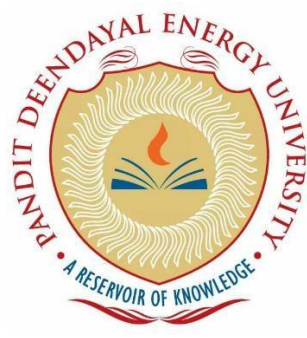


Synthesis And Characterization Of Tetrabromobisphenol A (TBBPA)

Report submitted in partial fulfillment of the requirements for the B. Sc. Hons. Degree in Chemistry

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May 2023

Student Declaration

I, **ARYAN SOMANI** hereby declare that this written submission represents my ideas in my own words and where others' idea or words have been included, I have adequately cited and referenced the original sources. I also declare that I have adhered to all principles of academic honesty and integrity and have not misrepresented or fabricated or falsified any idea / data / fact / source in my submission. I understand that any violation of the above will be cause for disciplinary action by the PANDIT DEENDAYAL ENERGY UNIVERSITY and can also evoke penal action from the sources which have thus not been properly cited or from whom proper permission has not been taken when needed.

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UNDERTAKING

I submit that the edifice of this dissertation research report on "**Synthesis and Characterization of TBBPA**" is built and further developed the framework of extensive and critically focused literature survey with sources listed in the bibliography. In addition, the recourse has also been made to large number of references based on critical analysis of experts, special reports in journals, articles and research papers of journals and magazines, internet, newspapers, reports of government agencies, conceptual anchoring attributable to standard works including books and the vast treasure of secondary information. They have been indexed at the end of this dissertation research report.

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Date: 18/05/2023

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List of Abbreviations

TBBPA= Tetrabromobisphenol A

BPA= Bisphenol A

EDC= Ethylene dichloride

MCB= Monochlorobenzene

H₂O₂= Hydrogen Peroxide

BFR= Brominated flame retardants

ROS= Route of synthesis

Chapter – 1 Introduction

1.1 Introduction of TBBPA

Tetrabromobisphenol A (TBBPA) is a widely used flame retardant that has gained significant attention due to its extensive application in various industries. It belongs to the family of brominated flame retardants (BFRs) and is known for its high efficiency in preventing the spread of fires by inhibiting the ignition and combustion of materials. TBBPA is primarily used in electronic devices, such as printed circuit boards, electrical connectors, and plastic casings, where its flame retardant properties are crucial for ensuring safety. Its effectiveness lies in its ability to reduce the flammability of materials without negatively affecting their mechanical and electrical properties.

However, the widespread usage of TBBPA has raised concerns about its potential adverse effects on both human health and the environment. Studies have shown that TBBPA can persist in the environment and bioaccumulate in organisms, leading to potential long-term impacts on ecosystems. Furthermore, there is evidence suggesting that TBBPA may have endocrinedisrupting properties, which can disrupt hormonal balance and have implications for human health.

Given these concerns, there is a growing need for comprehensive research on TBBPA, including its occurrence, fate, and potential risks. Analytical methods have been developed to detect and quantify TBBPA in various environmental matrices, such as water, soil, sediment, and biota. These methods play a vital role in assessing the levels of TBBPA contamination and monitoring its distribution and behavior in different ecosystems.

In addition to TBBPA itself, the production and use of TBBPA derivatives, such as tetrabromobisphenol S (TBBPS), have gained attention as potential alternatives. These derivatives may also accumulate in the environment and raise similar concerns, necessitating the development of analytical techniques capable of identifying and characterizing these compounds.

This review aims to provide an overview of the analytical methods used for the detection and analysis of TBBPA and its derivatives. It also explores the identification of metabolites derived from these compounds, shedding light on the transformation products that may occur in environmental matrices. By highlighting recent developments in analytical approaches, this review contributes to the ongoing research on TBBPA and its derivatives, offering insights into their environmental occurrence and potential impacts.

TBBPA is commonly employed in electronic applications, such as printed circuit boards (PCBs), electrical connectors, and plastic housings, where its flame retardant properties are essential for preventing the rapid spread of fires. By impeding the ignition and combustion of materials, TBBPA effectively enhances the fire resistance of these products without significantly compromising their mechanical or electrical performance.

However, the extensive use of TBBPA has raised concerns about its potential adverse effects on human health and the environment. These concerns have prompted extensive research and regulatory scrutiny. Here are some key aspects and areas of interest regarding TBBPA:

Environmental Persistence and Accumulation: TBBPA has been found to persist in the environment due to its resistance to degradation processes. It has been detected in various environmental compartments, including water, soil, sediment, and biota. The accumulation of TBBPA in organisms can occur through bioaccumulation and biomagnification processes, potentially leading to ecological impacts.

