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Determination of Lead Content in Gasoline Samples Using Flame Atomic Absorption Spectrometry (FAAS)

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Abstract

The determination of lead content in gasoline remains essential for environmental monitoring and regulatory compliance, despite the global phase-out of leaded fuel. This study employed Flame Atomic Absorption Spectrometry (FAAS) to analyze lead concentrations in gasoline samples from four commercial brands: Petronas, Shell, Caltex, and Petron. Sample preparation involved chemical stabilization using iodine and Aliquat 336 in MIBK. Calibration was performed using lead standard solutions in the range of 0.528 to 2.642 ppm. Results indicated extremely low lead concentrations across all samples (0.1857 – 0.3697 ppm), aligning with Malaysian and international regulatory limits. Recovery tests using spiked samples yielded results within 99–106%, confirming methodological accuracy. This study reaffirms the effectiveness of FAAS for trace lead analysis and the continuing compliance of Malaysian fuel with environmental standards.

Keywords: FAAS, Gasoline, Lead, Analysis

1. Introduction

The measurement of lead (Pb) in gasoline remains a vital analytical task despite the global elimination of leaded fuel in vehicles. In areas with lingering contamination or where aviation gasoline and illegal fuel use continue to be of concern, accurate detection of trace lead in petroleum products is crucial for environmental monitoring, regulatory enforcement, and public health protection. Among available methods, atomic absorption spectrometry (AAS) is still a practical and common choice due to its selectivity, moderate sensitivity, and ease of use [1-5].

Historically, lead additives like tetraethyl lead were widely used to boost octane ratings in gasoline, leading to widespread atmospheric emissions and environmental pollution. Analytical techniques developed in the mid-20th century, particularly flame AAS and graphite furnace AAS, provided the sensitivity needed to measure lead at concentrations relevant to industrial formulations and evolving standards. Standard procedures such as ASTM D3237 and EPA Method 2 are still used today for compliance checks, especially in quality control labs where cost and throughput limits make more advanced instruments impractical [6]. While techniques like inductively coupled plasma mass spectrometry (ICP-MS) offer lower detection limits and the ability to analyze multiple elements, they usually require more complex sample prep, higher operational costs, and infrastructure that may not be available everywhere. X-ray fluorescence

(XRF) methods, although useful for quick field screening, generally lack the sensitivity and precision needed for trace-level measurements [7-10]. As a result, AAS remains central where reliable, validated lead quantification in gasoline is needed at detection limits aligned with current standards.

This study assesses AAS performance for measuring lead in gasoline, focusing on analytical precision, accuracy, and detection limits under current laboratory conditions. We also evaluate the method's suitability for ongoing regulatory monitoring and discuss its implications for environmental exposure risks associated with residual or illegally used lead-containing fuels. By sharing experimental results within the framework of modern analytical standards, this work helps clarify the operational strengths and limitations of AAS in trace lead analysis of petroleum products.

2. Methodology

2.1 Principle of FAAS

Lead in gasoline was determined by FAAS after chemical conversion of organolead species to iodide complexes and extraction into MIBK using Aliquat 336 as a phase-transfer reagent. Extracts were aspirated into an air–acetylene flame and measured at 283.3 nm. Concentration was obtained by interpolation from matrix-matched calibration standards.

2.2 Reagents and standards

All reagents were analytical grade. Key reagents included $PbCl_2$ (primary standard), Aliquat 336, MIBK, iodine solution (3 g iodine per 100 mL toluene), and isooctane (lead-free gasoline). Primary stock and working standards were prepared as follows:

A standard stock solution, containing 1321 ppm Pb equivalent and 5.0 g Pb per gallon, was prepared by dissolving 0.4433 g of dried $PbCl_2$ in 10% (v/v) Aliquat/MIBK and diluting to volume. An intermediate standard solution, with 264.2 ppm Pb equivalent and 1.0 g Pb per gallon, was made by diluting 50 mL of the stock solution to 250 mL with 1% Aliquat/MIBK. Later, calibration standards in the range of 0.528–2.642 ppm were created using aliquots (1–5 mL) of the intermediate solution mixed with 5 mL of 1% Aliquat/MIBK and then diluted to 50 mL with MIBK to achieve nominal concentrations of 0.528, 1.057, 1.580, 2.114, and 2.642 ppm Pb, Table 1.

