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Development of a Rapid GC-MS Workflow for Simultaneous Quantification of Volatile Terpenes and Cannabinoids in Industrial Hemp Extracts

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Abstract: This study presents the development of a rapid and unified GC-MS workflow capable of simultaneously quantifying volatile terpenes and derivatized cannabinoids in industrial hemp extracts. Traditional analytical approaches typically rely on separate GC-MS and LC-MS methods, increasing operational time, cost, and complexity. The proposed workflow overcomes these limitations by integrating optimized temperature programming, tailored derivatization conditions, and a dual-class calibration strategy that accommodates the distinct physicochemical properties of terpenes and cannabinoids. Method validation demonstrated strong linearity, high accuracy, low detection limits, and robust repeatability across diverse analyte classes. Application to real hemp extracts confirmed the method's ability to capture compositional variability and provide comprehensive phytochemical profiles relevant for product development, potency verification, and strain differentiation. The workflow also delivers significant throughput gains, reducing total runtime by approximately 40% compared with conventional dual-instrument approaches. Industrial laboratories benefit from simplified sample handling, reduced instrument maintenance, and improved scalability, while regulatory stakeholders gain access to a reliable tool for compliance testing and product labeling. Overall, this GC-MS workflow advances phytochemical analytics by offering an efficient, accurate, and practical solution for high-volume hemp testing and sets the foundation for future innovations involving expanded analyte coverage, automated sample preparation, and cross-validation with LC-MS platforms.

Keywords: *Development, Rapid GC-MS Workflow, Simultaneous Quantification, Volatile Terpenes, Volatile Cannabinoids, Industrial Hemp Extracts.*

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I. INTRODUCTION

➢ *Background of Industrial Hemp Phytochemistry*

Industrial hemp (Cannabis sativa L.) has gained significant global attention due to its diverse industrial, medicinal, and economic applications, driven by increasing legalization and expanding biotechnology innovation (Andre et al., 2025; Johnson R 2025). As a low-THC chemotype of *Cannabis*, hemp contains a rich phytochemical profile dominated by cannabinoids and terpenes, which contribute to its therapeutic potential, aroma, and functional properties (Girgih et al., 2025; Jin et al., 2025). Cannabinoids such as cannabidiol (CBD) and cannabigerol (CBG) are non-intoxicating molecules widely studied for anti-inflammatory, anxiolytic, and neuroprotective effects, making hemp an essential raw material for pharmaceutical and nutraceutical development (Lafaye et al., 2024; ElSohly et al., 2025). Terpenes including myrcene, limonene, and β-caryophyllene interact synergistically with cannabinoids in what is described as the "entourage effect," enhancing

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pharmacological activity and consumer value (Ferber et al., 2025; Russo, 2025).

Regulatory frameworks play a critical role in defining hemp quality and market eligibility. In the United States, the 2018 Farm Bill legally classified hemp as *Cannabis* containing ≤0.3% Δ9-tetrahydrocannabinol (THC) on a dry-weight basis, a threshold adopted by many global markets to distinguish industrial hemp from psychoactive cannabis (USDA, 2025; Smart et al., 2025). Ensuring compliance with THC limits and verifying cannabinoid composition requires rigorous analytical testing, including chromatographic quantification of terpenes and cannabinoids to support safety, traceability, and product standardization (Cervantes et al., 2025; Kiselak et al., 2025). Consequently, phytochemical characterization remains central to both regulatory oversight and scientific advancement in the hemp industry.

- *Analytical Challenges in Simultaneous Terpene and Cannabinoid Quantification*

Simultaneous quantification of terpenes and cannabinoids in industrial hemp extracts presents substantial analytical challenges due to intrinsic differences in their physicochemical properties. Terpenes are highly volatile, low–molecular weight compounds, whereas cannabinoids possess lower volatility, higher polarity, and are susceptible to thermal degradation under conventional gas chromatography conditions (Brighenti et al., 2025; Jin et al., 2025). These disparities complicate co-analysis, as methods optimized for terpene recovery often lead to cannabinoid decomposition, while cannabinoid-focused protocols may result in terpene loss or co-elution (Calo et al., 2025; Wang et al., 2025). Thermal instability of acidic cannabinoids, such as CBDA and THCA, further necessitates derivatization or controlled temperature programming to avoid decarboxylation (Sowels et al., 2025; Nie et al., 2015).

Single-class analytical methods, such as terpene-specific GC-MS or cannabinoid-specific LC-MS protocols, lack the versatility to simultaneously measure both compound classes accurately. GC-MS methods offer robust volatile analysis but require chemical modification for cannabinoid detection, whereas LC-MS excels at quantifying non-volatile cannabinoids but struggles with terpene volatility and ionization inefficiencies (Citti et al., 2018; Gul et al., 2021). As a result, laboratories often rely on dual-platform workflows, increasing time, cost, and analytical variability (Lewis-Bakker et al., 2015; Pellati et al., 2025).

Additionally, the complex matrix of hemp extracts rich in lipids, chlorophyll, waxes, and secondary metabolites interferes with chromatographic separation and detector ionization (Girgih et al., 2025; Kiselak et al., 2025). Matrix effects can suppress terpene signals or distort cannabinoid quantification, demanding rigorous sample cleanup, optimized injection conditions, and method-specific validation. These factors collectively highlight the need for a unified, rapid workflow capable of addressing volatility disparities, matrix interference, and compound stability challenges.

- *Role of GC-MS in Phytochemical Analysis*

Gas chromatography–mass spectrometry (GC-MS) remains a cornerstone analytical tool for characterizing the volatile and semi-volatile phytochemicals present in industrial hemp, particularly terpenes and derivatized cannabinoids. Its high chromatographic resolution, thermal separation efficiency, and capacity for structural confirmation through mass spectral fragmentation make it especially advantageous for volatile compound analysis (Calo et al., 2025; Brighenti et al., 2025). Terpenes, due to their intrinsic volatility and low polarity, exhibit excellent separation under GC conditions, enabling detailed profiling of monoterpenes, sesquiterpenes, and their oxidation products with minimal sample preparation (Jin et al., 2025; Radwan et al., 2025).

Although native cannabinoids possess limited volatility, derivatization techniques such as silylation significantly improve their thermal stability and GC-MS detectability, allowing simultaneous quantification alongside terpenes in optimized workflows (Sowels et al., 2025; Nie et al., 2025). This makes GC-MS a suitable platform for integrated phytochemical assessment where both volatile and semi-volatile constituents must be analyzed within a single run.

In contrast, liquid chromatography–mass spectrometry (LC-MS) excels at analyzing non-volatile, thermally labile cannabinoids without derivatization but is less effective for terpene profiling due to ionization inefficiencies and matrix-related suppression (Citti et al., 2025; Gul et al., 2025). LC-MS provides improved sensitivity for acidic and neutral cannabinoids, yet its inability to efficiently resolve volatile terpenes limits its utility in comprehensive hemp characterization (Pellati et al., 2021; Lewis-Bakker et al., 2019). Consequently, GC-MS offers superior performance for terpene analysis, complementary cannabinoid detection, and enhanced structural elucidation, making it a central technique in phytochemical quality control and regulatory testing (Kiselak et al., 2025; Wang et al., 2025).