Ecotoxicity and Health Risks: TBBPA has been associated with various adverse effects on aquatic organisms, including developmental and reproductive toxicity, endocrine disruption, and impacts on the immune system. It has also been found to exhibit cytotoxicity and genotoxicity in mammalian cells. Furthermore, there are concerns about potential human health risks, particularly regarding its endocrine-disrupting properties and possible links to hormonal imbalances, developmental disorders, and certain cancers. However, more research is needed to fully understand the extent of these risks.

Environmental Fate and Transformation: TBBPA can undergo various transformation processes in the environment, leading to the formation of degradation products and metabolites. These transformation products may have different properties and environmental behaviors compared to the parent compound, and their identification and characterization are crucial for assessing the overall environmental impact of TBBPA.

Analytical Methods: Analytical techniques play a vital role in the detection, quantification, and characterization of TBBPA in environmental samples. These methods include chromatographic techniques coupled with mass spectrometry (GC-MS, LC-MS), as well as immunoassays and other screening methods. Ongoing research focuses on developing more sensitive and selective analytical methods to accurately measure TBBPA and its derivatives in complex environmental matrices.

Regulation and Risk Management: Due to the potential environmental and health risks associated with TBBPA, regulatory measures have been implemented to restrict its use and mitigate its impacts. These measures include product labeling requirements, phase-out initiatives, and regulations on the disposal and recycling of electronic waste containing TBBPA.

Tetrabromobisphenol A (TBBPA) was first synthesized in the late 1950s as a flame retardant for use in the plastics industry. It was commercialized in the 1970s and quickly gained popularity due to its effectiveness in reducing the flammability of a wide range of materials.

The production of TBBPA involves the reaction of bisphenol A (BPA) with bromine in the presence of a catalyst. The reaction results in the formation of TBBPA, which is a white crystalline powder with a high melting point and good thermal stability.

In the early years of TBBPA production, the process used elemental bromine as the source of bromine. However, this method was later replaced by the use of brominated flame retardant compounds such as hexabromocyclododecane and polybrominated diphenyl ethers as a more cost-effective and environmentally friendly alternative.

Today, TBBPA is primarily produced in Asia, particularly in China and Japan, which account for the majority of global production. The largest applications of TBBPA are in electronic devices, such as PCBs and plastic housings, as well as in construction materials, such as insulation foams and coatings.

Despite its widespread use, the production and use of TBBPA have raised concerns regarding its potential adverse impacts on human health and the environment. These concerns have prompted increased regulatory scrutiny and efforts to develop safer alternatives to TBBPA.

1.2 Objectives

1. To develop an efficient and scalable synthesis method for TBBPA.
2. To optimize reaction conditions and parameters to maximize the yield of TBBPA.
3. To characterize the synthesized TBBPA using various analytical techniques, such as HPLC, ¹H NMR, and mass spectrometry, to confirm its purity and structural integrity.
4. To explore the potential applications of TBBPA and its derivatives in various industries, such as electronics, textiles, and construction.
5. To contribute to the understanding of TBBPA synthesis and its potential implications for human health and the environment.

1.3 Motivation

The synthesis of tetrabromobisphenol A (TBBPA) is driven by the need for safer and more environmentally friendly flame retardant materials. With concerns surrounding the potential health and environmental impacts of TBBPA, there is a strong motivation to explore efficient and sustainable synthesis methods. By focusing on TBBPA synthesis, this research aims to contribute to the development of greener flame retardant alternatives. Additionally, characterizing the synthesized TBBPA will provide insights into its chemical structure and purity, enabling better assessment of its performance and potential applications. Ultimately, this research seeks to advance knowledge and promote the use of safer flame retardants, ensuring the protection of both human health and the environment.

Chapter 2 – Literature Review

2.1 Production method for tetrabromobisphenol-A[1]

The bromination of Tetrabromobisphenol-A (TBBPA), known for its limited solubility in liquid-phase reactions, involves using a controlled deficiency of bromine. This leads to the formation of a precipitate, consisting of 50-95% TBBPA and 50-5% underbrominated bisphenol-A, within the reaction mixture. The isolation of this precipitate allows for the subsequent recovery of TBBPA, ideally during the bromination process. The underbrominated bisphenol-A can be recycled as a feed for further bromination, ensuring efficient utilization of raw materials. This innovative process technique enables the production of high-purity TBBPA with desirable characteristics such as low ionic halide content and excellent color. It not only minimizes waste product generation but also reduces disposal costs, making it an environmentally and economically advantageous approach.

2.2 Purification process for tetrabromobisphenol A mother liquor that is in use[2]

The invention discloses a method for purifying the circulating mother liquor of tetrabromobisphenol A (TBBPA). The process involves several steps, starting with the dissolution of bisphenol A in monochlorobenzene, followed by the addition of bromine to initiate a bromination substitution reaction, resulting in a monochlorobenzene solution of tetrabromobisphenol A. The purification process includes neutralization and washing with sodium sulfite, as well as the generation of solid tetrabromobisphenol A through operations such as cooling crystallization and centrifugation of the centrifuging mother liquor.

