

JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS K 2536-4 : 2011

(PAJ)

Liquid petroleum products—
Testing method of components—
Part 4: Determination of components
by tandem type gas chromatography

ICS 75.080

Reference number : JIS K 2536-4 : 2011 (E)

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Date of Establishment: 2003-06-20

Date of Revision: 2011-03-22

Date of Public Notice in Official Gazette: 2011-03-22

Investigated by: Japanese Industrial Standards Committee
Standards Board
Technical Committee on Chemical Products

JIS K 2536-4:2011, First English edition published in 2012-01

Translated and published by: Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN

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Printed in Japan

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry, through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by Petroleum Association of Japan (PAJ) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS K 2536-4:2003** is replaced with this Standard.

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Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public or utility model right which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public or utility model right which have the said technical properties.

JIS K 2536 consists of the following 6 parts under the general title “*Liquid petroleum products—Testing method of components*”:

Part 1: Fluorescent indicator adsorption method

Part 2: Determination of total components by gas chromatography

Part 3: Determination of aromatic components by gas chromatography

Part 4: Determination of components by tandem type gas chromatography

Part 5: Determination of oxygenate compounds by gas chromatography

Part 6: Determination of oxygen content and oxygenate compounds by gas chromatography and oxygen selective detection

Liquid petroleum products— Testing method of components— Part 4: Determination of components by tandem type gas chromatography

Introduction

This Japanese Industrial Standard has been prepared based on **JIS K 2536** published in 1960, and has gone through 7 revisions. This Standard was established as **JIS K 2536-4** by dividing **JIS K 2536** in 2003. It is revised in order to correspond to change of the ingredient of subsequent petroleum products this time.

Further, no International Standard corresponding to this Standard has been established at this point.

1 Scope

This Standard specifies the method of determination of the components [benzene, toluene, xylene, methanol, ethanol, methyl tert-butyl ether (hereafter referred to as “MTBE”) and ethyl tert-butyl ether (hereafter referred to as “ETBE”) and kerosene content] in the liquid petroleum products of which the end point is not more than 250 °C by the atmospheric pressure distillation test method specified in **JIS K 2254** using the tandem type gas chromatography.

There are two types of tandem type gas chromatography, such as a double oven type and a single oven type. The applicable range by type and by object component shall be in accordance with table 1. It may be used for the determination of the oxygen compounds other than the alcohols and ethers shown in table 1, however, the components are determined after separating and identifying.

When a doubt arises about the test results obtained in this Standard, the test shall be carried out according to **JIS K 2536-2**.

NOTE : The test methods specified in series of **JIS K 2536** are shown in Annex A.

Warning : The use of this Standard may involve hazardous reagents, 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 test.

Table 1 Applicable range of concentration by component

Unit: volume fraction (%)

Component	Double oven type gas chromatography	Single oven type gas chromatography ^{a)}
Benzene	0.5 to 10	0.1 to 10
Toluene	1.0 to 30	0.1 to 30
Xylene	1.0 to 30	0.1 to 30
Methanol	0.5 to 20	0.1 to 20
Ethanol	—	0.1 to 15
MTBE	0.5 to 20	0.1 to 20
ETBE	—	0.1 to 15
Kerosene content	1.0 to 20	0.7 to 20
Note ^{a)} The single oven type is called a dual type gas chromatography in some cases.		

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS K 0114 *General rules for gas chromatographic analysis*

JIS K 0512 *Hydrogen*

JIS K 2249 *Crude petroleum and petroleum products—Determination of density and petroleum measurement tables based on a reference temperature (15 centigrade degrees)*

JIS K 2251 *Crude petroleum and petroleum products—Sampling*

JIS K 2254 *Petroleum products—Determination of distillation characteristics*

JIS K 2536-2 *Liquid petroleum products—Testing method of components Part 2: Determination of total components by gas chromatography*

JIS K 8101 *Ethanol (99.5) (Reagent)*

JIS K 8680 *Toluene (Reagent)*

JIS K 8839 *2-Propanol (Reagent)*

JIS K 8858 *Benzene (Reagent)*

JIS K 8891 *Methanol (Reagent)*

JIS K 9701 *Heptane (Reagent)*

JIS Q 0034 *General requirements for the competence of reference material producer*

JIS Q 0035 *Reference materials—General and statistical principles for certification*

JIS Z 8401 *Guide to the rounding of numbers*

JIS Z 8402-6 *Accuracy (trueness and precision) of measurement methods and results—Part 6: Use in practice of accuracy values*

3 Terms and definitions

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

3.1 kerosene content

calculated by multiplying the total amount [volume fraction (%)] of *n*-tridecane and *n*-tetradecane by coefficient, 14

3.2 xylene

a generic term for the components of ethylbenzene, *o*-xylene, *m*-xylene and *p*-xylene

In the case of concentration, it means the total amount of each component.

