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Synthesis and Characterisation of Borate Ester for Brake Fluid Applications

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Abstract

Brake fluids are indispensable for ensuring the safety and reliability of automotive braking systems. Despite the increasing adoption of electric vehicles and regenerative braking technologies, conventional hydraulic brake fluids remain critical due to their ability to deliver rapid response times and minimal stopping distances. Among commercial formulations, glycol- and borate ester based fluids such as DOT4 are widely used; however, there is limited research on the systematic synthesis and detailed characterization of their constituent compounds.

In this study, a borate ester was synthesized through the esterification of boric acid with triethylene glycol monomethyl ether (TEGMME) and subsequently characterized using a suite of physicochemical and spectroscopic techniques. Key performance parameters,including equilibrium reflux boiling point (ERBP), wet ERBP, pH, density, and viscosity,were measured to evaluate the ester's suitability for brake fluid applications. FTIR and GC-MS analyses confirmed the intended molecular structure and highlighted the presence of higher molecular weight species compared to commercial DOT4 fluids, suggesting potential improvements in thermal stability and performance.

The results demonstrate that the synthesized borate ester exhibits excellent thermal stability, favorable rheological properties, and compatibility with established brake fluid specifications. These findings provide valuable insights into the design of advanced brake fluid formulations and establish a foundation for future studies on long-term durability, corrosion resistance, and formulation optimization.

1 Introduction

Brake fluids are a critical component in ensuring the safety and efficiency of automotive braking systems. They are responsible for transmitting force from the driver's input at the pedal to the brake assemblies while maintaining stability under extreme conditions

such as high temperatures, varying pressures, and moisture contamination. Among the various brake fluid classes, glycol-based fluids such as DOT3 and DOT4 remain the most widely adopted due to their effective balance of performance and cost [1]. However, despite their ubiquity, there is limited research detailing the controlled synthesis of their key constituents and the systematic evaluation of their physicochemical properties.

Borate esters, in particular, play a central role in enhancing the thermal stability, boiling point, and overall reliability of DOT4 brake fluids [2]. Their ability to resist vaporization and maintain consistent performance under demanding conditions makes them indispensable in both conventional and high-performance braking applications. Yet, existing literature often focuses on commercial formulations without fully addressing the synthesis process, reaction mechanisms, or the fundamental material properties that underpin their performance.

This study aims to bridge that gap by presenting a detailed synthesis of a borate ester via esterification of boric acid with triethylene glycol monomethyl ether (TEGMME) [2], followed by extensive characterization. Key parameters including boiling point, pH, density, viscosity, FTIR, and GC-MS analyses were investigated to confirm the structure and assess the suitability of the synthesized ester for use in brake fluid applications [3–5]. By systematically analyzing these properties, this work provides valuable insights into the development of safer, more stable, and potentially more sustainable brake fluid formulations.

2 Methods

2.1 Synthesis of Borate Ester

The esterification of boric acid with triethylene glycol monomethyl ether (TEGMME) in stoichiometric amounts was carried out to synthesize the borate ester. This reaction is an equilibrium process, represented by the following equation:

$$\mathrm{B(OH)_3} + 3\,\mathrm{TEGMME} \rightleftharpoons \mathrm{Borate\,Ester} + 3\,\mathrm{H_2O}$$

Table 1: Chemicals and their specifications

Chemical Species	Molecular Weight (g/mol)	Amount used (g)
Boric acid	61.833	6.17
TEGMME	164.20	63.98 (30 wt% excess)
Water	18.0153	5.4
Borate ester	500.3858	50

To drive the esterification reaction forward and maximize the yield of the borate ester, it is essential to continuously remove the water formed during the process. This is accomplished by employing toluene as a dehydrating agent in conjunction with a Dean–Stark apparatus. Toluene forms an azeotrope with water that boils at a lower temperature than pure toluene, enabling selective vaporization of the toluene–water mixture [6]. The vapors are condensed in a vertical condenser and collected in the Dean–Stark trap, where phase separation occurs: the denser water settles at the bottom while the toluene layer overflows and refluxes back into the reaction flask. This continuous removal of water

not only drives the reaction toward ester formation but also maintains efficient refluxing. Careful control of the toluene volume is critical; insufficient toluene reduces the rate of water removal, while excessive amounts result in significant heat consumption for toluene vaporization rather than for sustaining the esterification process. The reaction mixture is maintained at 120 °C to provide the required activation energy. This elevated temperature is necessary because esterification is an endothermic process, and sufficient thermal input is required to promote bond formation and ensure completion of the reaction [7].

The expected reaction mechanism is as given in Figure 1.