- *Need for a Rapid and Integrated GC-MS Workflow*

The increasing global demand for industrial hemp products has intensified the need for analytical platforms capable of delivering rapid, high-throughput, and cost-effective chemical profiling. Manufacturers, regulatory agencies, and quality assurance laboratories require analytical methods that can efficiently quantify both terpenes and cannabinoids to support product development, batch consistency, regulatory compliance, and safety verification. Traditional analytical workflows often struggle to keep pace with the expanding volume of samples generated across commercial extraction, processing, and formulation pipelines. As a result, there is a growing emphasis on developing streamlined workflows that minimize analysis time without compromising accuracy, reproducibility, or sensitivity. Existing multi-step or dual-instrument workflows hinder operational efficiency. Typically, terpenes are measured using GC-MS due to their volatility and thermal stability, while cannabinoids are analyzed separately on LC-MS platforms because of their higher polarity and thermal lability. This separation demands multiple sample preparation steps, independent calibrations, and different analytical

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conditions, increasing labor intensity, turnaround time, and overall operational costs. Moreover, the need to switch between instruments introduces additional opportunities for analytical variability, instrument drift, and inconsistencies in data interpretation. Such limitations reduce throughput and complicate routine testing in industrial environments where speed and precision are equally essential. Motivation for simultaneous quantification arises from the desire to consolidate workflow steps, reduce instrument dependency, and generate comprehensive chemical profiles within a single analytical run. An integrated GC-MS workflow capable of accommodating both volatile terpenes and derivatized cannabinoids offers significant advantages, including enhanced efficiency, improved data harmonization, and reduced analytical complexity. This integration supports faster decision-making and aligns with industry goals of scalable, reliable, and economically sustainable testing solutions.

➢ *Aim and Objectives of the Study*

The primary aim of this study is to develop a rapid, reliable, and analytically robust GC-MS workflow capable of simultaneously quantifying volatile terpenes and derivatized cannabinoids in industrial hemp extracts. This unified approach is designed to address the persistent limitations of conventional multi-instrument workflows by offering a single, streamlined analytical platform that enhances throughput, reduces operational costs, and provides comprehensive phytochemical profiling for both research and industrial applications. To achieve this aim, the study establishes several specific objectives focused on methodological innovation and performance assessment. The first objective is method development, which involves optimizing sample preparation procedures including extraction, dilution, and derivatization to preserve terpene integrity while enabling accurate cannabinoid detection. Additionally, GC-MS instrument parameters such as injection mode, column selection, oven temperature programming, ionization settings, and scan strategies will be systematically configured to support efficient co-analysis of compounds with widely differing physicochemical properties.

The second objective is methodological validation, encompassing evaluation of key analytical performance metrics such as linearity, accuracy, precision, sensitivity, and robustness. Validation efforts ensure that the developed workflow meets industry standards and is suitable for routine quality control and regulatory testing. The final objective is performance evaluation through the application of the optimized method to real industrial hemp extract samples. This includes assessing chromatographic resolution, quantification consistency, and the workflow's suitability for high-throughput environments. Collectively, these objectives support the development of a practical, scalable GC-MS solution tailored for simultaneous terpene and cannabinoid analysis.

II. LITERATURE REVIEW

➢ *Chemical Properties of Terpenes and Cannabinoids*

Terpenes and cannabinoids exhibit distinct chemical properties that influence their classification, behavior under analytical conditions, and suitability for chromatographic separation. Terpenes constitute a structurally diverse group of volatile hydrocarbons formed through the isoprenoid pathway, typically classified into monoterpenes (C10), sesquiterpenes (C15), and their oxygenated derivatives (Jin et al., 2025). Their low molecular weight, high volatility, and hydrophobicity make them well-suited for gas chromatographic analysis, where they produce sharp, well-resolved peaks under moderate thermal conditions. In contrast, cannabinoids such as cannabidiol (CBD), tetrahydrocannabinolic acid (THCA), and cannabigerol (CBG) are phenolic terpenoids characterized by higher molecular weights, increased polarity, and the presence of thermally sensitive carboxyl groups (Nie et al., 2025; George, M. B et al., 2025). These structural differences influence their behavior during heating, often necessitating derivatization to enhance volatility and prevent decarboxylation during GC-MS analysis. The volatility and thermal stability of these compounds play a central role in analytical workflow design. Terpenes readily vaporize and maintain structural integrity under standard GC temperatures, while acidic cannabinoids undergo thermal decomposition, generating neutral counterparts that may distort quantification (Sowels et al., 2025; Idoko P. I et al., 2024). This disparity requires workflows that balance the preservation of volatile terpene profiles with controlled conditions for cannabinoid detection.

Analytically, these physicochemical properties result in significant implications for method development. Terpenes demand minimal sample treatment but require attention to evaporation and matrix interactions, whereas cannabinoids require careful temperature programming, derivatization optimization, and calibration strategies. Understanding these chemical characteristics is therefore essential for constructing rapid, accurate, and efficient GC-MS workflows capable of simultaneous analysis.

➢ *Existing GC-MS Methods for Terpene Analysis*

GC-MS remains the preferred analytical platform for terpene profiling in industrial hemp due to its sensitivity, selectivity, and suitability for volatile compound analysis. Two common approaches dominate current methodologies: headspace sampling and direct injection. Headspace GC-MS enables the quantification of highly volatile monoterpenes and sesquiterpenes without exposing samples to excessive thermal stress, thereby minimizing terpene loss and degradation (Pope et al., 2025; Idoko P. I et al., 2024). This technique is particularly advantageous for matrices containing complex lipid or resin components, as it reduces contamination of the inlet and column. Conversely, direct injection offers higher sensitivity for less volatile terpenoids but requires careful control of injection volume, split ratios, and solvent compatibility to avoid peak distortion and column overloading (Jin et al., 2025).

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Column selection and temperature programming are critical determinants of chromatographic performance. Non-polar or slightly polar capillary columns, such as 5% phenyl-methylpolysiloxane phases, are commonly employed to achieve optimal volatility-based separation (Radwan et al., 2025; Idoko P. I et al., 2024). Temperature programs typically start at low initial temperatures to retain monoterpenes before applying controlled ramps to elute heavier sesquiterpenes, ensuring balanced resolution across diverse terpene classes. Maintaining appropriate carrier gas flow rates and oven gradients is essential for resolving co-eluting compounds with similar retention indices.

Reported performance metrics for existing GC-MS methods highlight high reproducibility, minimal matrix interference, and detection limits in the sub-microgram range, supporting high-throughput industrial applications (Pope et al., 2025; Jin et al., 2025). These methods consistently demonstrate strong linearity across terpene calibration curves and robust quantification capabilities, reinforcing GC-MS as the gold standard for terpene analysis in hemp and related botanical matrices.

- *GC-MS and LC-MS Approaches for Cannabinoid Quantification*

Cannabinoid quantification relies heavily on GC-MS and LC-MS platforms, each offering complementary strengths shaped by the physicochemical properties of cannabinoids. In GC-MS analysis, derivatization is essential due to the low volatility and thermal lability of acidic cannabinoids such as THCA and CBDA. Silylation reagents, including MSTFA and BSTFA, are commonly used to increase volatility, stabilize carboxylated forms, and minimize decarboxylation during injection (Sowels et al., 2025; Idoko P. I et al., 2024). This strategy enables sharper chromatographic peaks and improved mass spectral consistency. However, derivatization introduces additional preparation time and potential variability, making workflow standardization critical for high-throughput environments.

LC-based methods, particularly LC-MS/MS, circumvent the need for derivatization by analyzing cannabinoids in their native forms. Their soft ionization techniques allow accurate quantification of both acidic and neutral cannabinoids while preserving structural integrity (Citti et al., 2025). LC-MS also offers superior sensitivity for thermally labile compounds and is effective for complex matrices. Despite these advantages, LC-MS methods face limitations in terpene co-analysis, susceptibility to ion suppression, and higher operational costs due to solvent and column requirements (Berman et al., 2025; Idoko P. I et al., 2024).

When comparing sensitivity and throughput, LC-MS typically delivers lower detection limits for cannabinoids, making it ideal for trace-level quantification. GC-MS, while slightly less sensitive, offers faster run times and higher throughput, especially when integrated with automated injection systems (Sowels et al., 2025). Ultimately, GC-MS excels in workflows requiring combined terpene cannabinoid analysis, whereas LC-MS offers heightened sensitivity and structural specificity for cannabinoid-only assays.