This purification technique enhances the quality of the circulating mother liquor used in the synthesis of TBBPA. The reprocessed organic material present in the recovered mother liquor can be utilized as a sellable product, while the wastewater generated undergoes treatment in a biochemical treatment device to ensure compliant discharge, fulfilling the requirement for continuous production of TBBPA and maximizing the overall benefits for the enterprise.

2.3 Process for recycling tetrabromobisphenol a substance for the middle layer[3]

The invention presented addresses the field of wastewater treatment in the organic chemical industry and introduces a method for recycling and reusing intermediate layer material in the synthesis of tetrabromobisphenol A (TBBPA). The waste generated during the process is collected and analyzed to determine its composition. Based on the content of various brominated products, the required amount of bromine for re-bromination is calculated. Through the re-bromination process, the intermediate layer material is transformed into TBBPA, meeting the specified standards.

By brominating the bisphenol A, monobromo bisphenol A, dibromo bisphenol A, and tribromo bisphenol A present in the waste materials, the hazardous waste treatment input is minimized, and the yield of TBBPA is enhanced. Moreover, this method ensures no potential safety risks to the ecological environment, emphasizing both safety and environmental friendliness. The innovative approach contributes to waste reduction, increased TBBPA production efficiency, and aligns with sustainable practices in the organic chemical industry.

2.4 Tetrabromo biphenol A manufacturing technology: high whiteness[4]

The synthesis of tetrabromodiphenol A involves a substitution reaction between bromine and biphenol A in the presence of benzene chloride, resulting in the formation of tetrabromodiphenol A and hydrogen bromide (HBr) as a byproduct. Subsequently, tetrabromodiphenol A is produced by oxidizing HBr using hydrogen peroxide (H₂O₂). To obtain high-purity tetrabromodiphenol A with excellent whiteness, excess bromine and colored impurities are reduced through appropriate chemical treatments.

2.5 Process for making very pure tetrabromobisphenol A[5]

The invention presents a precise and efficient method for synthesizing tetrabromobisphenol A with exceptional purity. The preparation process involves the following sequential steps:

1. Dissolving bisphenol A in a solvent and adding a catalyst to initiate the reaction.

2. Simultaneously adding bromine and hydrogen peroxide dropwise at a specific temperature while continuously stirring the solution.
3. Heating the tetrabromobisphenol A solution obtained in step 2 to facilitate the curing reaction.
4. Allowing the cured tetrabromobisphenol A solution to stand and separating the water.
5. Conducting cooling and crystallization, followed by suction filtration and drying of the obtained crystals.
6. Collecting the filtrate from step 5 and adding water to it, then subjecting it to distillation and crystallization to obtain a product with a purity greater than 97%.

The resulting tetrabromobisphenol A exhibits remarkable purity. A cooling crystallization mode yields a product with a purity of 99.9% or higher, while a distillation crystallization mode produces tetrabromobisphenol A with a purity exceeding 97% from the residual feed liquid.

2.6 Review of the incidence of TBBPA and its derivatives in the environment, analytical methods, degradation, and toxicity[6]

Brominated flame retardants (BFRs), including tetrabromobisphenol A (TBBPA), are extensively used in various industries to enhance the fire resistance of polymeric materials and mitigate fire spread. However, the widespread use of TBBPA has resulted in its presence as a common indoor pollutant. Recent studies have raised concerns about the potential adverse effects of TBBPA and its derivatives on both human health and wildlife. This article provides a comprehensive review of the existing literature on TBBPA and its derivatives, focusing on their environmental levels and human exposure. Various analytical techniques and procedures have been employed to accurately and sensitively analyze TBBPA and its derivatives in different environmental compartments. These pollutants have been found to be ubiquitous, with distribution observed across global ecosystems. Once released into the environment, TBBPA can undergo processes such as adsorption, biological degradation, or photolysis. Implementing regulations on TBBPA production, usage, and waste treatment can help minimize environmental discharge and associated health risks. Effective treatment methods, including adsorption, ozonation, oxidation, and anaerobic degradation, have been developed to address TBBPA contamination. Previous investigations have reported detrimental effects associated with TBBPA and its derivatives. Human exposure studies have indicated that dietary intake and dust ingestion are the primary routes of exposure for the general population, with toddlers being particularly vulnerable due to inadvertent indoor dust consumption. Additionally, exposure to TBBPA can occur during pregnancy and through breastfeeding. This review serves to bridge the knowledge gap regarding the adverse consequences and hidden effects of TBBPA and its derivatives, contributing to a better understanding of their implications.