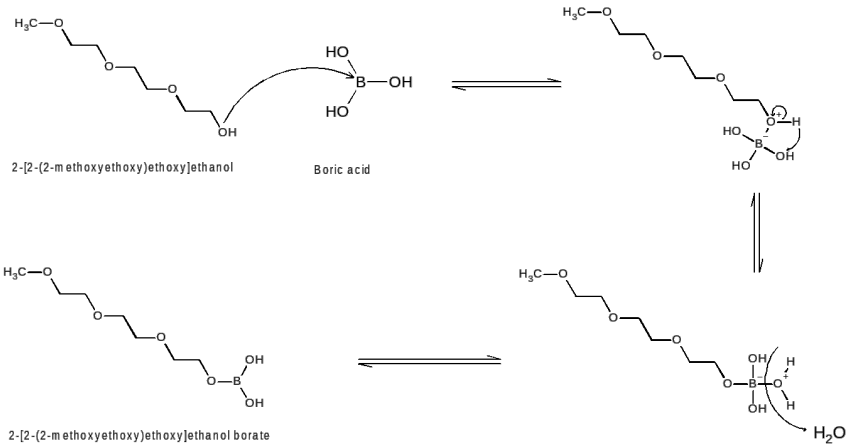


Figure 1: Expected reaction mechanism schematic.

Experimental Optimization and Observations

Several preliminary trials were performed to optimize the synthesis conditions. The first eight samples exhibited deviations due to process inefficiencies and design-related issues, which were gradually rectified.

- **Sample 1:** The experimental setup was adapted from the procedure reported in [8], but the Dean–Stark trap and associated joints were left uninsulated. This led to premature condensation of vapors before they reached the separation arm, causing the entire toluene–water mixture to condense back into the reaction flask. In addition, inadequate temperature control and contamination introduced during charging caused the reaction mixture to darken and thicken into a tar-like mass.

- **Sample 3:** The reaction proceeded as expected, but an excessive quantity of toluene was added. As a result, only toluene was effectively refluxing while the water separation rate remained low. This indicated that the reflux ratio and solvent charge needed optimization to balance vapor–liquid equilibrium and water removal efficiency.

- **Sample 7:** Through successive iterations, it was determined that a relatively small quantity of toluene was sufficient to sustain the azeotropic removal of water. The rate of water collection was found to be limited by the intrinsic rate of its formation rather than solvent quantity. Excess toluene diverted significant heat input toward solvent vaporization instead of promoting the reaction. Continued heating after the stoichiometric amount of water had been collected caused gradual browning of the mixture, indicating possible thermal degradation.

- **Sample 9:** The optimized run was conducted with controlled toluene volume, proper insulation of the Dean–Stark trap, and steady temperature maintenance at 120 °C. Water was efficiently separated, and a clear yellow product was obtained upon completion of solvent removal. This sample was used for subsequent characterization and property evaluation.

Upon completion, the separated water was discarded and the toluene phase decanted from the reaction mixture. The process was then switched from reflux to batch distillation. To prevent thermal degradation of the synthesized borate ester, the still temperature was maintained near the boiling point of toluene, ensuring efficient solvent removal while preserving product stability and integrity.

2.2 Characterisation of Borate Ester

After the synthesis of the borate ester, several techniques were employed to characterise the final product (sample-9). The key properties and measurements for the borate ester are outlined below.

2.2.1 Equilibrium Reflux Boiling Point (ERBP)

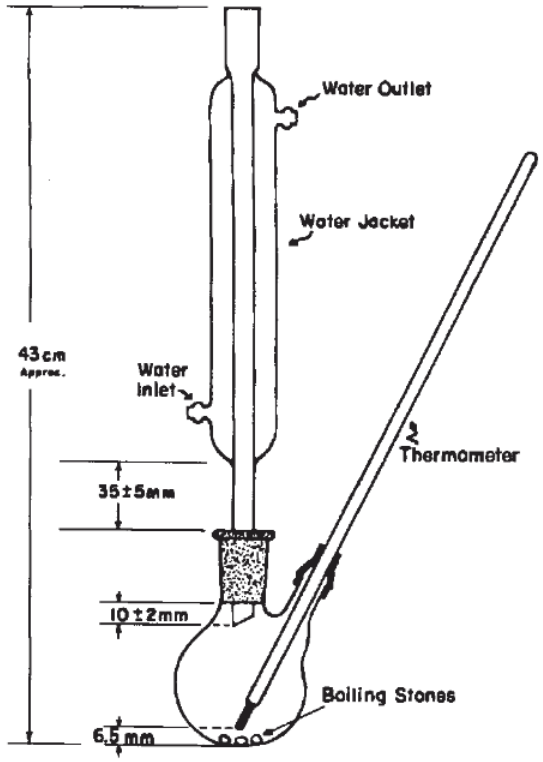
The **Equilibrium Reflux Boiling Point (ERBP)** is a critical parameter for evaluating the thermal stability and high-temperature performance of brake fluids. Because brake fluids are subjected to extreme conditions,such as heavy braking or high-performance driving,the ability to maintain a high boiling point directly impacts braking safety and reliability. ERBP is defined as the temperature at which the rates of vaporization and condensation are equal under atmospheric pressure (1 atm).

