

DOI: <https://doi.org/10.21009/JRSKT.092.11>

Evaluation Of Liquefied Petroleum Gas (LPG) Quality Conformity Based On Hydrocarbon Composition And Ethyl Mercaptan Content With Reference To The Directorate General Of Oil And Gas Standard

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Article Information

Received: 16/10/2023

Revised: 05/12/2023

Accepted: 11/12/2023

Online: 20/12/2023

Published: 30/12/2023

Abstract

Liquefied Petroleum Gas (LPG) is a major energy source in Indonesia's household, commercial, and industrial sectors; therefore, its quality must be controlled to ensure combustion performance and user safety. This study evaluated the hydrocarbon composition and ethyl mercaptan content of LPG samples and their conformity with the Decree of the Director General of Oil and Gas No. 116.K/10/DJM/2020. Five mixed-LPG samples were analysed at the Gas Composition Laboratory of BBPMGB LEMIGAS. Hydrocarbon composition (C₂–C₅) was determined by Gas Chromatography–Flame Ionization Detector (GC-FID) according to ASTM D2163, while ethyl mercaptan content was measured using a length-of-stain gas detector tube following ASTM D5305. The GC-FID system was verified using a Certified Reference Material (CRM), and all thirteen measured components were within the CRM acceptance ranges, confirming instrument suitability. Samples 1 and 2 complied with the C₂ limit (max 0.80 %vol), while Samples 3, 4, and 5 exceeded the limit with values of 0.9300, 0.8510, and 1.0281 %vol, respectively. All samples met the C₃+C₄ and C₅+ requirements. Ethyl mercaptan contents of Samples 1–4 (1.8–3.9 lb/10,000 AG) met the minimum requirement, whereas Sample 5 did not (0.96 lb/10,000 AG). Overall, only Samples 1 and 2 satisfied all tested parameters, indicating that LPG quality evaluation requires multiple quality parameters.

Keywords: Ethyl Mercaptan, Gas Chromatography-Flame Ionization Detector, Hydrocarbon Composition, Liquefied Petroleum Gas (LPG).

Introduction

Liquefied Petroleum Gas (LPG) is one of the most widely utilized gaseous fuels for household, commercial, and industrial in indonesia. The applications due to its high energy density, efficient combustion characteristics, and relatively convenient storage and distribution through pressurized

containers (Puzzolo et al., 2019). LPG mainly consists of light hydrocarbons, particularly propane (C_3H_8) and butane (C_4H_{10}), which can be liquefied under pressure, allowing efficient storage and distribution in pressurized cylinders (International Energy Agency, 2023). However, variations in physical and chemical characteristics during production, distribution, and utilization require strict quality control to maintain product performance and safety (Oke et al., 2020).

Hydrocarbon composition is an important quality parameter of LPG because it influences combustion characteristics, calorific value, and operational stability. Variations in the propane-to-butane ratio or the presence of hydrocarbon fractions outside the specification may affect LPG performance and combustion efficiency (Nofrizal et al., 2022). Previous research reported that LPG composition may vary throughout the distribution chain from refinery facilities to agents, emphasizing the importance of composition analysis to ensure compliance with applicable quality standards (Suryo et al., 2023).

In addition to hydrocarbon composition, odorant content is an essential safety parameter in LPG quality evaluation. LPG is naturally odourless; therefore, ethyl mercaptan (C_2H_5SH) is added as an odorant to enable early detection of leakage (Seguel et al., 2018). Insufficient ethyl mercaptan concentration may reduce leak detection capability, whereas excessive concentrations may cause undesirable environmental and health effects (Michanowicz et al., 2023). Therefore, monitoring ethyl mercaptan content is an important component of LPG quality control.