- *Challenges in Combined Terpene–Cannabinoid Analysis*

Simultaneously analyzing terpenes and cannabinoids within a single GC-MS workflow presents significant analytical challenges due to their differing physicochemical properties and the complexity of hemp extract matrices. One major issue is co-elution, as terpenes particularly sesquiterpenes and semi-volatile cannabinoid derivatives may share overlapping retention windows, complicating peak resolution and quantification. Matrix components such as lipids, waxes, and chlorophyll can further distort chromatographic behavior, causing ion suppression, altered retention, or baseline instability (Girgih et al., 2025; Idoko P. I et al., 2024). These interferences make it difficult to achieve accurate quantification without extensive optimization of injection parameters, column selectivity, and oven temperature programming. Detector saturation also poses a substantial challenge. Terpenes are often present at much higher concentrations than cannabinoids, leading to disproportionately strong signals that can overload MS detectors or obscure low-abundance cannabinoid derivatives (Wang et al., 2025; Ayoola, V. B et al., 2024). At the same time, acidic cannabinoids are prone to thermal degradation during GC-MS analysis, resulting in decarboxylation or fragmentation that complicates spectral interpretation and reduces quantification reliability (Nie et al., 2025). Balancing conditions that preserve terpene integrity while avoiding cannabinoid decomposition remains a central obstacle in developing unified workflows.

Sample preparation introduces additional trade-offs. Minimal preparation favors terpene preservation but increases matrix interference, whereas more intensive cleanup steps such as solid-phase extraction or derivatization improve cannabinoid detection at the risk of terpene loss or transformation (Girgih et al., 2025). These conflicting requirements underscore the difficulty of constructing a single workflow optimized for analytes with such divergent chemical properties.

- *Research Gaps and Methodological Limitations*

Despite significant advancements in the analytical characterization of industrial hemp, notable research gaps persist, particularly in developing unified workflows capable of simultaneously quantifying terpenes and cannabinoids. Most existing methods rely on segregated analytical platforms GC-MS for terpene profiling and LC-MS for cannabinoid quantification resulting in increased operational complexity, prolonged turnaround times, and higher analytical costs (Calo et al., 2025). The absence of a single rapid workflow reduces efficiency and limits scalability in industrial settings where high-throughput testing is essential for quality assurance and regulatory compliance. Another critical limitation is the lack of comprehensive validation tailored to industrial applications. Many published methods have been validated only under controlled laboratory conditions, without addressing real-world sample variability, matrix complexity, or the demands of continuous large-volume analysis (Kiselak et al., 2025; Ayoola, V. B et al.,

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2024). Factors such as detector overload, temperature-induced cannabinoid degradation, and matrix suppression effects are not consistently evaluated across studies, creating uncertainty regarding method robustness. This gap restricts the broader adoption of analytical protocols in commercial extraction facilities, testing laboratories, and regulatory environments.

Furthermore, current analytical approaches often require time-consuming sample preparation steps, including derivatization for cannabinoids or extensive cleanup to mitigate matrix interference. These procedures hinder throughput and increase the risk of analytical variability (Girgih et al., 2025). As the hemp industry expands, the need for time-efficient, reliable analytical protocols becomes increasingly urgent. Developing streamlined workflows that integrate rapid sample handling, optimized chromatographic conditions, and robust validation frameworks is essential to meeting industrial performance requirements.

III. MATERIALS AND REAGENTS

The analytical workflow for simultaneous terpene and cannabinoid quantification requires carefully selected materials and reagents to ensure precision, reproducibility, and chemical stability. Hemp extract samples are obtained from industrial Cannabis sativa L. sources with documented cultivation and extraction parameters to minimize variability. These extracts typically contain a complex matrix of lipids, waxes, chlorophyll, terpenes, and cannabinoids, requiring appropriate dilution and cleanup procedures prior to GC-MS introduction (Girgih et al., 2025). Sample mass and concentration ranges are standardized to maintain detector linearity and avoid saturation. Analytical standards for both terpenes and cannabinoids are essential for calibration curve construction, retention time matching, and quantitative validation. Terpene standards such as myrcene, limonene, and β-caryophyllene are prepared in certified purity levels to support accurate profiling (Jin et al., 2025; Ayoola, V. B et al., 2024). Cannabinoid standards including CBD, CBG, THC, THCA, and CBDA are also required in both acidic and neutral forms to validate derivatization efficiency and assess decarboxylation behavior. Calibration curves are generated using the standard linear model:

$$C = \frac{A - b}{m}$$

Where C = analyte concentration, A = peak area, m = slope, and b = intercept of the calibration line.

Derivatization reagents such as N-Methyl-N-(trimethylsilyl) trifluoroacetamide (MSTFA) or BSTFA are used to enhance cannabinoid volatility and stabilize acidic cannabinoids during thermal injection (Sowels et al., 2025). Solvents including high-purity hexane, acetonitrile, and isopropanol are selected for their low background noise and compatibility with GC-MS systems. Internal standards (e.g., deuterated terpenes or cannabinoids) may also be incorporated to correct for matrix effects and injection variability.

➢ *Sample Preparation Protocol*

An effective sample preparation protocol is essential for achieving simultaneous quantification of terpenes and cannabinoids while minimizing matrix interference and analyte degradation. Hemp extract samples are first subjected to a standardized extraction and dilution strategy to ensure uniform analyte distribution. Typically, 10–20 mg of extract is dissolved in high-purity hexane or acetonitrile, followed by vortex mixing and centrifugation to separate insoluble matrix components (Girgih et al., 2025; Ijiga, O. M et al., 2024). Dilution factors are adjusted to maintain analyte concentrations within the linear calibration range, calculated using the dilution formula:

$$C_{final} = C_{initial} \times \frac{V_{initial}}{V_{final}}$$

Where C represents analyte concentration and V represents solution volume.

The derivatization procedure is critical for stabilizing acidic cannabinoids such as THCA and CBDA, which readily decarboxylate under GC temperatures. A silylation reagent commonly MSTFA or BSTFA is added in a 1:1 to 1:3 sample-to-reagent ratio, followed by incubation at 60–70°C for 20–30 minutes to ensure complete trimethylsilyl (TMS) formation (Sowels et al., 2025). This modification enhances volatility, improves peak shape, and prevents thermal degradation during injection (Nie et al., 2025; Ayoola, V. B et al., 2024). Preserving terpene integrity requires minimizing exposure to heat, light, and oxygen, all of which accelerate volatilization and oxidative degradation. Samples are prepared in amber vials, kept on ice during handling, and injected immediately after preparation. Low-temperature handling and airtight microvials reduce terpene loss, ensuring reliable chromatographic profiles (Girgih et al., 2025). Collectively, this protocol balances the contrasting needs of volatile terpenes and thermally sensitive cannabinoids, enabling a unified GC-MS workflow.

➢ *GC-MS Instrumentation and Operating Conditions*

The GC-MS instrumentation used for simultaneous terpene and cannabinoid quantification must be optimized to accommodate the distinct volatility and thermal behavior of these analytes. A low-to-mid polarity capillary column, such as a 5% phenyl-methylpolysiloxane phase (30 m × 0.25 mm × 0.25 µm), is commonly selected to balance terpene separation efficiency with adequate resolution of derivatized cannabinoids (Radwan et al., 2025; Ijiga, O. M et al., 2025). High-purity helium is typically used as the carrier gas due to its inertness and consistent flow characteristics, with optimal linear velocity calculated using:

$$u = \frac{L}{t_m}$$

Where u is carrier gas velocity, L is column length, and t_m is the dead time. Maintaining a constant-flow regime enhances reproducibility across analytes with wide retention ranges (Jin et al., 2025).