4 Determination method for component by double oven type gas chromatography

4.1 Principle of test

The sample injected to gas chromatography is branched to two columns with different polarity and benzene, toluene, xylene, methanol, MTBE, and *n*-tridecane and *n*-tetradecane as the index of kerosene in the sample are separated to elute and identified by one injection of the sample. Each component is eluted in both columns, and the different gas chromatograms are obtained depending on the difference in polarity of each component. The content of each component is determined so that the gas chromatogram, in which the base material of gasoline and the object component are not overlapped, is selected and the working curve prepared beforehand is used.

4.2 Reagents

- a) **Benzene**, specified in **JIS K 8858**.
- b) **Toluene**, specified in **JIS K 8680**.
- c) **Xylene**, the total of *o*-xylene, *m*-xylene, *p*-xylene and ethylbenzene is not less than 99 %.
- d) **Methanol**, specified in **JIS K 8891**.
- e) **MTBE**, not less than 99 % in purity.
- f) ***n*-tridecane**, not less than 99 % in purity.
- g) ***n*-tetradecane**, not less than 99 % in purity.
- h) ***n*-heptane**, specified in **JIS K 9701**.
- i) **Helium**, not less than 99.995 % in purity (volume fraction).

4.3 Test apparatus

The test apparatus shall be as follows.

4.3.1 Gas chromatography The gas chromatography is constructed so that the flow path, where the sample is injected into the column, is connected through the temperature controllable connecting part, two columns capable of controlling temperature

according to the programme are independently provided and a packed column and a capillary column are separately set. In addition, the gas chromatography in which the branch mechanism linked to the sample injector is included and the additive gas mechanism and the detector corresponding to each column are included, shall be used. The example of the constitution of double oven type gas chromatography is shown in figure 1.

The general matters common to gas chromatography shall be in accordance with **JIS K 0114**.