2.7 The study of TBBPA/TBBPS, TBBPA/TBBPS derivatives, and their transformation products has recently advanced.[7]

Tetrabromobisphenol A (TBBPA) is widely acknowledged as the predominant brominated flame retardant used in various applications. TBBPA derivatives, including TBBPA and tetrabromobisphenol S (TBBPS), are reactive substances utilized in the production of TBBPA/S compounds, offering an alternative to TBBPA. The presence and transformation of TBBPA/S and its derivatives in different environmental contexts raise concerns regarding their potential impacts on human health and environmental safety. Therefore, it is essential to establish reliable sample preparation techniques and sensitive analytical methods capable of detecting and identifying TBBPA/S and its derivatives in diverse environmental matrices. This study provides a comprehensive review of analytical methods employed for the analysis of TBBPA/S and its derivatives. Furthermore, it offers an extensive overview of techniques used for the identification of previously unknown metabolites derived from these compounds. The viewpoint section of our review focuses on recent advancements in analytical approaches for investigating novel TBBPA/S compounds.

Chapter 3 - Methodology and Experimental Work

3.1 General Experimental

Apparatus used: The apparatus used in research includes RBF, measuring cylinders, beakers, glass rod, stoppers, bottles, glass jars, thermometer, head stirrer, funnels, etc.

Raw materials used: All the solvents were of commercial grade. Chemicals included were Bisphenol-A (BPA), Bromine, Aq. H₂O₂ solution (35%), Aq. HBr (~48%) solution, EDC, SLS, 25% Sodium Sulfite Solution, Water, Chlorobenzene

Techniques and instruments:

Compound analysis involves the application of identification and separation procedures using a variety of instruments.

Chromatography: Chromatography is a fundamental technique utilized for the separation, purification, and analysis of compounds. It operates by exploiting the differential interactions between the mobile and stationary phases. The mobile phase, which can be a gas or liquid, dissolves the compound mixture and carries it through a

system (such as a column, capillary tube, plate, or sheet) where a stationary phase material is immobilized. This differential interaction leads to the separation of the mixture into its individual compounds.

NMR - Nuclear Magnetic Resonance (NMR) spectroscopy is a highly valuable analytical technique used to gather information about the local chemical environment of magnetically active nuclei, including ^1H , ^{13}C , and others. In NMR spectroscopy, the nuclei in a sample are excited by radio waves, inducing nuclear magnetic resonance. This resonance is then detected using sensitive radio receivers while the sample is placed in a magnetic field. The ^1H -NMR spectra were annotated using specific abbreviations to describe the observed signals. These abbreviations include s (singlet), d (doublet), t (triplet), m (multiplet), dd (double doublet), ddd (double of double doublet), and dddd (double doublet of double doublet). These descriptors assist in characterizing the multiplicity and splitting patterns of signals in the ^1H -NMR spectra.

Mass spectroscopy - In mass spectrometry, the ionization of a molecule occurs when it is subjected to high-energy electrons (70 eV). This electron impact causes the molecule to break apart into fragment ions with different masses. Mass spectrometry provides information about the molecular weight, molecular formula, molecular structure, and isotopic abundance of the analyzed molecule. The process involves recording low-resolution mass spectra using a mass spectrometer, specifically referred to as LR-EIMS (Low-Resolution Electron Impact Mass Spectrometry).

HPLC - High-performance liquid chromatography (HPLC) is a widely used analytical technique for separating, identifying, and quantifying components in a mixture. It is based on the principle of chromatography, which involves the differential interactions between sample components and two phases: a mobile phase (liquid solvent) and a stationary phase (solid or liquid adsorbent material).

3.2 Synthesis

3.2.1 ROS-1

Reaction scheme:



Stoichiometry:

Compound Name	Weight taken(gm)	Molecular Weight(g/mol)	No. of mol	Mole equivalent
Bisphenol A	50	228.29	0.2190	1.00
EDC	220	98.96	-	4.4
Bromine	70.78	160	0.4423	2.02

Procedure:

Prepare a clean and dry 1-liter round-bottom flask (RBF) and place it in a water bath with an overhead stirrer. Charge the RBF with 50g of bisphenol A (BPA), 2.5g of an unspecified sol., 100g of EDC, and 22g of water. Start stirring and gradually raise the temperature to 37-38°C. Set up two additional funnels, one containing 45g of a hydrogen peroxide (H_2O_2) solution (33.5%) and the other containing 70.78g of bromine along with 120g of EDC. Simultaneously add bromine and the H_2O_2 solution, maintaining a ratio of H_2O_2 to bromine as half in terms of EDC quantity. Once the addition is complete, increase the reaction temperature to 55-60°C. After maintaining this temperature for 1 hour, monitor the reaction using high-performance liquid chromatography (HPLC). Begin the work-up process once the desired result is achieved.