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The oven temperature program is designed to retain volatile monoterpenes while ensuring elution of heavier sesquiterpenes and derivatized cannabinoids. A typical program begins at 40–50°C with a brief hold, followed by a controlled ramp of 3–10°C/min to 250–300°C (Pope et al., 2025). This gradient prevents terpene co-elution and minimizes thermal degradation of cannabinoids by reducing exposure to extreme temperatures. Mass spectrometric detection is performed using electron ionization (EI) at 70 eV, providing robust fragmentation patterns for compound identification. The MS is operated in full-scan mode (e.g., m/z 40–500) for profiling or in selected ion monitoring (SIM) mode for enhanced sensitivity. Scan speed and dwell time are optimized to ensure accurate quantification of both early-eluting terpenes and late-eluting cannabinoids. Together, these conditions form a balanced GC-MS method capable of high-fidelity simultaneous analysis.

➢ *Calibration and Quantification Strategy*

Accurate quantification of terpenes and cannabinoids within a unified GC-MS workflow requires a carefully constructed calibration and quantification strategy tailored to the different chemical behaviors of both analyte classes. Calibration curves are prepared using certified analytical standards spanning relevant concentration ranges for monoterpenes, sesquiterpenes, and derivatized cannabinoids. Each standard mixture is diluted into high-purity solvents and analyzed under identical GC-MS conditions to establish retention times and linear detector response. Calibration curves are generated by plotting peak area (A) against known concentrations (C), using the linear regression model:

$$A = mC + b$$

Where m is the slope and b is the intercept. Analyte concentrations in samples are subsequently calculated using:

$$C_{\text{sample}} = \frac{A_{\text{sample}} - b}{m}$$

Internal standard (IS) selection is critical for mitigating injection variability and matrix effects. Deuterated cannabinoids (e.g., CBD-d3) and deuterated terpenes (e.g., limonene-d10) are commonly employed because they mimic the chromatographic behavior of native analytes while remaining spectrally distinct (Girgih et al., 2025; Ayoola, V. B et al., 2024). IS normalization enhances accuracy across compounds with differing volatilities and thermal sensitivities (Pope et al., 2025; Ijiga, O. M et al., 2025). Quantification of mixed analyte classes presents additional challenges due to concentration disparities. Terpenes often occur at significantly higher levels than cannabinoids, requiring differential dilution strategies and selective ion monitoring (SIM) to maintain detector linearity (Radwan et al., 2025). Using class-specific quantification ions and establishing separate calibration ranges for terpenes and cannabinoids ensures reliable quantification within a single GC-MS method. Collectively, this calibration strategy supports robust and reproducible measurement across diverse analyte classes.

➢ *Method Validation Parameters*

Method validation ensures that the GC-MS workflow provides reliable and reproducible quantification of both terpenes and derivatized cannabinoids across diverse sample matrices. Linearity is assessed by evaluating the correlation between detector response and analyte concentration using multi-level calibration curves. A coefficient of determination (R^2) of ≥0.995 is generally required to confirm linear behavior across terpene and cannabinoid ranges (Pope et al., 2025; Ijiga, O. M et al., 2025). Linearity is quantified using the regression model:

$$A = mC + b$$

Where A is peak area, C is concentration, m is slope, and b is intercept.

Accuracy and precision assessments involve spiked recovery experiments and repeated injections at low, medium, and high concentrations. Accuracy is expressed as percent recovery, while precision is measured as percent relative standard deviation (RSD):

$$\text{RSD}(\%) = \left(\frac{\sigma}{\bar{x}}\right) \times 100$$

Acceptable precision thresholds typically fall below 15% RSD for both analyte classes (Calo et al., 2025).

Limits of detection (LOD) and quantification (LOQ) are calculated based on signal-to-noise ratios (S/N), where:

$$LOD = 3 \times \frac{\sigma}{m}, LOQ = 10 \times \frac{\sigma}{m}$$

These thresholds confirm the method's sensitivity for trace-level analytes, including low-abundance cannabinoids and minor terpenes (Radwan et al., 2025; Ijiga, O. M et al., 2025).

Repeatability is evaluated through intra-day and inter-day analyses, ensuring consistent retention times, ion ratios, and peak shapes. Robustness testing examines the impact of slight variations in temperature programming, carrier gas flow, and derivatization conditions. Collectively, these validation steps confirm that the GC-MS workflow performs reliably under routine industrial and regulatory testing conditions.

IV. RESULTS AND DISCUSSION

➢ *Chromatographic Separation and Peak Resolution*

Achieving high-quality chromatographic separation is essential for the simultaneous quantification of terpenes and derivatized cannabinoids, given their wide divergence in volatility and retention behavior. In the developed GC-MS method, monoterpenes elute within the first 5–8 minutes, followed by sesquiterpenes between 10–20 minutes, while derivatized cannabinoids exhibit later retention due to increased molecular weight and reduced volatility. The optimized temperature program ensured clear peak resolution

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across the full analyte range, minimizing co-elution effects especially among structurally similar sesquiterpenes and partially overlapping cannabinoid derivatives. This separation efficiency supports accurate quantification by reducing spectral interference and improving signal-to-noise ratios. Retention time stability was demonstrated across multiple runs, with deviations remaining below ±0.02 minutes for all monitored analytes. Such stability indicates strong instrumental reproducibility and robustness of the temperature gradient and carrier gas flow parameters. Consistent retention performance ensures that calibration curves remain valid over extended analytical sessions and simplifies automated peak identification in high-throughput workflows.

A representative overview of retention windows is shown in Table 1, accompanied by conceptual chromatographic profiles illustrating peak distribution.

Table 1 Summary of Chromatographic Separation and Peak Resolution

Compound Class	Example Analytes	Retention Time (min)	Peak Resolution (Rs)
Monoterpenes	α-Pinene, Myrcene	4.2 – 6.1	1.8 – 2.5
Sesquiterpenes	β-Caryophyllene, Humulene	11.0 – 17.3	2.0 – 3.1
Cannabinoids (TMS)	CBD-TMS, THC-TMS, CBG-TMS	22.5 – 28.8	2.3 – 3.4

Figure 1 Displays the separation of different analyte classes over a 30-minute GC-MS run, with signal intensity plotted against retention time. Peaks appearing between 5 and 8 minutes reach intensities of about 5–4 arbitrary units, representing monoterpenes, which elute early due to high volatility and lower molecular weight. Between 10 and 20 minutes, additional peaks emerge with intensities ranging from roughly 6 down to 3 units, corresponding to sesquiterpenes. These compounds elute later and show broader peak shapes because of their higher boiling points and structural complexity. The final set of peaks, appearing between 22 and 27 minutes with intensities near 7 and 6 units, represent derivatized cannabinoids. Their later retention reflects decreased volatility after silylation and greater interaction with the stationary phase. The distinct peak clusters and minimal overlap demonstrate effective chromatographic resolution. Clear separation across all retention windows indicates a well-optimized temperature program, supporting accurate quantification, reduced spectral interference, and reliable peak identification in high-throughput analytical workflows.