Accurate determination of ERBP requires strict adherence to the ASTM D1120 standard, which ensures reproducibility across laboratories. To prevent measurement errors, the brake fluid must be completely dehydrated before testing, as even small amounts of absorbed moisture can lower the boiling point. Proper storage in a dry, cool, and moisture-free environment is therefore essential to preserve fluid integrity prior to evaluation.

During testing, the sample is gradually heated under controlled conditions with continuous monitoring of both temperature and condensation. The ERBP is recorded when the system reaches a steady reflux state, typically indicated by a condensation rate of 1-2 drops per second[9]. At this point, the boiling fluid has reached equilibrium, and the corresponding temperature is defined as the ERBP.

2.2.2 Wet ERBP

The Wet Equilibrium Reflux Boiling Point (Wet ERBP) is a crucial parameter for assessing the performance of brake fluid under conditions that mimic real-world usage, where the fluid may absorb moisture over time. In the case of borate esters, these compounds are inherently hygroscopic, meaning they readily absorb moisture when exposed to air. This absorption of water can significantly impact the thermal stability of the brake fluid, leading to a decrease in its boiling point. As a result, the Wet ERBP is a key measure in determining the fluid's performance under moisture-laden conditions, which could occur during long-term use or when exposed to environmental factors. The measurement procedure for Wet ERBP follows the same standard protocol as the ERBP, as defined by



(a) Schematic representation of the setup.



(b) Experimental setup (ASTM D1120 apparatus).

Figure 2: Setup for ERBP measurement following ASTM D1120.

ASTM D1120, to ensure consistency and reproducibility. However, before beginning the test, the borate ester is intentionally exposed to ambient air for a specified period. This exposure allows the fluid to absorb a controlled amount of moisture from its surroundings, simulating the typical conditions under which brake fluids are likely to operate. The period of exposure is carefully controlled to ensure that the level of moisture absorption is consistent and predictable, allowing for a realistic evaluation of the brake fluid's performance in the presence of water. Once the borate ester has absorbed the moisture, it is tested in the same manner as the ERBP test. The brake fluid is gradually heated under controlled conditions while the temperature is monitored. The Wet ERBP is defined as the temperature at which the rate of condensation stabilizes at approximately 1–2 drops of liquid per second, indicating the fluid has reached a steady-state boiling condition. The temperature at this point is recorded as the Wet ERBP, providing insight into how the presence of water affects the fluid's thermal stability.

2.2.3 pH Measurement

The pH of the borate ester solution was assessed to evaluate its neutrality, which is an important factor in understanding its stability and reactivity in potential applications. The pH of the substance is particularly relevant because it can influence the ester's compatibility with other components in brake fluid formulations and its overall performance. A neutral to basic pH in brake fluid helps ensure the longevity of the storage vessel, as it reduces the risk of corrosion over time, making the fluid more compatible with materials used in the braking system. Given that the borate ester is an anhydrous

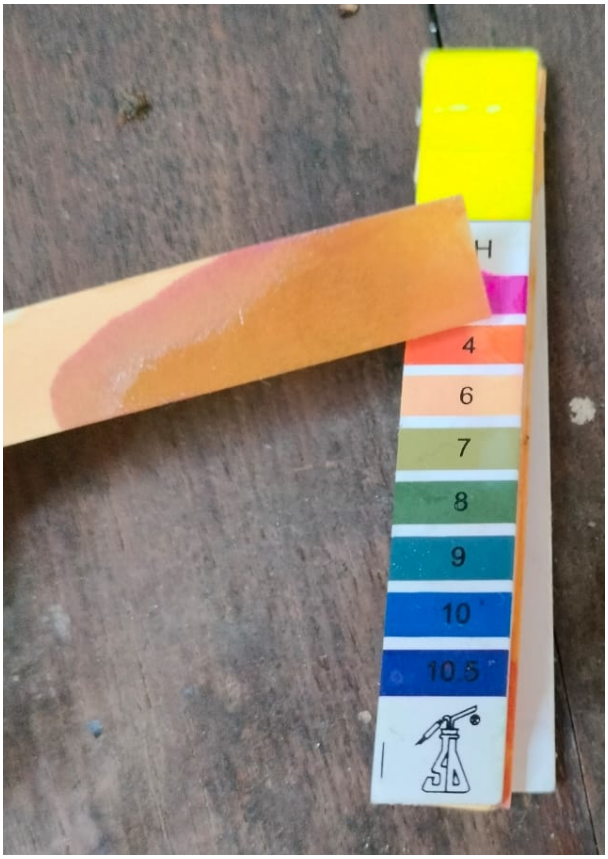


Figure 3: Qualitative measurement of pH using a universal pH paper, along with reference.

compound and contains no water, tra- ditional pH measurement methods—such as those using standard pH meters—are not suitable. Water is a necessary component for the electrodes in conventional pH meters to function correctly. Therefore, alternative methods were employed for this analysis. Universal pH indicator strips were used as a crude but effective solution, providing an estimation of the pH value based on the color change observed when the strips were exposed to the ester solution. It is important to note that the product being tested is the borate ester precursor, not the final brake fluid. As such, its pH characteristics may differ from those of commercial brake fluids. Brake fluids typically exhibit a slightly basic pH, but the borate ester in its unmodified form may not reflect the same properties. The pH of the borate ester solution can thus vary depending on the specific formulation and the moisture content, but it is generally mild in nature. Borate esters are commonly associated with mildly acidic or neutral properties, which is consistent with their behavior in other industrial and chemical applications.