The quality specification of LPG marketed in Indonesia is regulated by the Decree of the Director General of Oil and Gas No. 116.K/10/DJM/2020. For mixed LPG, the specification includes hydrocarbon composition and odorant content parameters, including a maximum C_2 content of 0.80 %vol, a minimum C_3+C_4 content of 97.0 %vol, a maximum C_5+ content of 2.0 %vol, and a minimum ethyl mercaptan content of 1.0 lb/10,000 AG (Directorate General of Oil and Gas, 2020). These parameters are evaluated using standardized analytical methods, including ASTM D2163 for hydrocarbon composition analysis and ASTM D5305 for ethyl mercaptan determination (ASTM International, 2023a, 2023b).

Based on these considerations, this study focuses on evaluating LPG quality conformity through hydrocarbon composition and ethyl mercaptan content analysis. The objectives of this study are to determine the hydrocarbon composition of LPG samples using Gas Chromatography–Flame Ionization Detector (GC-FID), determine the ethyl mercaptan content, and evaluate the conformity of LPG samples with the quality standard established by the Directorate General of Oil and Gas.

Method

Materials

The materials used consisted of a Certified Reference Material (CRM) for LPG mixed, mixed LPG samples, and carrier gas (helium/nitrogen).

Instrumentation

The equipment used in this LPG analysis comprised a gas regulator, a Gas Chromatography–Flame Ionization Detector (GC-FID) instrument (Agilent Technologies 6890), connecting tubing, seal tape, a 500CC sample cylinder, a computer set, an adjustable wrench, a Gastec pump, a pressure gauge, a gas detector tube, and a Gastec pump assembly

Procedure

Verification of the Gas Chromatography Instrument

Instrument verification was performed using a Certified Reference Material (CRM) prior to LPG sample analysis to confirm the performance of the GC-FID system according to ASTM D2163, with adjustments following the operational conditions of the BBPMGB LEMIGAS laboratory. The CRM gas was transferred into a 500 cc sample cylinder and introduced into the GC-FID system after flushing the transfer line to minimize contamination. The analysis was conducted using a capillary column with helium as the carrier gas, an FID temperature of 200 °C, injector temperature of 80 °C, oven temperature program from 35 °C to 170 °C, injector pressure of 5.10 psi, and hydrogen and air flow rates of 30 and 300 mL/min, respectively. The measurement was performed in duplicate to evaluate repeatability. The obtained concentrations of thirteen hydrocarbon components were compared with the certified CRM values, and the system was considered acceptable when all components were within

the certified accuracy range. If results exceeded the acceptance criteria, potential causes such as contamination, instrument instability, or operational errors were investigated and verification was repeated.

Analysis of Hydrocarbon Composition (C_2 – C_5) using GC-FID (ASTM D2163)

The hydrocarbon composition of LPG samples was determined using GC-FID according to ASTM D2163, with modifications based on the analytical conditions established at BBPMGB LEMIGAS. The LPG samples were introduced into the GC-FID system through a sample cylinder after conditioning to ensure uniform gas composition. Analysis was performed using the validated analytical method, and each sample was measured at least in duplicate to evaluate repeatability. The obtained hydrocarbon concentrations were reported as liquid volume percentages (%vol) and were accepted when the ESTD value ranged between 98–102% for each component. The concentrations of individual compounds and liquid volume percentages were calculated based on response factors, peak areas, molecular weights, and specific gravity values according to Eqs. (1)–(5).

$$\text{Response Factor (\%)} = \frac{\text{concentration of standar (\%)}}{\text{area of standar}} \quad (1)$$

$$\text{Concentration of compound} = \text{response factor} \times \text{peak area of the compound} \quad (2)$$

$$\text{Mass of gas} = \% \text{Volume of gas} \times \text{molecular weight of gas} \quad (3)$$

$$\% \text{Volume of liquid} = \frac{\text{volume of liquid}}{\text{total volume of liquid}} \times 100 \quad (4)$$

$$\text{SG of liquid sample} = \frac{\% \text{volume of liquid} \times \text{SG of liquid}}{100} \quad (5)$$