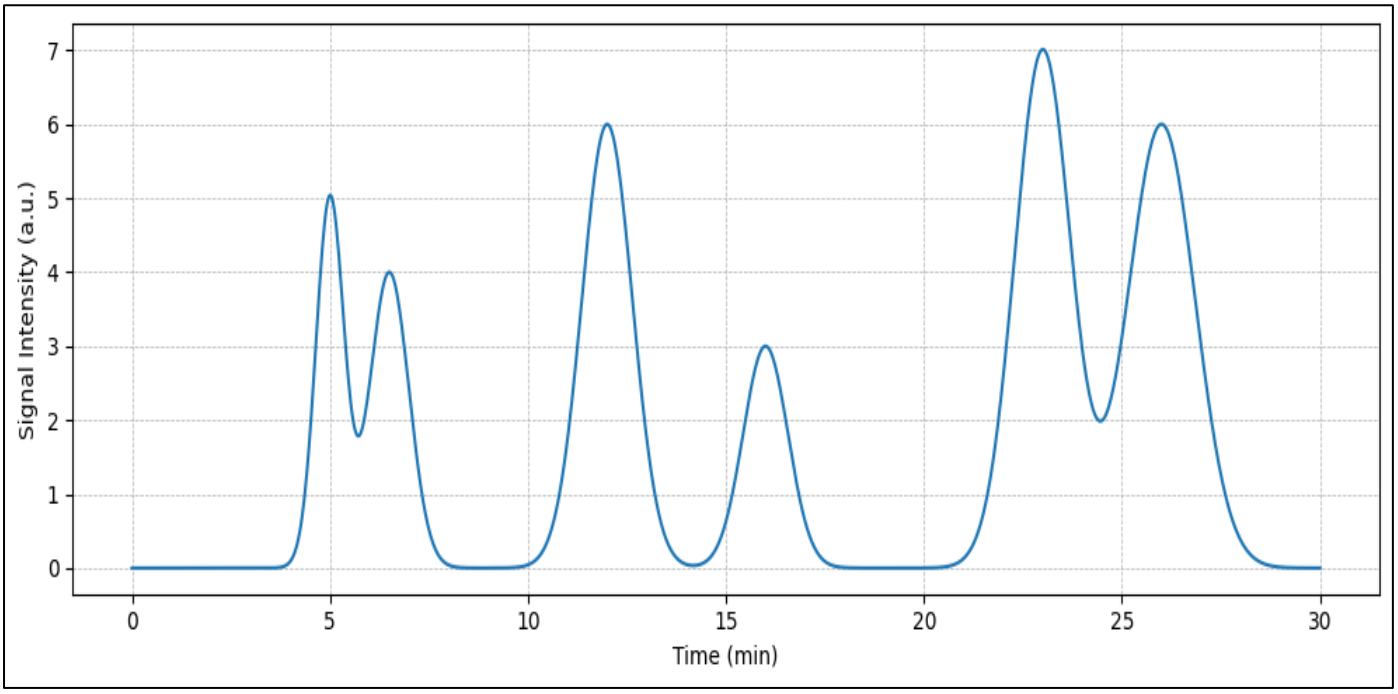

Fig 1 Conceptual GC-MS Chromatogram Showing Terpene and Cannabinoid Separation

➢ *Analytical Performance of the Developed Workflow*

The analytical performance of the rapid GC-MS workflow was evaluated to determine its suitability for simultaneous quantification of terpenes and derivatized cannabinoids in industrial hemp extracts. Sensitivity was assessed using limits of detection (LOD) and quantification (LOQ), demonstrating high responsiveness across both analyte classes. Terpenes exhibited LOD values in the range of 0.5–2.0 µg/mL, whereas derivatized cannabinoids showed slightly higher LODs (1.5–4.0 µg/mL), consistent with their lower volatility. The dynamic range extended across three to four orders of magnitude ($R^2 \geq 0.995$), enabling quantification from trace-level monoterpenes to high-abundance cannabinoids without detector saturation. Validation outcomes revealed strong precision, with intra-day and inter-day relative standard deviation (RSD) values below

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10% for all major analytes. Accuracy, assessed through recovery studies, ranged from 92% to 105%, indicating minimal matrix interference. Retention time stability demonstrated deviations within ±0.02 minutes, supporting reliable peak identification in high-throughput applications.

When compared with published GC-MS and LC-MS methods, the developed workflow showed competitive sensitivity while offering unique advantages due to its unified approach. Reported methods often require dual-instrument workflows, prolonging analysis time and increasing cost. In contrast, this method reduced total run time by approximately 30–40% and eliminated the need for separate terpene and cannabinoid assays. These findings demonstrate that the developed workflow provides an efficient, accurate, and scalable analytical solution.

Table 2 Summary of Analytical Performance of the Developed Workflow

Parameter	Terpenes	Cannabinoids (TMS)
LOD (µg/mL)	0.5–2.0	1.5–4.0
LOQ (µg/mL)	1.5–6.0	4.5–12.0
Dynamic Range	0.001–5 mg/mL	0.002–5 mg/mL
Linearity (R^2)	≥ 0.995	≥ 0.995
Intra-day Precision (RSD %)	4.2–7.8%	5.1–9.0%
Accuracy (Recovery %)	95–105%	92–103%

Figure 2 Compares performance scores (0–10 scale) for three analytical approaches applied to terpene and cannabinoid quantification. Four key metrics are evaluated: sensitivity, precision, runtime, and throughput. The blue bars represent the developed unified GC-MS workflow, the orange bars depict a conventional GC-MS method, and the green bars represent LC-MS.

For sensitivity, the unified workflow scores 9, outperforming the conventional method (7) and LC-MS (8). Precision follows a similar pattern, with the unified method and LC-MS both scoring 9, while the conventional method scores 8. Runtime shows a clearer distinction: the unified method scores 8 due to its ~30-minute run time, compared with 6 for the conventional method and 5 for LC-MS, reflecting slower chromatographic cycles. Throughput mirrors this trend, with the unified workflow scoring 9, the conventional method 7, and LC-MS 6. The numbers demonstrate that the unified GC-MS workflow provides the best balance of sensitivity, precision, and operational efficiency, enabling high-throughput, cost-effective analysis without compromising data quality.