2.2.4 Density

The density of the synthesized borate ester was determined using a calibrated glass pycnometer following the procedure described in ASTM D891 [10] and the method of Singh [11]. Measurements were carried out at a controlled temperature of 25.4 °C using a thermostatic water bath with a stability of 0.1 °C. Prior to use, the pycnometer was thoroughly cleaned with detergent, rinsed with deionized water, finally rinsed with acetone, and dried in a dust-free environment. The empty pycnometer and stopper were weighed

on an analytical balance with a readability of 0.1 mg, and the mass was recorded as M_0.

The pycnometer was then filled with deionized water previously equilibrated to 25.4 °C, ensuring that no air bubbles were trapped. After thermal equilibration, the filled pycnometer was weighed and recorded as M_{H_2O}. The effective volume of the pycnometer, V, was determined using the known density of water at 25.4 °C ($\rho_{H_2O} = 0.99650$ g cm^{-3}) according to Eq. (1).

$$V = \frac{M_{H_2O} - M_0}{\rho_{H_2O}} \tag{1}$$

After calibration, the pycnometer was emptied, dried, and refilled with the borate ester sample, carefully avoiding the inclusion of air bubbles. The filled pycnometer was re-equilibrated at 25.4 °C, and the total mass was recorded as M_s. The density of the sample was then calculated using Eq. (2):

$$\rho_{sample} = \frac{M_s - M_0}{V} \tag{2}$$

Each measurement was repeated in triplicate, and the mean value along with the standard deviation was reported. Buoyancy correction and thermal equilibrium were ensured according to the recommendations in ASTM D891. The pycnometer volume was periodically verified with deionized water to confirm calibration stability.

2.2.5 Viscosity

The viscosity of the borate ester was measured using a Brookfield Ametek DV2T viscometer at a controlled temperature of 25.2C. To ensure accuracy and consistency, the sample was allowed to equilibrate at this temperature before measurements were taken. Viscosity is a key property that influences the handling and flow behavior of the ester, making it a critical factor in evaluating its suitability for applications, particularly in formulations like brake fluids where flow characteristics under standard operating conditions are essential. The measurement process was conducted to ensure that the results were reliable and reproducible. Understanding the viscosity also provides insights into the ester's performance in real-world conditions, such as its ability to maintain consistent flow in braking systems over a range of temperatures.

2.2.6 FTIR and GC-MS

To confirm the structure and composition of the synthesized borate ester, FTIR and GC-MS analyses were conducted on the sample following the removal of toluene. Additionally, theoretical IR spectra were simulated using Gaussian 16 software [12, 13], employing the B3LYP level of theory with the 6-31++G(d,p) basis set. Both geometry optimization and frequency calculations were carried out using the same level of theory and basis set to ensure accurate vibrational spectra. This computational approach enabled a detailed comparison with the experimental data, supporting the identification of functional groups and validating the proposed molecular structure.

Table 2: Key physical and chemical properties of the synthesized borate ester.

Property	Measured Value	DOT4[14, 15]
ERBP	260 °C	246 °C
Wet ERBP	156 °C	160 °C
pH	4-6	8
Density	1.0915 g/cc @ 25.4 °C	1.1 g/cc @ 20 °C
Viscosity	12 cP @ 25.2 °C	16-18 cP @ 20 °C[15]

3 Results and Discussion

3.1 Characterisation of Borate Ester

3.1.1 Equilibrium Reflux Boiling Point (ERBP)

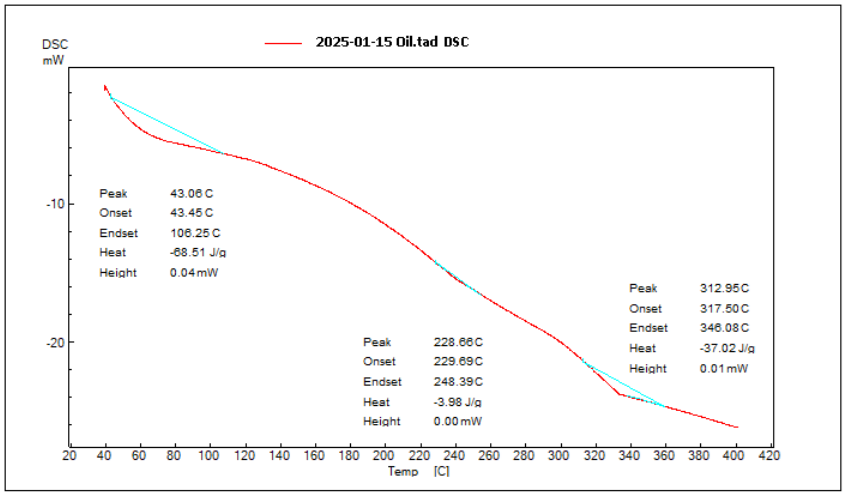


Figure 4: Differential Scanning Calorimetry (DSC) profile of the synthesized borate ester, showing the absence of thermal transitions in the operational range. Minor peaks at 43 °C and 228 °C correspond to residual toluene and unreacted TEGMME, respectively.

