1.4 Columns

Condition all columns used according to the manufacturers' instructions prior to use. The recommended analytical column for this test method is 100 m length, internal diameter 0.25 mm and film thickness 0.5 µm of 100 percent

dimethylpolysiloxane capillary column.

2 REAGENTS AND MATERIALS

2.1 Carrier Gases

For carrier gases, it is recommended to install commercial active oxygen scrubbers and water dryers, such as molecular sieves, ahead of the instrument to protect the system's chromatographic columns. Follow manufacturer's instructions in the use of such gas purifiers and replace as necessary.

2.1.1 Helium — 99.995 percent minimum purity, <0.1 ppm H₂O. The use of appropriate scrubbers may be sufficient to obtain the desired purity.

2.2 Detector Gases

2.2.1 Hydrogen — 99.995 percent minimum purity. The use of appropriate scrubbers may be sufficient to obtain the desired purity.

NOTE — Hydrogen is a flammable gas under high pressure.

2.2.2 Nitrogen — 99.995 percent minimum purity. The use of appropriate scrubbers may be sufficient to obtain the desired purity.

2.2.3 Air — less than 10 ppm each of total hydrocarbons and water. The use of appropriate scrubbers may be sufficient to obtain the desired purity.

NOTE — Improper handling of compressed gas cylinders containing air, nitrogen, hydrogen, or helium can result in explosion. Rapid release of nitrogen or helium can result in asphyxiation.

2.3 Reference Standards

2.3.1 Purity of Reagents

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

2.3.2 Reference DME standard

Analytical grade DME reference standard commercially available and may be used to establish quantitative determination of DME content in DME-LPG blended fuel.

3 PREPARATION OF APPARATUS

3.1 Set up the instrumentation in accordance with the manufacturer's instructions or as specified herein.

3.2 Install and condition the column according to manufacturer's instructions.

3.3 Set the GC instrument to the operating parameters. Allow the instrument to stabilize before proceeding with calibration and sample injections. Typical operating conditions for 100 percent dimethylpolysiloxane column are provided in Table 2.

Table 2 Typical Operating Condition
(Clause 3.3)

Column	100 m × 0.25 mm × 0.5 µm of 100 percent Dimethyl polysiloxane
Carrier Gas	Helium
Carrier Gas flow	2.0 ml/min
Injector temperature	250 °C
Injection type	Split with split ratio 150:1
Purge flow	3.0 ml/min
Injection volume	250 µl
Oven program	Initial temperature 35 °C for 10 min; First Ramp at 2.5 °C/min to 120 °C hold for 0 min Second Ramp at 15 °C/min to 220 °C hold for 5 min
Detector	FID Temperature: 250 °C Hydrogen flow: 40 ml/min Air flow: 400 ml/minute Make-up gas: Nitrogen Make-up gas flow: 30 ml/min
Analysis time	55.6min

3.4 Obtain duplicate chromatograms of the standard or sample, or both. Ensure that none of the peaks obtained have exceeded the upper range limit of the data handling device (at full scale on the data handling device, all peaks are on scale and display symmetrical, Gaussian shapes as opposed to flat peak tops). Use the same sample size (split ratio) and range for all runs. Example chromatograms are provided in Fig. 1.