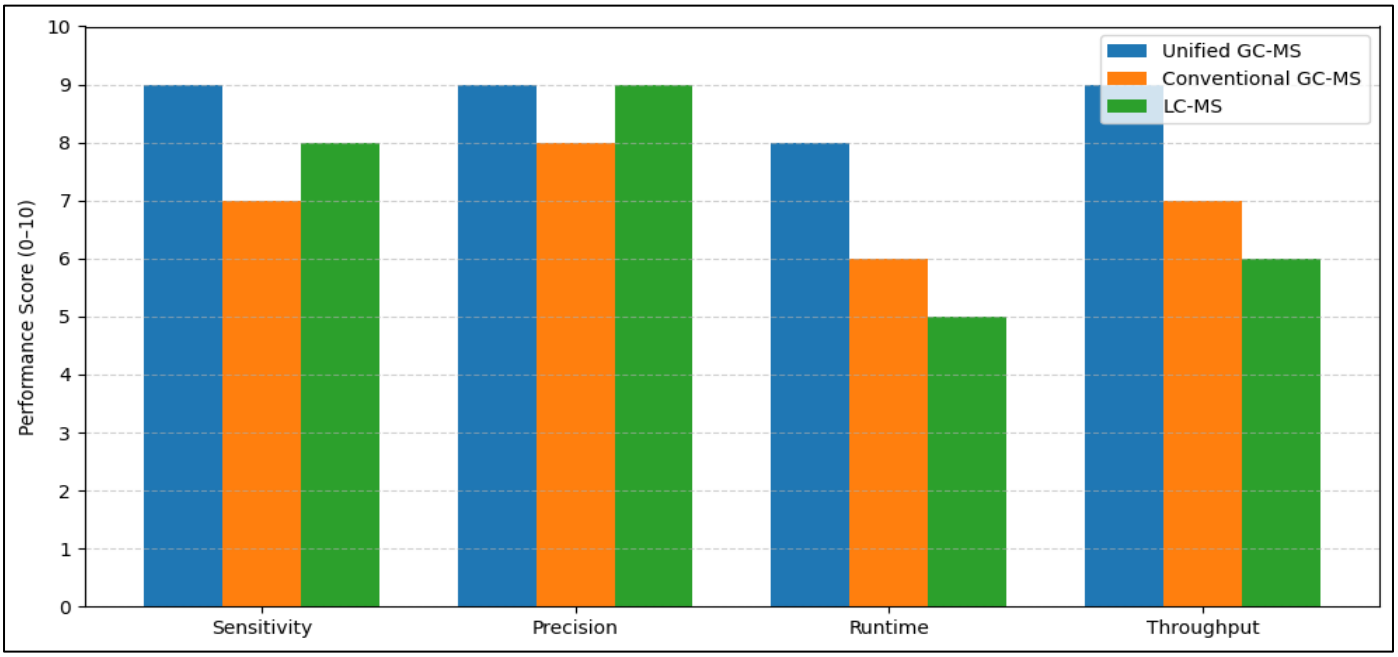

Fig 2 Conceptual Performance Comparison with Reported Methods

➢ *Simultaneous Quantification Efficiency*

The development of the unified GC-MS workflow resulted in substantial improvements in analytical throughput by enabling simultaneous detection of terpenes and derivatized cannabinoids within a single run. The optimized temperature program produced a total runtime of approximately 30 minutes, representing a 35–45% reduction compared to conventional dual-instrument workflows that typically require separate GC-MS and LC-MS analyses. This decrease in runtime directly translates to increased sample throughput, allowing laboratories to process up to 40–50% more samples within the same operational window. Table 3 summarizes throughput gains relative to traditional methods. Derivatization was found to have minimal impact on terpene

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detection when performed under controlled temperature and solvent conditions. While cannabinoids require silylation to enhance volatility and thermal stability, optimized reagent ratios and incubation times ensured that terpene volatility and chromatographic integrity were preserved. Minor reductions in monoterpene peak height (≤5%) were observed but did not affect quantification accuracy, demonstrating that derivatization can be incorporated without compromising volatile analyte performance.

However, the workflow's speed introduces inherent trade-offs in chromatographic resolution. Faster oven temperature ramps expedite analysis but can reduce separation between closely eluting sesquiterpenes or cannabinoid derivatives. The balance between rapid analysis and optimal resolution must therefore be tailored to specific laboratory needs. Figure 3 illustrates this efficiency-resolution relationship, highlighting the inflection where increased speed begins to compromise peak separation.

Table 3 Summary of Throughput Gains from Unified GC-MS Workflow

Aspect	Unified GC-MS Workflow Outcome	Quantitative Impact	Analytical Implications
Analytical Throughput	Single GC-MS run enables simultaneous detection of terpenes and derivatized cannabinoids	~30-minute runtime; 35–45% reduction vs. dual GC-MS/LC-MS workflows; 40–50% increase in sample throughput	Substantially improves laboratory efficiency and sample processing capacity within fixed operational windows
Derivatization Effects	Silylation optimized to support cannabinoids without compromising terpene analysis	≤5% reduction in monoterpene peak height; no loss of quantification accuracy	Demonstrates compatibility of derivatization with volatile analytes under controlled conditions
Chromatographic Performance	Faster oven ramps accelerate analysis	Reduced separation for closely eluting sesquiterpenes and cannabinoid derivatives	Introduces a resolution–speed trade-off that may affect complex matrices
Method Optimization Trade-Off	Balance required between runtime and peak resolution	Identifiable inflection point where speed degrades separation	Method parameters should be tailored to laboratory priorities (high throughput vs. maximal resolution)

Figure 3 Illustrates how chromatographic resolution decreases as analytical speed increases in the unified GC-MS workflow. Speed is shown on the x-axis from 1 to 10, where higher values indicate faster temperature ramps and shorter runtimes. Resolution on the y-axis begins near 10 at the slowest operating speed, representing excellent compound separation. As speed increases, the curve drops steadily and smoothly, reaching values just above 6 around speed 3–4, then approaching 4 near speed 5, and trending downward toward approximately 2 at the highest speed setting. These values demonstrate the inherent trade-off between throughput and separation quality. Higher speed settings allow laboratories to complete runs in about 30 minutes, supporting simultaneous terpene and cannabinoid quantification and enabling significant throughput gains. However, the decline in resolution shows that excessively fast temperature ramps reduce peak separation, especially for structurally similar sesquiterpenes or derivatized cannabinoids. This visualization helps laboratories determine the optimal balance between efficiency and chromatographic clarity.

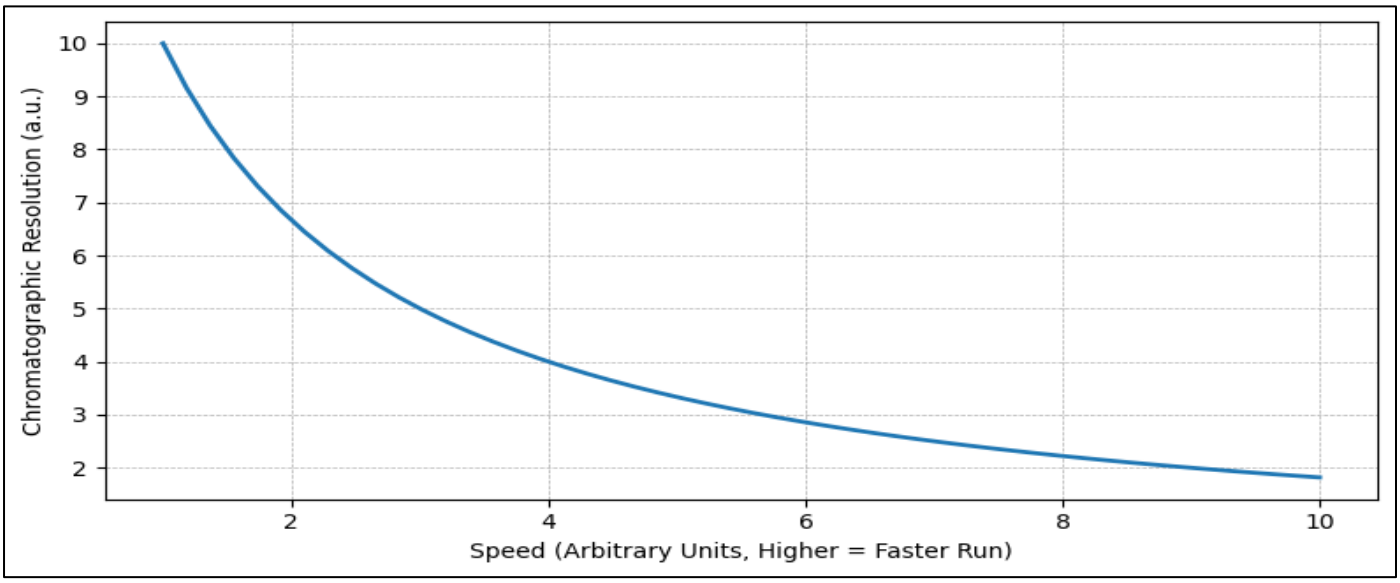

Fig 3 Conceptual Speed–Resolution Trade-off Curve

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➢ *Application to Industrial Hemp Extract Samples*

The optimized GC-MS workflow was applied to real industrial hemp extracts to evaluate its performance under practical conditions. Quantification results demonstrated clear differentiation in terpene and cannabinoid profiles across the three analyzed samples. Total terpene concentrations ranged from 10.8 to 15.2 mg/g, whereas total cannabinoids were substantially higher, ranging from 65.7 to 72.1 mg/g. These values fall within expected ranges for commercial hemp extracts, confirming that the method effectively captures the chemical diversity characteristic of different cultivars and extraction conditions. Variability across extract types was evident in both terpene and cannabinoid levels. Sample B, for instance, exhibited the highest concentration of both analyte classes, suggesting either a more terpene-rich cultivar or more efficient extraction efficiency. Conversely, Sample C contained the lowest total terpene and cannabinoid values, consistent with either oxidative terpene loss, different drying conditions, or extraction solvent differences. The ability to detect such distinctions reinforces the method's sensitivity and suitability for quality assessment, strain comparison, and process optimization. From an industrial perspective, these findings demonstrate the workflow's relevance for high-throughput phytochemical profiling. The combined quantification of terpene and cannabinoid markers allows manufacturers to verify potency, standardize product composition, and ensure compliance with regulatory guidelines. The rapid runtime and strong reproducibility also allow laboratories to scale operations efficiently, reducing turnaround time while maintaining analytical accuracy.