Analysis of Ethyl Mercaptan (C_2H_5SH) using a Gas Detector Tube (ASTM D5305)

The ethyl mercaptan (C_2H_5SH) content in LPG samples was determined using a length-of-stain gas detector tube method according to ASTM D5305, with adjustments following the operational procedures of BBPMGB LEMIGAS. The analysis was conducted using the vapour phase of LPG samples collected from the vessel after flushing the sampling line to minimize contamination. Ethyl mercaptan concentration was determined based on the colour change observed in the detector tube, and measurements were repeated when no detectable colour change occurred. The obtained concentration values were corrected and converted according to the calculation procedures described in Eqs. (6)–(9).

$$\text{Corrected concentration (ppm)} = \text{ppm read} \times \frac{\text{specified stroke}}{\text{actual stroke}} \quad (6)$$

$$\text{ppm vapor} = \text{ppm read} \times \frac{760 \text{ mmHg}}{p \text{ (mmHg)}} \quad (7)$$

$$\text{ppm liquid} = \frac{\text{ppm vapour}}{\text{constant}} \quad (8)$$

$$\text{Ethyl mercaptan lb/10,000} = \frac{\text{ppm liquid}}{16.7 \text{ ppm}} \quad (9)$$

*The constant in Eq. (8) is taken from ASTM D5305 according to the temperature, and 1 lb/10,000 AG is equivalent to 16.7 ppm (ASTM D5305).

Result and Discussion

Verification of the GC-FID Instrument using a Certified Reference Material (CRM)

Before being used to analyse the LPG samples, the GC-FID instrument was first verified using a Certified Reference Material (CRM). The verification results were evaluated through chromatogram assessment, conformity with certified values, and repeatability evaluation to ensure the reliability of GC-FID measurements. Gas chromatography is widely applied for fuel quality assessment because it enables separation and identification of hydrocarbon components with high analytical reliability (Harold M. McNair, 2019; Kaplitz & Schug, 2023). The chromatogram of the CRM test shown in Fig.

1 displays a number of peaks appearing at different retention times, indicating that the hydrocarbon components in the CRM mixture could be separated by the chromatographic system (Zuas et al., 2016).

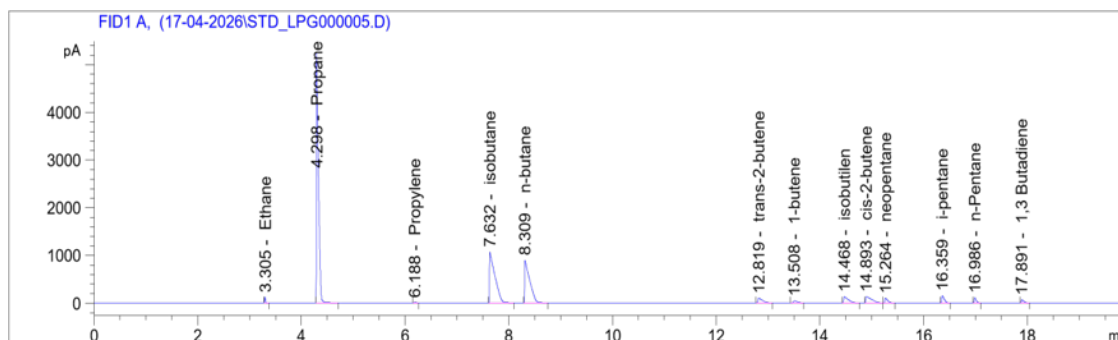


Figure 1. Chromatogram of the GC-FID verification using the CRM.

Based on Table 1, all the components analysed gave first and second measurement results that remained within the minimum and maximum ranges of the CRM. For example, the certified concentration of ethane was 0.7912 %mol with an acceptance range of 0.77537 to 0.80702 %mol. The measured ethane values in the first and second tests were 0.78847 and 0.789466 %mol, respectively, so that both lay within the acceptance range. The same was observed for propane as the component with the highest concentration, with a certified value of 54.444 %mol and measured values of 54.26728 and 54.39664 %mol. All the other components, from propylene through to 1,3-butadiene, likewise showed results within the stipulated minimum and maximum ranges.