Equilibrium Reflux Boiling Point (ERBP) measurements were carried out, yielding a value of **260 °C** for the synthesized brake fluid across multiple trials. Differential Scanning Calorimetry (DSC) (Figure 4) determined the boiling point of the synthesized borate ester mixture to be **312.95 °C**.The mixture is easily comparable to the commercial brake fluid as the chemical constituents in both are largely the same. Such a high boiling point is considered excellent for brake fluid applications, as it reflects strong thermal stability,an essential requirement for fluids exposed to elevated temperatures in braking systems.

Minor peaks were also observed at approximately 43 °C and 228 °C, which are attributed to the boiling of other components present in the reaction product mixture, such as residual toluene, unreacted TEGMME, or partially formed borate esters. The absence of additional thermal transitions in the relevant working range underscores the thermal stability of the ester under practical operating conditions.

The elevated boiling point of the borate ester can be attributed to its high molecular weight, which enhances its resistance to evaporation and thermal degradation. These

properties are advantageous, ensuring the brake fluid maintains its effectiveness under extreme thermal stresses without undergoing phase changes. Furthermore, the high boiling point minimizes the likelihood of vapor formation during braking, thereby reducing the risk of vapor lock and contributing to the reliable performance of the braking system.

3.1.2 Wet ERBP

Wet Equilibrium Reflux Boiling Point (Wet ERBP) measurements were carried out with the borate ester sample pre-exposed to ambient air to allow moisture absorption. This simulates real-world conditions where brake fluids are susceptible to water contamination over time due to their hygroscopic nature.

The Wet ERBP was determined to be **156 °C**, which, although lower than the dry ERBP, remains within an acceptable range for brake fluid applications. The reduction in boiling point highlights the impact of moisture uptake on thermal performance, a critical consideration for long-term storage and use.

Despite this expected decrease, the Wet ERBP of the borate ester is still relatively high compared to conventional glycol-based brake fluids, suggesting that it retains a strong thermal profile even in the presence of absorbed water. This behavior can be attributed to the molecular structure of the ester, which provides resistance against drastic thermal degradation despite hydration.

3.1.3 pH Measurement

The pH of the synthesized borate ester was measured to be in the range **4-6**. This slightly acidic value is consistent with the expected chemical nature of borate esters, which often display mild acidity due to the presence of residual hydroxyl groups or boric acid-like functionalities.

While a pH of 6.5 lies below the slightly basic range generally preferred for commercial brake fluids, this observation is neither unexpected nor problematic. The measurement here pertains to the isolated borate ester component rather than a fully formulated brake fluid. In practical formulations, the ester typically accounts for only 30-50% of the total composition [16], with the remaining constituents,commonly glycols or glycol ethers,adjusting the overall pH toward neutral or slightly basic values.

Accordingly, the measured pH is considered acceptable for the borate ester and does not present a limitation in its role as a precursor for brake fluid formulations.

3.1.4 Density

The density of the synthesized borate ester was measured to be **1091.5 kg/m^3 at 25.4 °C**, with a moisture content of **2.0925%** in the liquid during testing. This value falls within the range typically reported for commercial brake fluids, highlighting the compatibility of the synthesized ester with existing formulations. Such consistency is encouraging, as it indicates that the borate ester can be incorporated into brake fluid systems without significant deviation from established specifications.

3.1.5 Viscosity

The viscosity of the synthesized borate ester was measured to be **12 cP at 25.2 °C**, with a moisture content of **2.0925%** in the liquid during testing. This value lies within

the range typically observed for conventional brake fluid components, underscoring the suitability of the ester for such applications. Viscosity is a critical parameter, as it governs the fluid's flow behavior and overall performance under varying thermal and mechanical conditions. The comparable viscosity of the synthesized ester indicates that it can be incorporated into brake fluid formulations without necessitating significant rheological modifications.