Quench the excess bromine and adjust the pH to 3.5-4 by adding a 20% sodium metabisulphite (Na_2SO_5) solution. Separate the layers and remove the upper aqueous layer. Treat the organic layer with 50g of water for 30 minutes, followed by layer separation. Under vacuum distillation, remove the organic layer until a solid mass is observed. Cool the mass to 10-15°C and perform filtration. The obtained wet cake from the first crop is 104g, which is further processed into a white powder. After drying, the dry weight of the first crop is 102g, resulting in a yield of 85.71% with an HPLC purity of 99.89%.

For the isolation of the second crop, collect the lower EDC layer weighing 81g. Under vacuum distillation, remove the organic layer until a solid thick mass is observed. Cool the mass to 1015°C and perform filtration. The resulting wet cake from the second crop is 10g, which is converted into a white powder. After drying, the dry weight of the second crop is 8.5g.

Result:

Overall, the total yield from the first and second crops combined is 110g, with a yield of 92.85%.

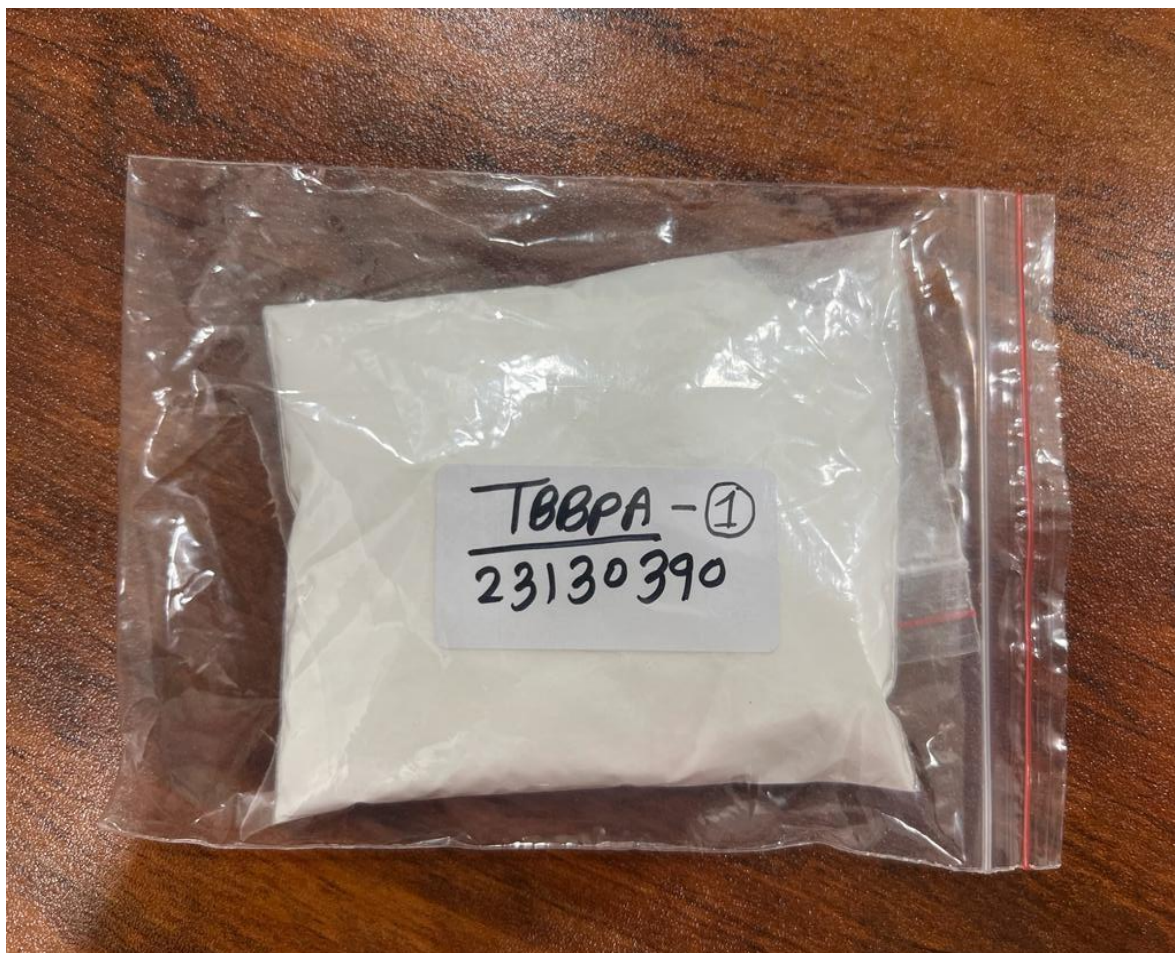
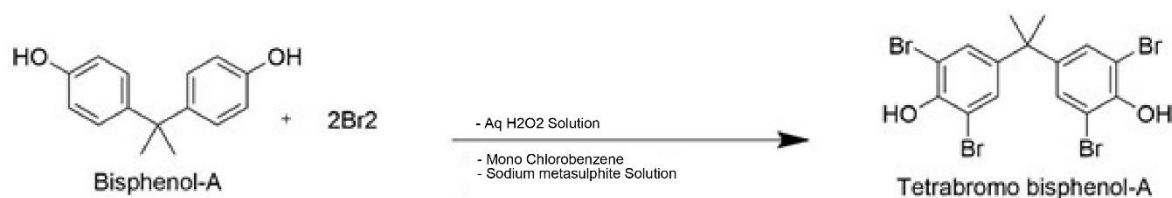