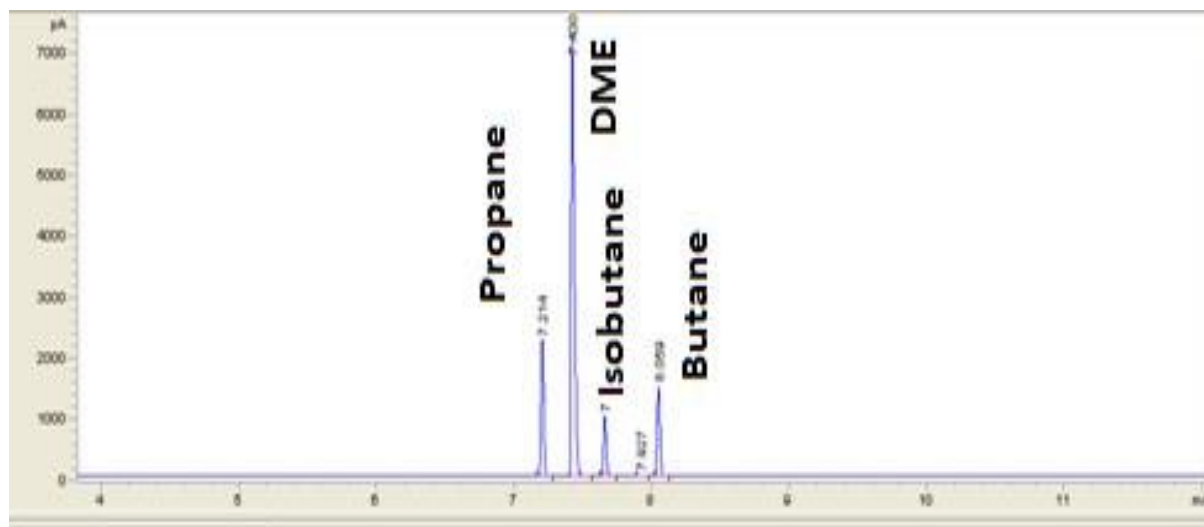


FIG. 1. — EXAMPLE CHROMATOGRAM USING THE DIMETHYLPOLYSILOXANE COLUMN

3.5 Gas Sampling Valve

Set valve on and off times to comply with manufacturer's instructions

4 CALIBRATION AND STANDARDIZATION

4.1 Qualitative

Determine the retention time of DME by analyzing known reference standard in the same manner as the samples. Typical retention time of DME is 7.48 min.

4.2 Quantitative

Determine the quantity of DME in DME-LPG blended fuel is interpreted by comparing peak areas with those obtained for the pure DME reference standard.

5 PROCEDURE

5.1 Sampling

Sampling at the sample source and at the chromatograph shall always be done in a manner that ensures that a representative sample is being analyzed. Lack of precision and accuracy in using this test method can most often be attributed to improper sampling procedures.

5.2 Gas Sample Valve Injection

Flush a gas sample loop with 5 ml to 10 ml of standard and sample (approximate 45s time), close cylinder valve, and allow the sample pressure to equilibrate to atmospheric pressure (stopped flow) before introducing the sample into the carrier gas stream.

5.3 Sample Analysis Procedure

5.3.1 Adjust the instrument operating variables to the values specified in Table 2.

5.3.2 Equilibrate the chromatographic system and inject the air blank until a representative chromatogram is obtained.

5.3.3 Inject an appropriate size DME reference standard (as determined in Table 2) into the injection port and start the analysis. Obtain a chromatogram and a peak integration report. Repeat the same standard for six times consecutively.

5.3.4 Inject a minimum of one air blank to check for carryover after six DME reference standards.

5.3.5 Inject an appropriate size sample (as determined in Table 2) into the injection port and start the analysis. Obtain a chromatogram and a peak integration report. Repeat the sample in duplicate.

5.3.6 Inject DME standard throughout the analysis, with a minimum of one injection at the end of sequence. One injection after every sixth sample analysis is recommended.

5.3.7 Record the peak area response for each analyte of interest, if present. Note all observed peaks in the blank injection and exclude these as artifacts from any calculation.

6 SYSTEM SUITABILITY

6.1 The percent RSD for peak area response of DME from the first six injections of the DME standard should be ≤ 10 percent.

6.2 The percent RSD for peak area response of DME from all injections of the DME standard throughout the analysis should be ≤ 10 percent.

7 CALCULATION

7.1 Identify DME peak by matching retention times with those for known reference standards. Obtain the area for

DME peak.

$$\text{DME content (percent } m/m) = (A_{\text{sample}}/A_{\text{standard}}) \times \text{purity of standard}$$

where

A_{sample} = Peak area of DME in sample

A_{standard} = Mean peak area of DME in first six standard injections

8 REPORT

8.1 Report the concentration of DME component as percent (m/m), to the nearest 0.1 percent(m/m).

9 PRECISION

9.1 Repeatability

The difference in two test results obtained by the same operator with the same apparatus in a given laboratory under constant operating conditions on test samples taken from the same laboratory sample should, in the long run in the normal and correct operation of the test method not exceed the values given in Table 3.

Table 3 Repeatability values of the method obtained with the 20 percent DME – 80 percent LPG blended fuel
(Clause 9.1)

Sl. No. (1)	20 percent DME + 80 percent LPG blended fuel(percent m/m) (2)	Calculated DME content (percent m/m) (3)	Percent RSD (4)
i)	18.5 % DME standard	18.1	3.6

NOTE — Calculated average value of 6 replications