Table 1: Pb standard solution series

Concentration of Lead, Standard Solutions(g Pb/gal)	Volume of 1.0 g Pb/gal Lead Standard Solutions taken (ml)	Volume of Aliquot 336/ MIBK Solution (1% v/v) (ml) added	Concentration of Lead, Standard Solutions(ppm)	Final volume of volumetric flask (ml)
0.02	1.0	5.0	5.280	50
0.04	2.0	5.0	10.568	50
0.06	3.0	5.0	15.800	50
0.08	4.0	5.0	21.140	50
0.10	5.0	5.0	26.42	50

2.3 Sample collection and preparation

Gasoline samples were collected from four petrol stations (Petronas, Shell, Caltex, Petron). For analysis, 5.0 mL of each gasoline sample was pipetted into a 50 mL volumetric flask containing 30 mL of MIBK. Then, 0.10 mL of iodine solution was added, and the mixture was allowed to react for approximately 1 minute. Next, 5.0 mL of Aliquat 336/MIBK (1% v/v) was added, and the flask was filled to the mark with MIBK and mixed before analysis. Spiked samples were prepared in the same way, with the addition of 5.0 mL of a 1.0568 ppm Pb standard.

2.4 Instrumentation and analytical procedure

FAAS measurements were performed at 283.3 nm using an air–acetylene flame under oxidizing conditions. Burner height and aspiration rate were optimized using the reagent blank and a mid-range standard. Calibration was performed with matrix-matched standards (isooctane + Aliquat/MIBK + iodine). MIBK was aspirated between samples to reduce carryover. Instrument make/model, lamp current, slit width, burner height and gas flow rates should be reported in the methods for reproducibility.

2.5 Quality control and calculations

A reagent blank and a quality control (QC) solution (1.580 ppm) were analyzed in the same run. Spike recovery assessments were performed by the addition of 1.0568 ppm Pb to each gasoline sample. Concentrations were calculated from the calibration regression (absorbance y vs concentration x, ppm):

$$\mathbf{y = 0.0174\,x - 0.000004 \quad (R^2 = 0.9891)}$$

Concentration (x) calculated as $x = (y + 0.000004)/0.0174$, **Figure 1**.