Figure 1 White powder product of TBBPA by ROS 1

3.2.2 ROS-2

Reaction scheme:



Stoichiometry:

Compound Name	Weight taken(gm)	Molecular Weight(g/mol)	No. of mol	Mole equivalent
Bisphenol A	50	228.29	0.2190	1.00
Chlorobenzene	150	112.56	-	3.0
Bromine	72.53	160	0.4523	2.07

Procedure:

Prepare a 1-liter round-bottom flask (RBF) and place it in a water bath with an overhead stirrer. Ensure that the setup is clean and dry. Add 50g of bisphenol A (BPA), 0.17g of an unspecified compound, 150g of chlorobenzene, 45.5g of a 33.5% hydrogen peroxide (H₂O₂) solution, and 50g of water to the RBF. Start stirring the mixture and gradually cool the temperature to 20-30°C. Within 2 hours, slowly add neat bromine while maintaining the reaction temperature between 20-30°C. Once the addition is complete, increase the reaction temperature to 80°C. After maintaining this temperature for 1 hour, monitor the reaction using high-performance liquid chromatography (HPLC) to check if the desired product, tetrabromobisphenol A (TBPA), is present at a purity of >98.0%.

Once the desired purity is achieved, begin the work-up process. Raise the reaction temperature to 60°C and add a 20% sodium metabisulphite (Na₂SO₅) solution to quench excess bromine. Then, cool the reaction mass to 10-15°C and perform filtration. The obtained wet cake from the first crop is 110g, which is further processed into a white powder. After drying, the dry weight of the first crop is 107g, resulting in a yield of 89.9% with an HPLC purity of 99.88%.

Collect the filtrate and perform layer separation. Remove the upper aqueous layer and transfer the lower chlorobenzene layer back into the RBF. Distill the chlorobenzene until a solid thick mass is observed. Cool the mass to 10-15°C and carry out filtration. The resulting wet cake from the second crop is 7.5g, which is converted into a white powder. After drying, the dry weight of the second crop is 6g, resulting in a second crop yield of 4.2%.

Result:

Overall, the total yield from the first and second crops combined is 113g, with a yield of 95%.