In addition, the closeness of the measurement results to the CRM values and the consistency of the results between repetitions (Result 1 and Result 2) in Table 2 indicate that the GC-FID system had adequate performance at the time the verification was carried out. The use of a CRM to evaluate the conformity of measurement results is an important part of analytical quality control because it helps to demonstrate that the instrument and the method give reliable results. CRM are also used in the laboratory for instrument performance checks, method validation, and routine quality control (Zuas et al., 2016). Accordingly, on the basis of the verification results obtained, the GC-FID system could be used for the analysis of the hydrocarbon composition of the LPG samples.

Tabel 1. Comparison of the concentrations obtained in the verification with the CRM certificate.

No.	Component	Certified Conc (%mol)	Min	Max	Result 1 (%mol)	Result 2 (%mol)	Status
1	Ethane	0.7912	0.77537	0.80702	0.78847	0.789466	✓
2	Propane	54.444	53.3551	55.5329	54.26728	54.39664	✓
3	Propylene	0.1002	0.09819	0.1022	0.099479	0.100168	✓
4	Isobutane	19.98	19.5804	20.3796	19.88624	19.97074	✓
5	n-Butane	15.79	15.4742	16.1058	15.71396	15.78417	✓
6	Trans-2-Butane	1.501	1.47098	1.53102	1.49407	1.50202	✓
7	1-Butane	0.4957	0.48578	0.50561	0.49315	0.49591	✓
8	Isobutylene	1.996	1.95608	2.03592	1.98678	1.99617	✓
9	Cis-2-Butene	0.9118	0.89356	0.93004	0.907542	0.911172	✓
10	Neopentana	1.994	1.95412	2.03388	1.98432	1.99487	✓
11	Isopentana	0.9999	0.9799	1.01989	0.999354	0.997133	✓
12	N-Pentane	0.4973	0.48735	0.50724	0.501741	0.496936	✓
13	1.3-Butadiene	0.4989	0.48892	0.50887	0.496908	0.499366	✓

Analysis and Evaluation of the Hydrocarbon Composition of LPG

The results of the hydrocarbon composition analysis of the five LPG samples shown in Table 2 reveal differences in composition between the samples. The evaluation was carried out on three groups of components, namely C_2 , C_3+C_4 , and C_5+ . Based on KepDirjen Migas No. 116.K/10/DJM/2020, mixed LPG must have a maximum C_2 content of 0.8 %vol, a minimum C_3+C_4 content of 97.0 %vol, and a maximum C_5+ content of 2.0 %vol (Directorate General of Oil and Gas, 2020).

For the C_2 parameter, Samples 1 and 2 had contents of 0.1234 and 0.0394 %vol, respectively, and therefore remained below the maximum limit. Samples 3, 4, and 5, on the other hand, had C_2 contents of 0.9300, 0.8510, and 1.0281 %vol, which exceeded the maximum limit of 0.8 %vol. Sample 3 exceeded the limit by about 16.25 %, Sample 4 by about 6.38 %, and Sample 5 by about 28.51 % relative to the required maximum value. Thus, three of the five samples did not meet the composition requirement for the C_2 parameter, while the other two samples still complied. The differences in C_2 content between samples show that the composition of LPG can vary (Amin et al., 2023). This result is in line with (Shahin et al., 2020; Suryo et al., 2023), who emphasised the importance of composition analysis along the LPG supply chain from the refinery through the SPBE to the agent, because changes or variations in composition may occur throughout the distribution process. That study also showed that changes in the operating parameters of the production unit and in the blending process can cause variation in the specification of the final product and produce off-spec LPG. A high ethane content in LPG can affect the vapour pressure of the product (Shuard et al., 2017). LPG originating from oil refineries contains various hydrocarbon components in varying proportions, depending on the type of crude oil and the processing route (Lelevic et al., 2021; Paczuski et al., 2016). Ethane has a very low boiling point ($-88.6\text{ }^{\circ}\text{C}$), so that an excessive ethane content can raise the pressure inside the cylinder and potentially create a safety risk during storage and distribution. Furthermore, an increase in the ethane content of the fuel can lower the Gross Heating Value (GHV) and produce a less stable flame, thereby affecting combustion efficiency (Dabo Jnr et al., 2022).