3.1.6 IR spectrum

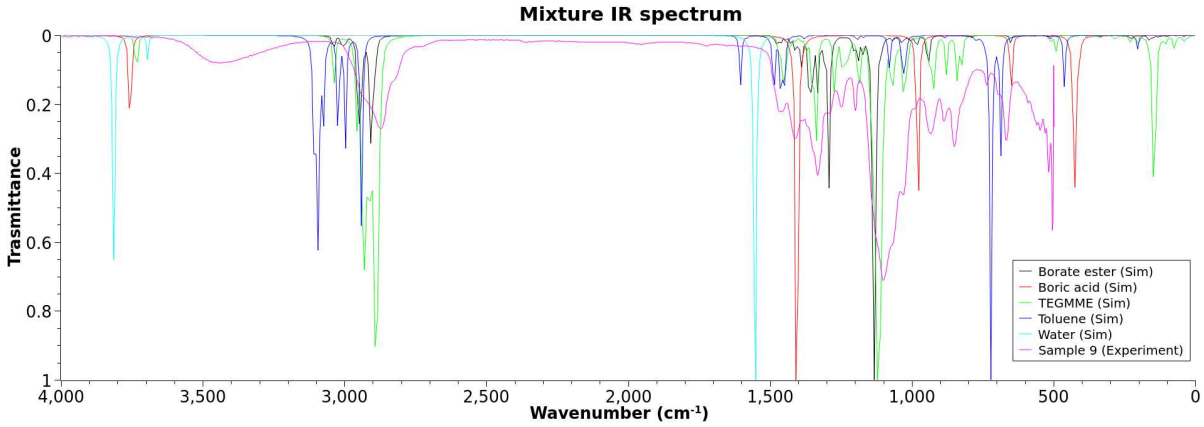


Figure 5: FTIR spectra of the synthesized brake fluid (Sample 9, experimental) compared with simulated spectra of expected constituents. All simulated IR data have been scaled by 0.97.

The FTIR spectrum of the synthesized brake fluid (Sample 9) was compared with simulated spectra of the expected constituents (borate ester, boric acid, TEGMME, toluene, water). The IR spectrum shown here is of the synthesized sample and the comparison is between experiment and simulation. Major absorption bands are summarized in Table 3.

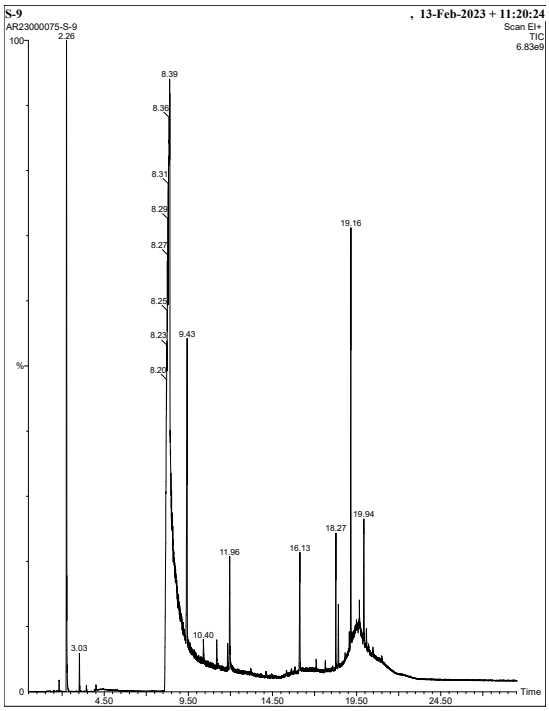
The simulated spectra, scaled by 0.97 as is commonly done to reconcile theoretical and experimental frequencies, align closely with the experimental data [17]. The major absorption bands show excellent matching in both position and relative intensity, with no missing functional group signatures. This high degree of agreement provides strong evidence that the synthesized brake fluid contains the intended borate ester and solvent environments, and that the synthesis successfully reproduced the expected bonding motifs. Minor deviations between experiment and simulation are attributable to the limitations of the chosen basis set and the use of purely harmonic vibrational calculations.

3.1.7 Gas Chromatography-Mass Spectroscopy

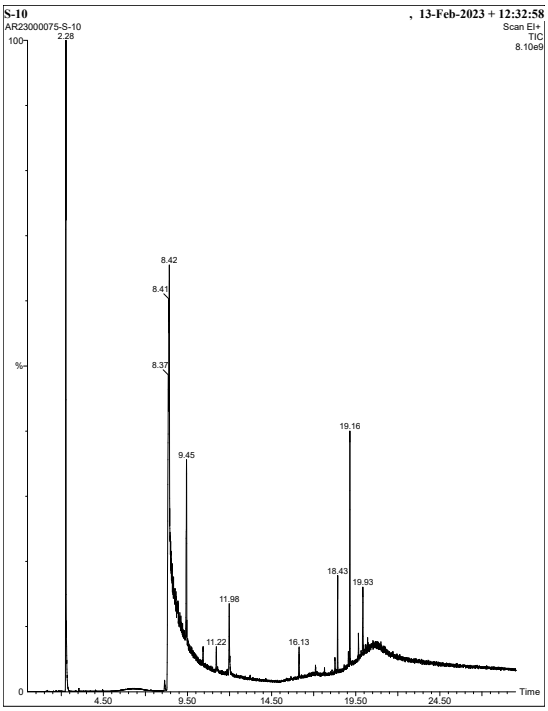
GC-MS analysis was performed on both the commercial SHELL DOT4 brake fluid and the synthesized brake fluid. The total ion chromatograms (TICs) for both samples are presented in Figure 6. Two runs of the synthesized brake fluid are shown alongside the TIC for the commercial fluid.