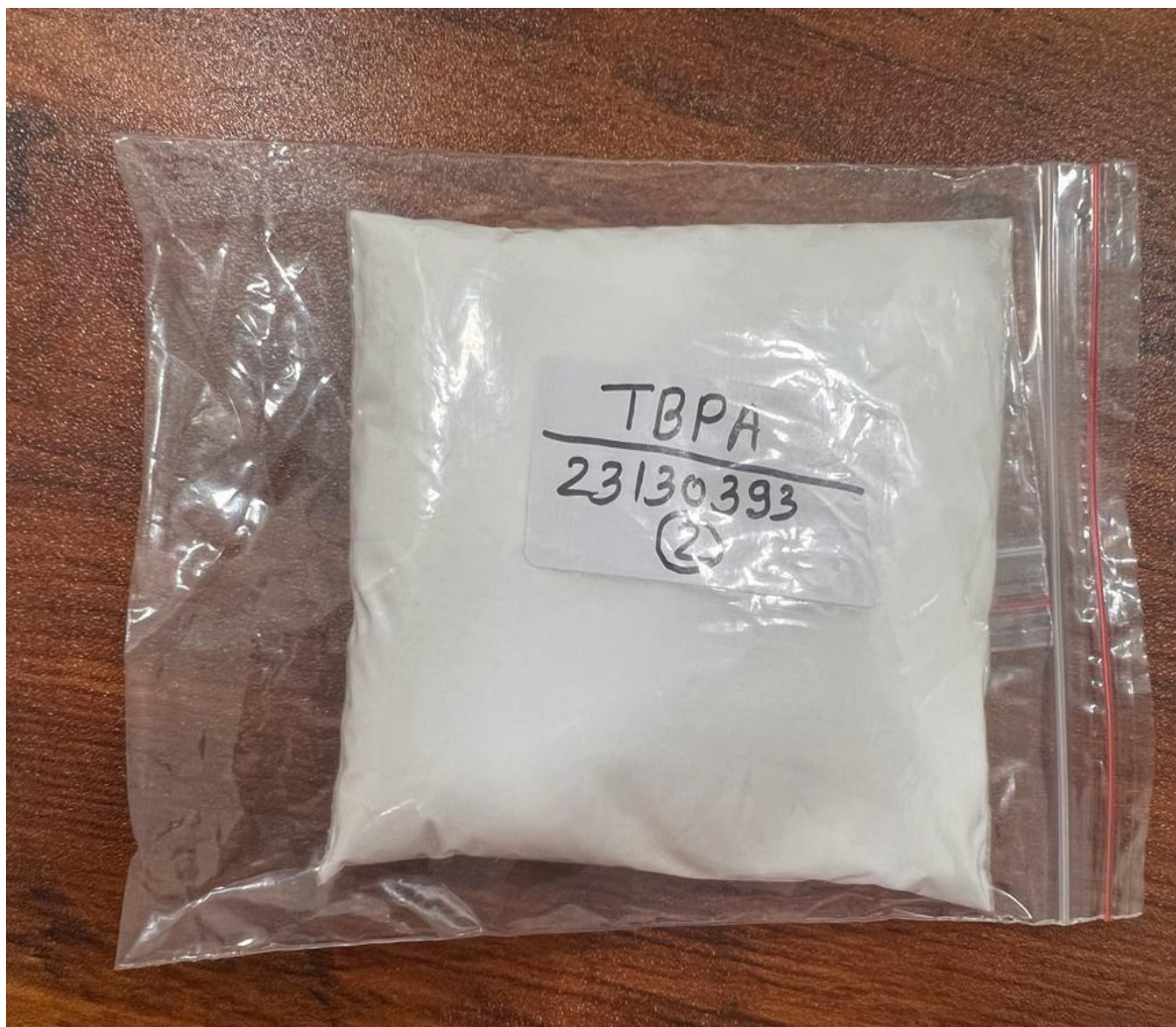


Figure 2 White powder product if TBBPA

Difference between Two ROS:

ROS 1(EDC ROUTE)	ROS 2(MCB ROUTE)
MORE OPERATION	Single step process
LOW BOILING POINT OF ETHYLENE DICHLORIDE SO LESS RECOVERY	High boiling point of chlorobenzene so good recovery
LOW VOLATILE	High volatile
LESS FEASIBLE	More feasible
LAYER SEPARATION FOR YIELD	Layer separation is not involved
LESS YIELD THAN ROS-2	High yield
MORE COST DUE TO MORE OPERATION	Cost effective

Chapter 4 - Characterization

In this project, the characterization of tetrabromobisphenol A (TBBPA) was conducted using several analytical techniques, including high-performance liquid chromatography (HPLC), ^1H nuclear magnetic resonance (NMR) spectroscopy, and mass spectrometry.

4.1 HPLC

HPLC was employed to separate and quantify TBBPA in the sample. This technique utilizes the differential interactions between the sample components and the mobile and stationary phases. The HPLC system, equipped with a suitable column and detector, allowed for the accurate measurement of TBBPA content. The retention time and peak area of TBBPA were compared to known standards to confirm its presence and determine its concentration.