In contrast to the C_2 results, all the samples met the C_3+C_4 requirement. The C_3+C_4 contents lay in the range 98.6579 to 99.0083 %vol, so that all the results were above the minimum limit of 97.0 %vol. The highest value was obtained for Sample 2 at 99.0083 %vol, while the lowest was found in Sample 5 at 98.6579 %vol. These results show that the composition of all five samples remained dominated by the C_3 and C_4 fractions and satisfied the requirement used in the evaluation. This is consistent with (Nofrizal et al., 2022), who analysed LPG composition using gas chromatography and reported that the samples analysed met the C_3+C_4 requirement of more than 97 %. Propane occupies a fairly large proportion because of its high calorific value, whereas butane is blended in so that the LPG is heavier than air; if the butane content is too high, the calorific value of the LPG is reduced and its use becomes less economical (Shen et al., 2017). However, if the butane content is too low, the LPG evaporates more readily and any leakage of the cylinder may rapidly lead to an explosion. LPG is a gas produced from the refining of petroleum and is used as a fuel for transport and household needs (Coutinho et al., 2022).

The C_5+ results likewise show that all the samples met the requirement. The C_5+ values lay in the range 0.2850 to 0.9521 %vol, well below the maximum limit of 2.0 %vol. The highest value was found in Sample 2 at 0.9521 %vol, while the lowest was found in Sample 3 at 0.2850 %vol. This result also agrees with the work of Nofrizal et al. (2022), who used a C_5+ limit of less than 2.0 in the evaluation of LPG composition and obtained samples that met the specification. Emphasizes that hydrocarbon composition analysis is essential for evaluating LPG quality because variations in hydrocarbon distribution can affect fuel properties and processing performance. Excessive heavy hydrocarbon fractions may negatively influence LPG characteristics. therefore, maintaining low C_5+ content is necessary to ensure product quality and compliance with specifications (Speight, 2019).

Overall, on the basis of the three composition parameters analysed, Samples 1 and 2 met all the hydrocarbon composition requirements, while Samples 3, 4, and 5 did not comply because their C_2 contents exceeded the maximum limit. All the samples, however, met the C_3+C_4 and C_5+ requirements. These results show that the parameter causing the non-conformity of the three samples is the C_2 content, and not the main C_3+C_4 fraction or the heavy C_5+ fraction.

Tabel 2. Results of the hydrocarbon composition analysis of the LPG samples.

Component	Volume Liquid (%mol)					Quality Standard
	1	2	3	4	5	
C2	0.1234	0.0394	0.93	0.851	1.0281	Max 0.80
C3+C4	98.997	99.0083	98.7848	98.6739	98.6579	Min 97.0
C5+	0.8789	0.9521	0.285	0.475	0.3139	Max 2.0

Analysis and Evaluation of the Ethyl Mercaptan Content

The results of the ethyl mercaptan analysis of the five LPG samples using the gas detector tube are shown in Table 3. Based on the LPG quality standard stipulated in KepDirjen Migas No. 116.K/10/DJM/2020, the ethyl mercaptan or butyl mercaptan content is required to have a minimum value of 1.0 lb/10,000 AG. Ethyl mercaptan concentration is an important safety parameter because odorization provides an effective warning system for detecting LPG leakage before reaching hazardous concentrations (Qian et al., 2021).