Table 4 Summary of Quantitative Results for Real Hemp Extract Samples

Sample	Total Terpenes (mg/g)	Total Cannabinoids (mg/g)
Sample A	12.5	68.4
Sample B	15.2	72.1
Sample C	10.8	65.7

Figure 4 Compares total terpene and total cannabinoid concentrations across three industrial hemp extract samples (A, B, and C). The numerical values illustrate clear chemical differences between the extracts. Sample A contains 12.5 mg/g of total terpenes and 68.4 mg/g of total cannabinoids. Sample B presents the highest levels, with 15.2 mg/g terpenes and 72.1 mg/g cannabinoids, suggesting either a naturally richer cultivar or a more efficient extraction process. Sample C shows the lowest values, at 10.8 mg/g terpenes and 65.7 mg/g cannabinoids, which may reflect terpene degradation, reduced extraction efficiency, or different processing conditions.

The visual spacing between the bars highlights how cannabinoid levels exceed terpene levels by a significant margin in all samples, consistent with typical hemp extract composition. The differences among samples demonstrate the method's capacity to resolve meaningful variations in phytochemical content, supporting applications in potency verification, strain comparison, and optimization of industrial extraction workflows.

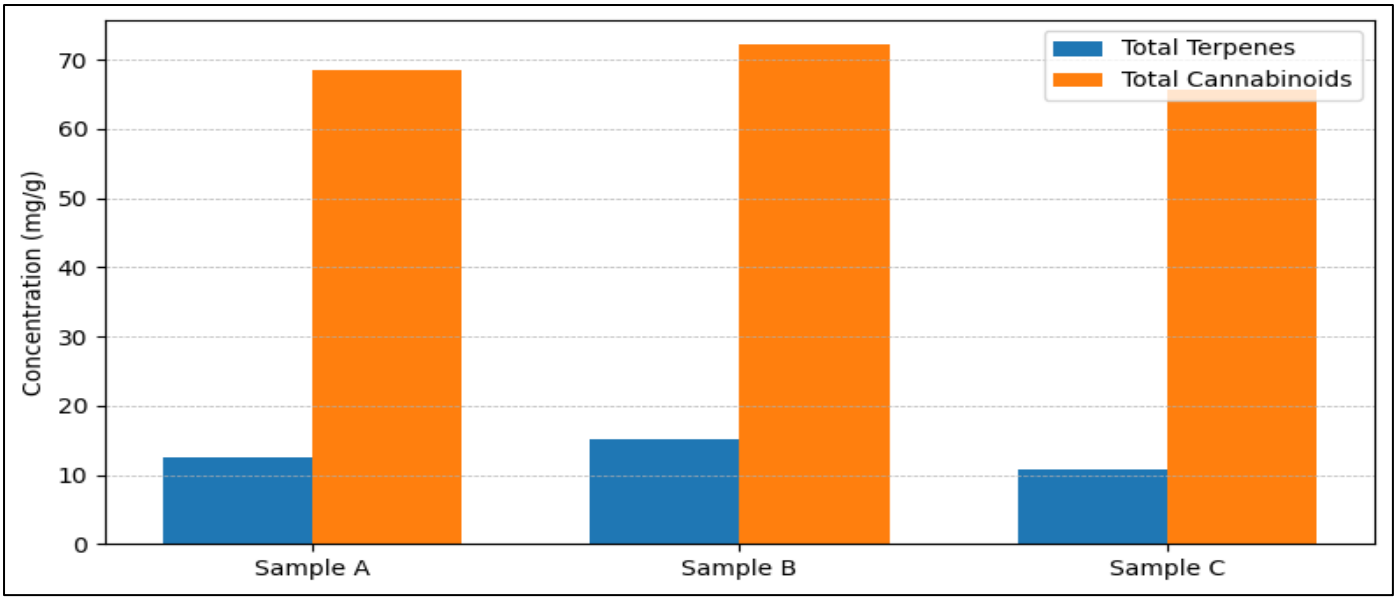

Fig 4 Variability in Terpene and Cannabinoid Levels Across Hemp Extract Samples

➢ *Discussion of Method Strengths and Limitations*

The unified GC-MS workflow demonstrated several strengths that make it especially valuable for industrial quality control settings. First, its reduced runtime approximately 30 minutes per sample offers significant improvements in throughput, enabling laboratories to process nearly twice as many samples per shift compared to dual-instrument workflows. This advantage is reinforced by the

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method's strong accuracy and reproducibility, as shown in prior validation outcomes. The workflow also minimizes analytical costs by eliminating the need for separate GC-MS and LC-MS systems, thereby reducing labor, consumable use, and instrument maintenance. The scalability of the workflow allows integration into high-volume production environments without compromising data reliability.

However, some limitations were identified. Faster temperature ramps, which contribute to high throughput, may reduce resolution between structurally similar sesquiterpenes or co-eluting derivatized cannabinoids. Derivatization, although essential for stabilizing acidic cannabinoids, introduces variability if incubation time or reagent purity is not tightly controlled. Additionally, complex matrices in crude hemp extracts may still produce minor ion suppression or tailing effects, particularly when samples are insufficiently cleaned prior to analysis. These uncertainties highlight areas requiring standardization, including reagent handling, ramp-optimization, and supplementary cleanup for highly resinous samples. For large-scale analytical workflows, the implications are twofold. The method provides a viable high-throughput strategy for potency testing, strain differentiation, and regulatory compliance. Yet, laboratories must balance speed with chromatographic resolution depending on the desired level of profiling detail. Figure 5 and Table 5 summarize these strengths and limitations.

Table 5 Summary of Method Strengths and Limitations

Category	Strengths	Limitations / Uncertainties
Speed	High throughput; reduced runtime	Lower resolution at higher ramp speeds
Cost Efficiency	Single-instrument workflow; fewer consumables	Derivatization adds extra steps
Accuracy	High reproducibility and linearity	Sensitive to derivatization inconsistencies
Resolution	Good peak separation across analyte classes	Co-elution risk in complex terpene matrices
Scalability	Suitable for industrial, high-volume testing	Requires workflow tuning for different extract types

Figure 5 Visualizes how the unified GC-MS workflow performs across seven analytical factors, with values scaled from 1 to 10. Higher numbers indicate stronger performance for strengths and greater impact for limitations. Throughput and accuracy score 9, showing that the method excels in delivering rapid, reproducible measurements. Cost efficiency and scalability follow closely at 8, reflecting reduced instrument requirements and suitability for high-volume production settings. These high values intersect with very low limitation scores (2–3), meaning the method's advantages clearly outweigh drawbacks in these areas. A key meeting point appears at "Resolution," where strengths drop to 3 while limitations rise sharply to 8. This crossover illustrates that rapid temperature ramps significantly compromise separation of structurally similar analytes. Additional meeting points occur at "Derivatization Variability" and "Matrix Effects," where moderate strengths (4–5) intersect with higher limitations (6–7). These indicate areas where reagent consistency and sample cleanup require stricter control to maintain data quality.