The mass spectrum of the SHELL DOT4 brake fluid (Figure 7) was analyzed using the NIST library. The major components identified include:

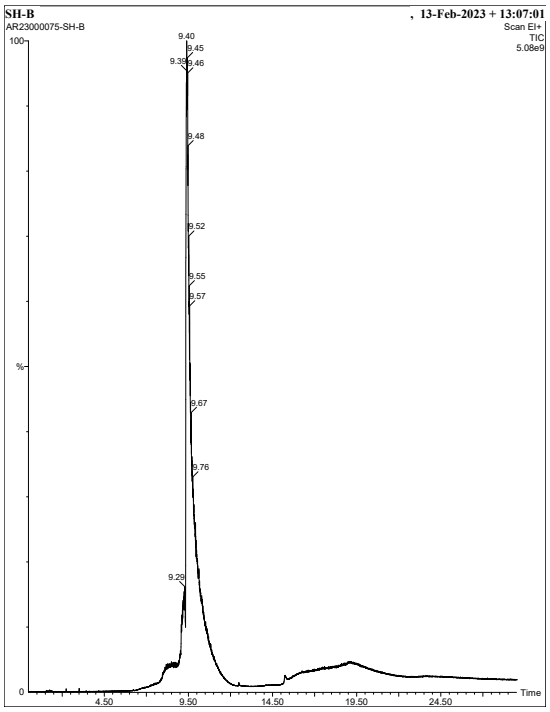
- Ethanol, 2-[2-(2-ethoxyethoxy)ethoxy]- (C8H18O4; probability 87.1%)



(a) Synthesized brake fluid (run 9)



(b) Synthesized brake fluid (run 10)



(c) SHELL DOT4 brake fluid

Figure 6: Total Ion Chromatograms (TICs) for the synthesized brake fluid (two runs) and commercial SHELL DOT4 brake fluid.

Table 3: Major FTIR absorption peaks of synthesized brake fluid (Sample 9) with best-matching assignments relevant to TEGMME, toluene, water, boric acid and borate esters.

Wavenumber (cm^{-1})	**Assignment**	**Likely Source**
3433	O–H stretching (hydrogen-bonded)	Water, boric acid
2872	C–H stretching (alkane/ether backbone); possible O–H broad contribution	TEGMME, residual alcohols
1457	C–H bending	Alkyl groups in TEGMME / toluene
1411	O–H bending; C–H bending	Water/boric acid, alkyl groups
1331	B–O stretching; C–O stretching contributions possible	Borate ester
1247	C–O stretching (alkyl aryl ether / ester)	TEGMME, borate ester
1199	C–O stretching (alkyl aryl ether / ester)	TEGMME, borate ester
1100	C–O stretching (aliphatic/ether/alcohol)	TEGMME, borate ester
866	Aromatic C–H bending (out-of-plane)	Toluene ring
849	Aromatic C–H bending (out-of-plane)	Toluene ring
734	Aromatic C–H bending (mono-substituted/disubstituted benzene)	Toluene ring
666	Aromatic C–H bending / B–O deformation	Toluene ring, borate framework

- Triethylene glycol monomethyl ether (C10H22O5; probability 8.53%)
- Additional long-chain glycol ethers

These results align with the known composition of commercial brake fluids, which typically contain a mixture of short- and long-chain glycol ethers, including diethylene glycol monomethyl ether (DEGMME), triethylene glycol monomethyl ether (TEGMME), diethylene glycol dimethyl ether (DEGDME), and tetraethylene glycol dimethyl ether (TEGDME) [16]. These components contribute to the fluid's desired viscosity, thermal stability, and low volatility.

The TICs of the synthesized brake fluid show numerous peaks compared to the commercial SHELL DOT4 fluid. This is expected, as the reaction mixture may contain residual impurities from the reactants, residual water, and traces of toluene. The early peaks at 2.2 and 3.0 min correspond to toluene, consistent with incomplete solvent removal. Peaks between 8.3 and 9.4 min indicate the presence of small-chain glycol ethers used in the reaction, such as:

- 2.2 min: Toluene, **C7H8**

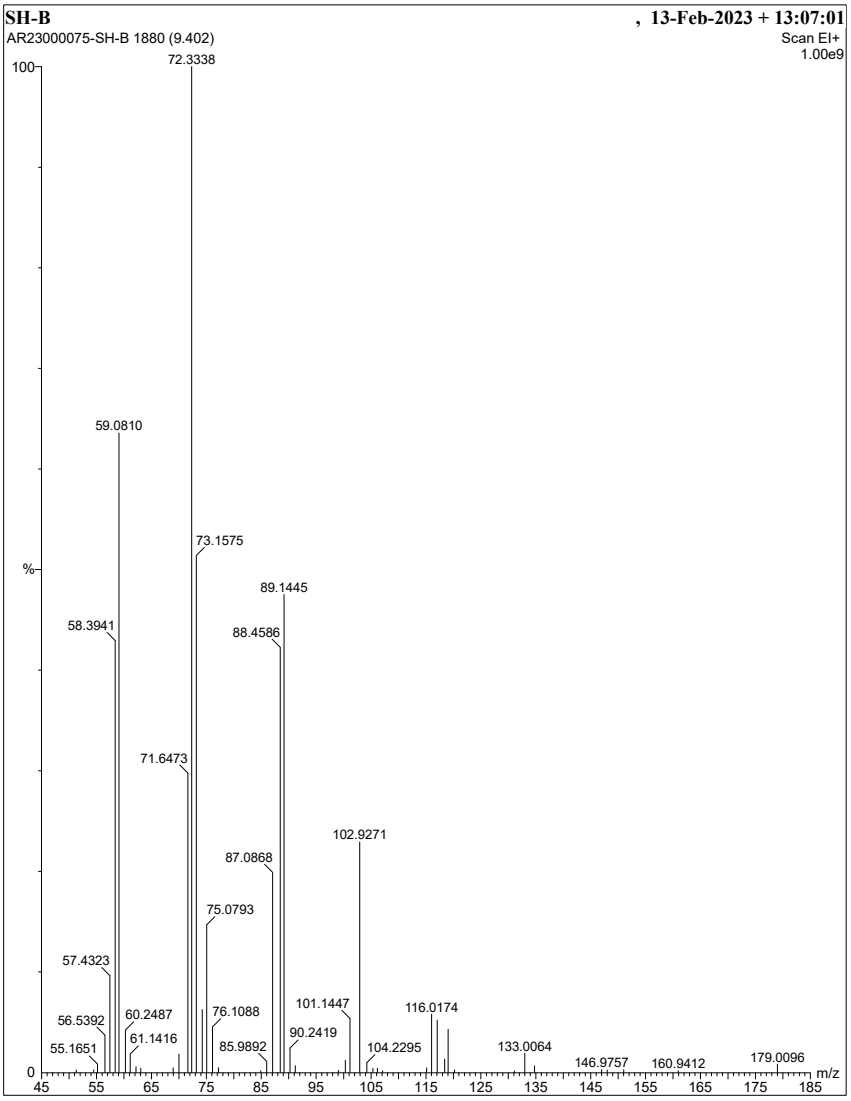


Figure 7: Mass spectrum of SHELL DOT4 brake fluid used for compound identification and NIST library matching.

- 3.0 min: Toluene, **C7H8**
- 8.3 min: Methoxytriethylene glycol, **C7H16O4**
- 8.3 min: Tetraethylene glycol monomethylether, **C9H20O5**
- 9.4 min: Methoxytriethylene glycol, **C7H16O4**
- 9.4 min: 2,5,8,11,14-Pentaoxahexadecan-16-ol, **C11H24O6**

Peaks at 10.4, 11.6, and 16.13 min may correspond to sulfur-containing impurities from the toluene, which is a common occurrence in commercial-grade solvents. Peaks from 18.2 to 20 min correspond to higher molecular weight, glycol ether species. Notably, the peak at 19.9 min corresponds to:

- 19.9 min: 2-(Nonanoyloxy)propane-1,3-diyl dioctanoate, **C28H52O6; MW: 484.38 g/mol**

This molecular weight (484.38 g/mol) is close to the expected molar mass of the borate ester (500.3858 g/mol), indicating successful synthesis. Compared to SHELL DOT4, the synthesized brake fluid contains higher molecular weight components, where the maximum molecular weight in the commercial fluid is 252.16 g/mol. This suggests that the synthesized DOT4 fluid is enriched in high-molecular-weight species, indicative of a potentially superior formulation.

4 Conclusion

This study successfully demonstrated the synthesis and characterization of a borate ester suitable for DOT4 brake fluid applications. The synthesized compound exhibited a high equilibrium reflux boiling point of 312.95 °C, confirming excellent thermal stability, while the wet ERBP remained within an acceptable range despite moisture absorption,an expected outcome for hygroscopic fluids. Supporting measurements of pH (≈6.5), density (1.0915 g/cc), and viscosity (12 cP) aligned well with values reported for commercial brake fluids, reinforcing the compatibility of the synthesized ester with existing formulations.

Structural validation through FTIR and GC-MS further confirmed the intended borate ester framework, with GC-MS identifying molecular species close to the theoretical molar mass of the target compound. The presence of higher molecular weight components compared to commercial samples suggests potential advantages in stability and performance, though optimization of synthesis conditions could reduce residual impurities.

Overall, the findings highlight the feasibility of producing high-quality borate esters through straightforward synthesis routes while providing a foundation for further refinement of brake fluid formulations. Future work should focus on long-term stability testing, corrosion resistance evaluations, and formulation blending to ensure comprehensive performance in real-world braking systems.

5 Supplementary Data

All supporting data, including FTIR and GC–MS spectra, as well as additional experimental observations and characterization results, are available in the project's public repository at: `Github`.

The repository contains raw and processed spectral files, instrument output data, and supplementary notes related to synthesis and analysis.

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