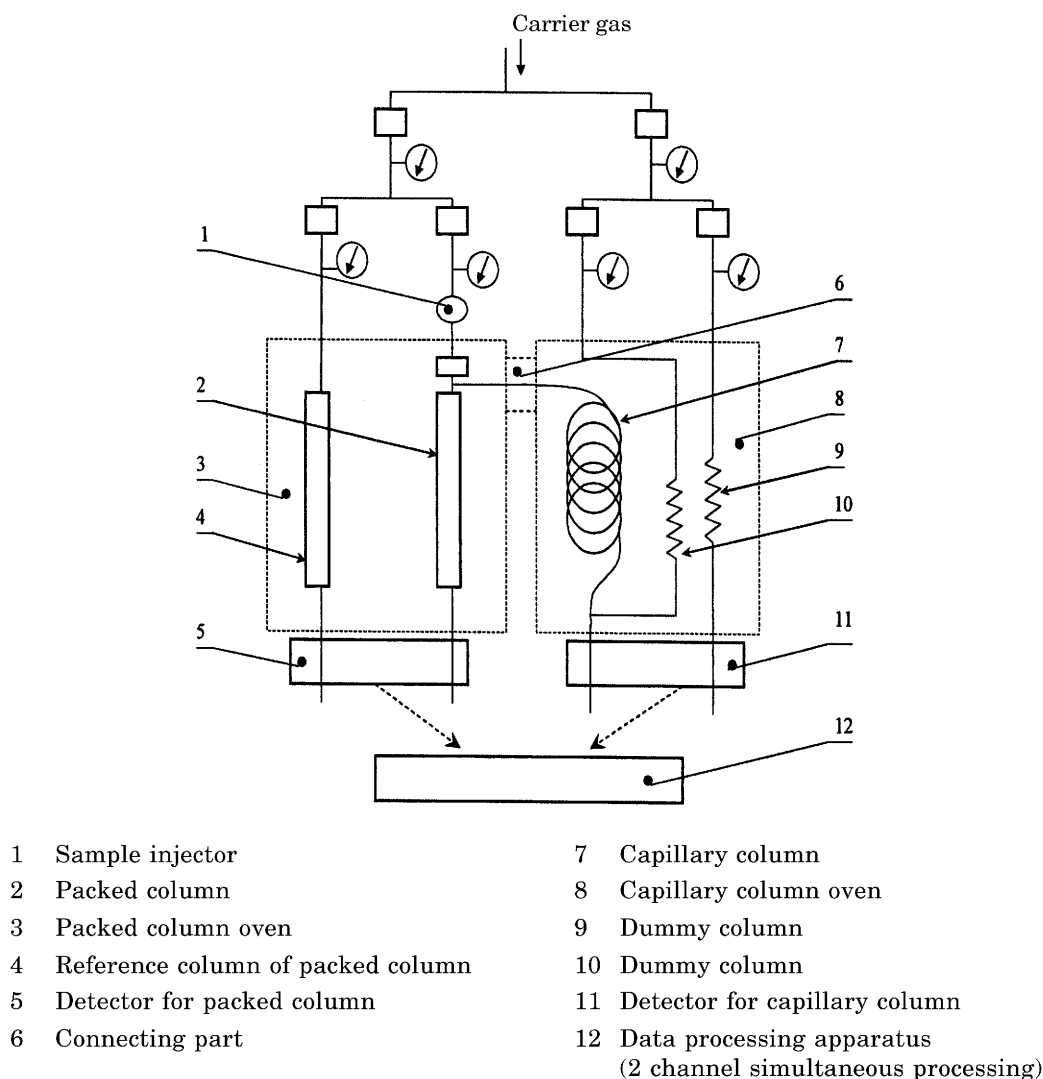


Figure 1 Example of constitution of double oven type gas chromatography

- a) **Sample injector** The sample injector is composed of an injecting opening and a split vent, provided with the mechanism capable of branching the injected sample and capable of maintaining at the test temperature of 200 °C to 250 °C.
- b) **Column oven** The column oven is constructed so as to be capable of controlling the multi-stage programmed temperature in the range of 40 °C to 250 °C.

- c) **Column** The column shall be the combination of the packed column and capillary column indicated as follows. The example of column is shown in table 2.
- 1) **Packed column**, prepared so that a stainless steel pipe is packed with the support of 180 μm to 250 μm or 150 μm to 180 μm in particle size of diatomaceous earth as a material on which the main material of TCEP [1,2,3-tris (2-cyanoethoxy) propane] as polar stationary liquid is added with polyhydric alcohol to elute methanol between benzene and toluene so as not to overlap with *n*-dodecane each other.
 - 2) **Capillary column**, made of molten silica, and the coated with methyl silicon.

Table 2 Example of column of double oven type gas chromatography

Item	Packed column	Capillary column
Stationary liquid	TCEP + polyhydric alcohol	Methyl silicon
Film thickness of liquid phase μm	—	0.5
Retention amount of stationary liquid		
TCEP %	25	—
Polyhydric alcohol %	1 to 2	—
Inside diameter mm	3	0.20 to 0.35
Length m	3 to 4	25 to 50

- d) **Detector container and detector** The detector container and the detector are independently used in packed column and capillary column respectively. The detector container is capable of maintaining at the column temperature or higher. For the detector, a thermal conductivity detector is used.
- e) **Data processing apparatus**, capable of processing simultaneously two channels so as to be capable of recording individual peak retention time and peak area value independently every detector as well as chromatogram, and also having a peak area processing mechanism by tangent method with 1 count per 1 $\mu\text{V}\cdot\text{s}$ or over of integral sensitivity. The separation treatment of overlapped peak is provided with the peak area processing mechanism by the tangent method or perpendicular method.

4.3.2 Automatic sample injector That which is capable of sampling and injecting the amount of sample required for test by using a microsyringe capable of weighing out 2 μl of sample. The precision of sample injection shall be not more than 2.5 % in the coefficient of variation for peak area when *n*-heptane solution containing 5 % in volume fraction of toluene is repeatedly measured 5 times under the same condition as that at the time of preparing the working curve.

4.4 Preparation of test

The preparation of test shall be as follows.

4.4.1 Adjustment of gas chromatography