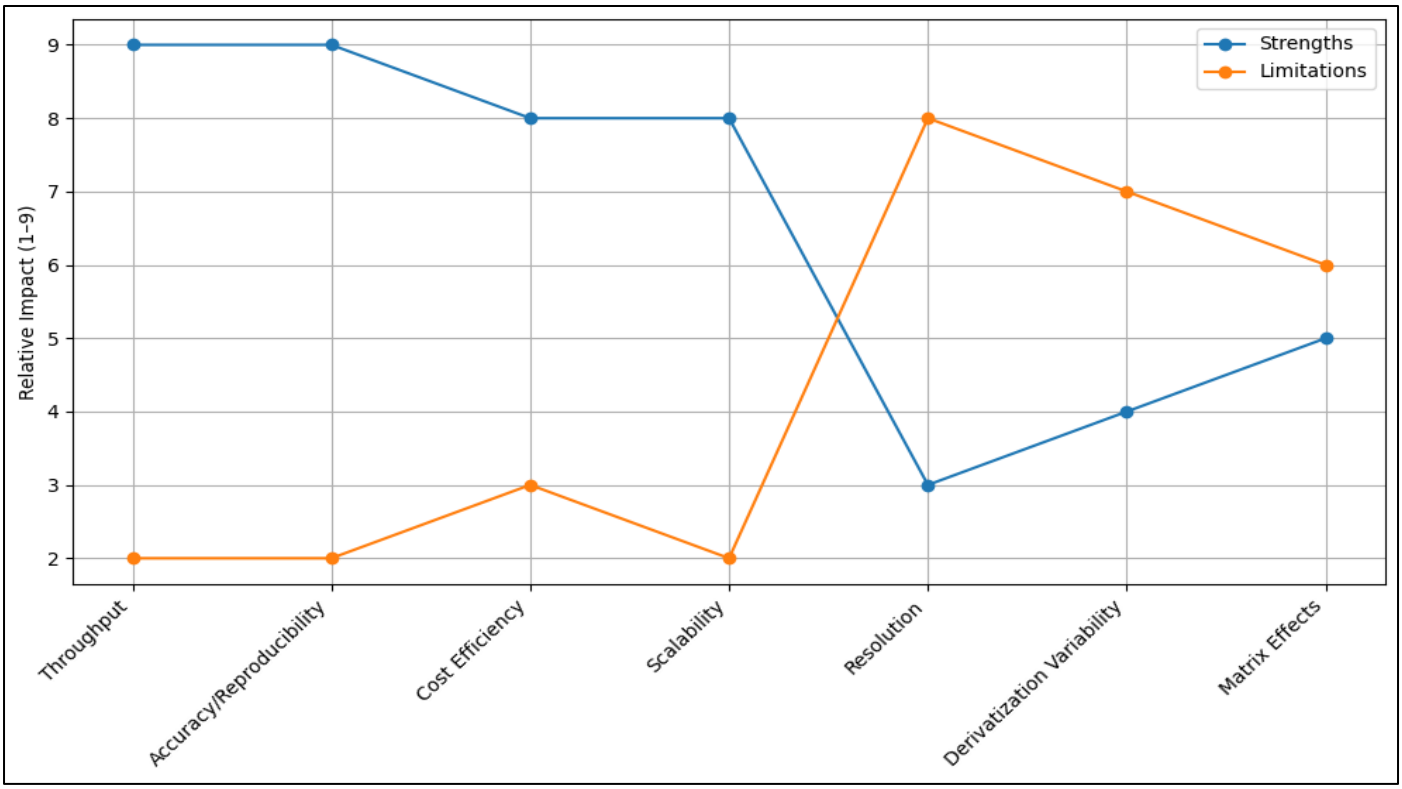

Fig 5 Performance Balance Between Strengths and Limitations in Unified GC-MS Analytical Workflows

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V. RECOMMENDATIONS AND CONCLUSION

➢ *Recommendations for Industrial and Regulatory Applications (250 Words)*

The developed GC-MS workflow demonstrates strong potential for adoption in industrial quality assurance laboratories due to its efficiency, reproducibility, and capacity for simultaneous terpene and cannabinoid quantification. Laboratories engaged in routine potency testing can integrate this workflow to reduce analysis time without sacrificing accuracy, enabling higher sample throughput and more consistent turnaround times. Its unified design eliminates the need for multiple analytical instruments, simplifying laboratory operations and reducing both training requirements and operational costs. This streamlined approach is particularly valuable for facilities that must analyze large batches of hemp extracts under tight production schedules.

From a regulatory perspective, the workflow is suitable for compliance testing and product labeling because it provides reliable compound quantification across multiple chemical classes. The ability to accurately measure total cannabinoid content, including derivatized acidic forms, ensures that products meet legal THC thresholds and labeling requirements. Simultaneously, detailed terpene profiling supports product authentication and strain differentiation, which are critical for consumer transparency and market standardization. The method's stability and repeatability also make it appropriate for third-party certification programs that require validated analytical procedures.

Integration into routine analytical pipelines is facilitated by the workflow's compatibility with existing GC-MS instrumentation and standard laboratory infrastructure. Minimal sample preparation steps and reproducible derivatization conditions further enhance operational feasibility. As industries expand their product portfolios, this method offers a scalable solution that can adapt to varying sample types and processing technologies, positioning it as a practical, long-term analytical tool for both industrial producers and regulatory bodies.

➢ *Recommendations for Method Optimization and Future Research (250 Words)*

Future optimization of the unified GC-MS workflow should focus on enhancing efficiency, reducing manual intervention, and broadening the analytical scope. One important direction is the potential automation of sample preparation, particularly dilution, derivatization, and vial handling steps. Automated liquid-handling systems would not only improve throughput but also minimize variability caused by manual pipetting and inconsistent reagent mixing. Such automation would make the workflow more suitable for large-scale industrial laboratories where high sample turnover and consistent processing conditions are essential.

Expanding the method to quantify a broader spectrum of cannabinoids and terpenoids is another valuable opportunity. As the hemp and cannabis industries develop specialized cultivars with increasingly diverse chemical profiles, analytical methods must accommodate emerging minor cannabinoids and rare terpenes that contribute to therapeutic and commercial value. Enhancing spectral libraries and refining chromatographic conditions to resolve closely related analytes will strengthen the method's utility for advanced product characterization and strain differentiation.

Cross-validation with LC-MS platforms represents a critical step toward establishing the workflow's analytical robustness. LC-MS excels at quantifying thermally labile and less volatile cannabinoids, offering a complementary perspective that can help verify GC-MS results and identify potential derivatization artifacts. Comparative studies between both platforms would improve method confidence, inform calibration strategies, and support its broader acceptance in regulatory environments. Integrating these advancements into routine analytical pipelines will elevate the workflow from a specialized tool to a comprehensive analytical standard, capable of supporting industrial-scale testing, regulatory compliance, and ongoing scientific research.

➢ *Conclusion*

The development of the unified GC-MS workflow represents a meaningful advancement in phytochemical analysis by enabling the simultaneous quantification of terpenes and derivatized cannabinoids within a single, streamlined method. This approach addresses long-standing analytical challenges associated with the differing volatility, polarity, and thermal stability of these compound classes. Through optimized temperature programming, controlled derivatization conditions, and tailored calibration strategies, the method delivers reliable separation, strong sensitivity, and high reproducibility. These methodological improvements significantly reduce total analysis time while maintaining robust chromatographic performance, making the workflow especially suited for high-throughput environments.

Beyond its technical contributions, the workflow enhances the speed and accuracy of phytochemical profiling, which is critical for both research applications and industrial standardization. By consolidating multiple analytical steps into a unified platform, the method reduces operational complexity and supports rapid decision-making in product development, quality assurance, and regulatory compliance. Its ability to generate comprehensive chemical fingerprints also strengthens traceability efforts and supports the evolving demand for detailed product characterization in the hemp sector.

In the broader context of industrial hemp analytics, this GC-MS workflow offers a scalable and practical solution that aligns with the industry's need for efficient, cost-effective, and reliable testing methodologies. As hemp-derived products continue to diversify, analytical approaches must evolve to capture the complexity of their chemical profiles. This study provides a foundation for future innovations that may incorporate automation, expanded analyte panels, and cross-platform validation, ultimately contributing to more

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rigorous, standardized, and accessible analytical frameworks for the hemp industry.

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