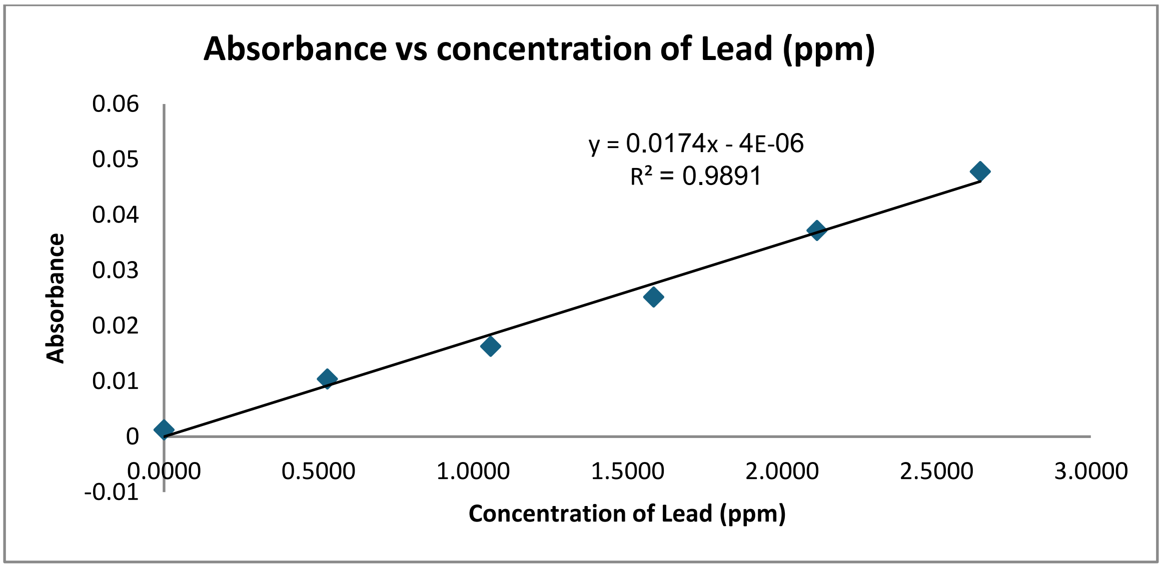

Figure 1: Calibration Curve of Absorbance versus Concentration of Lead (ppm)

3. Results

3.1 Calibration

Calibration absorbances and concentrations are presented in Table 1. The calibration showed a linear response over the working range with $R^2 = 0.9891$. A calibration plot with residuals is recommended for submissions.

Table 1. Calibration standards and absorbances

Concentration (ppm)	Absorbance
0.000 (blank)	0.0012
0.528	0.0104
1.0568	0.0163
1.580	0.0252
2.114	0.0372
2.642	0.0478

3.2 Sample concentrations

Measured absorbances and calculated Pb concentrations for unspiked samples are shown in Table 2. All samples were below national and international regulatory limits.

Table 2. Lead concentrations in gasoline samples

Sample	Absorbance	Pb (ppm)
Petronas	0.00250	0.1972
Shell	0.00550	0.3697
Caltex	0.00230	0.1857
Petron	0.00400	0.2834
QC sample	0.0256	1.6388

3.3 Spike recovery and QC

Spike recovery data are provided in Table 3. Recoveries between 99% and 106% indicate acceptable accuracy and negligible matrix effects under the present conditions.

Table 3. Spike recovery results (spike = 1.0568 ppm)

Sample	Spiked Conc. (ppm)	Unspiked Conc. (ppm)	Recovery (%)
Petronas	1.3200	0.1972	106.25
Shell	1.4362	0.3697	100.92
Caltex	1.2347	0.1857	99.26
Petron	1.3712	0.2834	102.93

4. Discussion

The FAAS method with iodine complexation and Aliquat 336–MIBK extraction produced reproducible results and acceptable spike recoveries, indicating the method is fit for routine monitoring of Pb in gasoline at low ppm levels. All tested gasoline samples contained Pb concentrations (0.186–0.370 ppm) far below regulatory limits (e.g., Malaysia <13.2 ppm). Key uncertainties include a lack of formally reported LOD/LOQ and replicate precision; these should be calculated and reported prior to final submission. Potential contamination sources (reagent impurities, sampling/handling) were mitigated through blanks and QC, but reagent blank statistics should be provided to quantify LOD. A calibration plot (with residuals) and replicate data will strengthen the manuscript and satisfy typical journal requirements.

The use of iodine reduced variation due to differing alkyl lead forms by forming stable iodide complexes. Aliquat 336, a quaternary ammonium salt, acted as a phase transfer agent, enhancing extraction and analytical signal stability. Isooctane addition mimicked the gasoline matrix, improving method selectivity. Lead contamination from reagents was controlled by using blanks, amber glassware, and high-purity reagents.

5. Conclusion

This study demonstrated that FAAS remains a robust and reliable method for determining trace levels of lead in gasoline. The use of chemical modifiers like iodine and Aliquat 336 enhanced stability and detection. All gasoline samples analyzed were well below regulatory limits, confirming the effective implementation of unleaded fuel policy in Malaysia. Spike recovery and quality control results validated the accuracy and precision of the analytical method employed.

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