From the test results, the ethyl mercaptan contents of Samples 1, 2, 3, and 4 were 1.9; 1.8; 3.9; and 3.4 lb/10,000 AG, respectively, and therefore met the quality requirement. Sample 5, on the other hand, had a content of 0.96 lb/10,000 AG and this did not meet the stipulated minimum limit. These results show that most of the samples had an odorant content that still conformed to the standard, while one sample experienced a decrease in ethyl mercaptan content. Variation in the ethyl mercaptan content between samples may be influenced by storage conditions, vessel material, and distribution processes. Previous studies have reported that mercaptan-based odorants may decrease due to adsorption and chemical interactions with metal oxide surfaces during storage and distribution systems (Seguel et al., 2018).

The low ethyl mercaptan content in Sample 5 may be associated with odor fading phenomena during storage and distribution. Esfandiari et al. (2021) reported that odor fading may occur due to interactions between mercaptan compounds and metal oxide surfaces, particularly through chemical reactions with iron oxide present in distribution systems. Quantitative determination is required because human odor perception is affected by individual sensitivity and environmental conditions, resulting in limited reliability for regulatory compliance evaluation (Thiele et al., 2023).

In addition, the difference between the odour observation and the measured content shows that organoleptic identification cannot replace quantitative testing. Testing of the ethyl mercaptan content therefore remains necessary in order to ensure that the LPG meets the applicable quality standard.

Conclusion

Based on the testing and the evaluation of the quality conformity of Liquefied Petroleum Gas (LPG) with respect to hydrocarbon composition and ethyl mercaptan content at BBPMGB LEMIGAS, it can be concluded that the hydrocarbon composition analysis using Gas Chromatography showed variation in composition between the LPG samples. On the basis of the C₂, C₃+C₄, and C₅+ parameters, Sample 1 and Sample 2 met the quality requirements stipulated in KepDirjen Migas No. 116.K/10/DJM/2020, whereas Sample 3, Sample 4, and Sample 5 did not comply because their C₂ contents exceeded the stipulated maximum limit.

The ethyl mercaptan test results showed that most of the samples met the minimum odorant requirement of 1.0 lb/10,000 AG. Sample 1, Sample 2, Sample 3, and Sample 4 had ethyl mercaptan contents in accordance with the standard, whereas Sample 5 did not comply, with a content of 0.96 lb/10,000 AG.

Based on the overall evaluation of the hydrocarbon composition and ethyl mercaptan parameters, only Sample 1 and Sample 2 met all the quality parameters tested. This shows that the evaluation of LPG quality must be based on several parameters simultaneously, because conformity with one parameter does not guarantee that every aspect of LPG quality meets the applicable standard. It is further recommended that periodic LPG quality testing continue to be carried out using the hydrocarbon composition and ethyl mercaptan parameters in order to maintain product consistency during distribution, and that subsequent studies consider factors that may affect changes in LPG quality, such

as storage conditions, vessel material, storage duration, and the distribution history of the samples, with a larger number of replicates so that precision and repeatability can be evaluated more thoroughly.

Acknowledgments

The author would like to thank BBPMGB LEMIGAS, in particular the Gas Composition Working Group of the Gas Technology Laboratory, for the opportunity, guidance, and facilities provided during the internship, and the Chemistry Study Program, Faculty of Mathematics and Natural Sciences, Universitas Negeri Jakarta, for its support during the preparation of this work.

Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of this article. The authors declare that the research was conducted independently, and no financial or commercial relationships influenced the study design, data analysis, interpretation of results, or manuscript preparation.

Author Contributions

A.N.S. contributed to conceptualization, methodology development, experimental analysis, data interpretation, and manuscript preparation. F.K.H. and M.W. contributed to laboratory supervision, analytical procedure verification, and technical review of the experimental results. G.F. contributed to data validation, scientific discussion, and manuscript review. All authors have read and approved the final version of the manuscript.

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