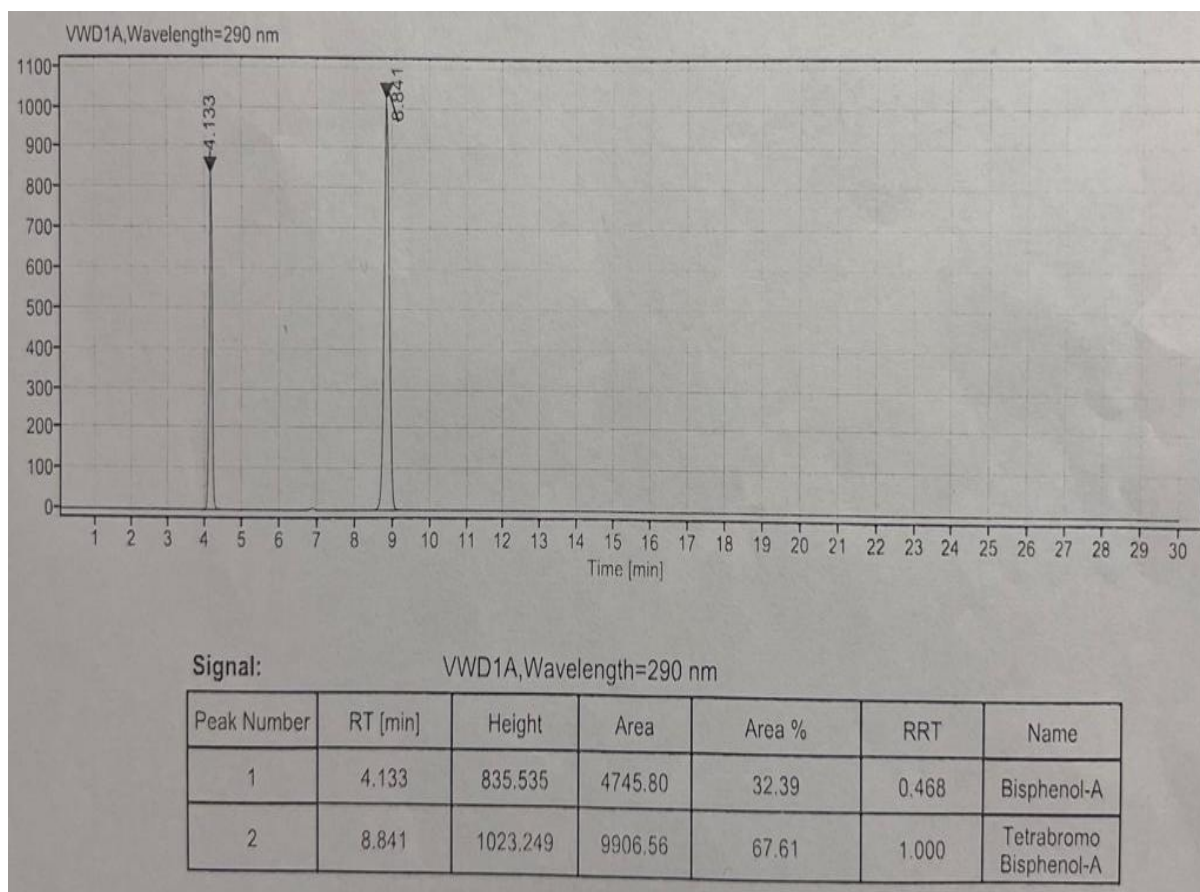


Figure 3 Standard peaks of Bisphenol A and TBBPA

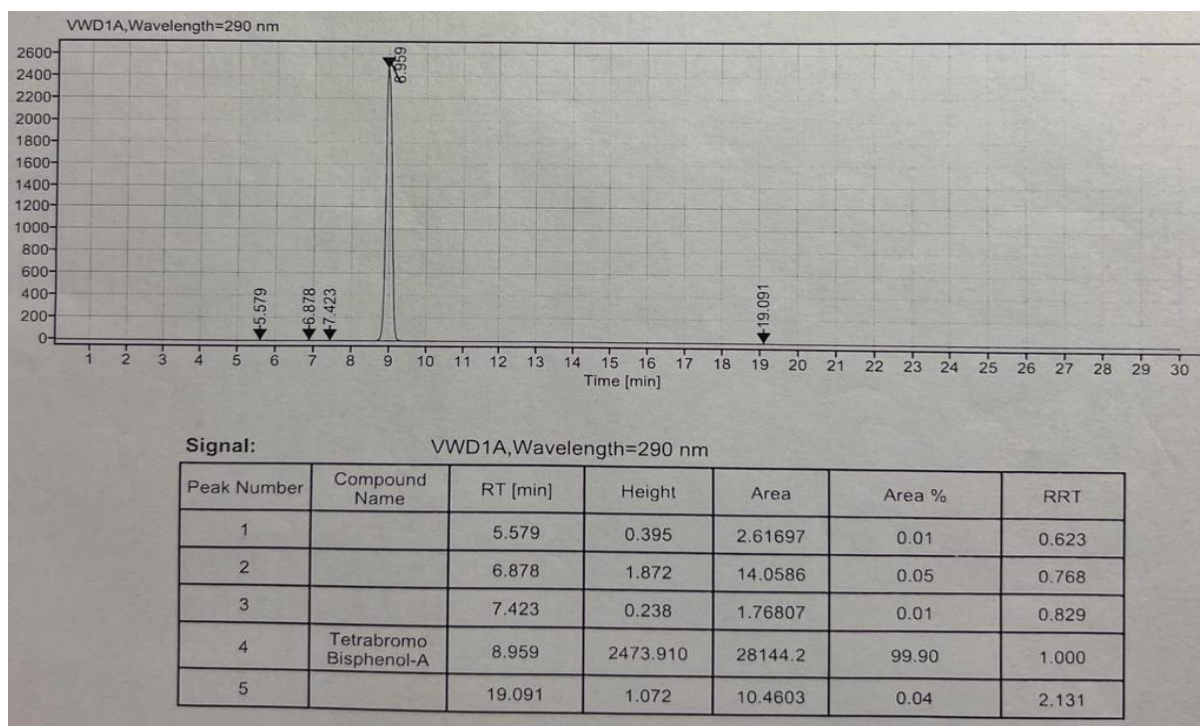
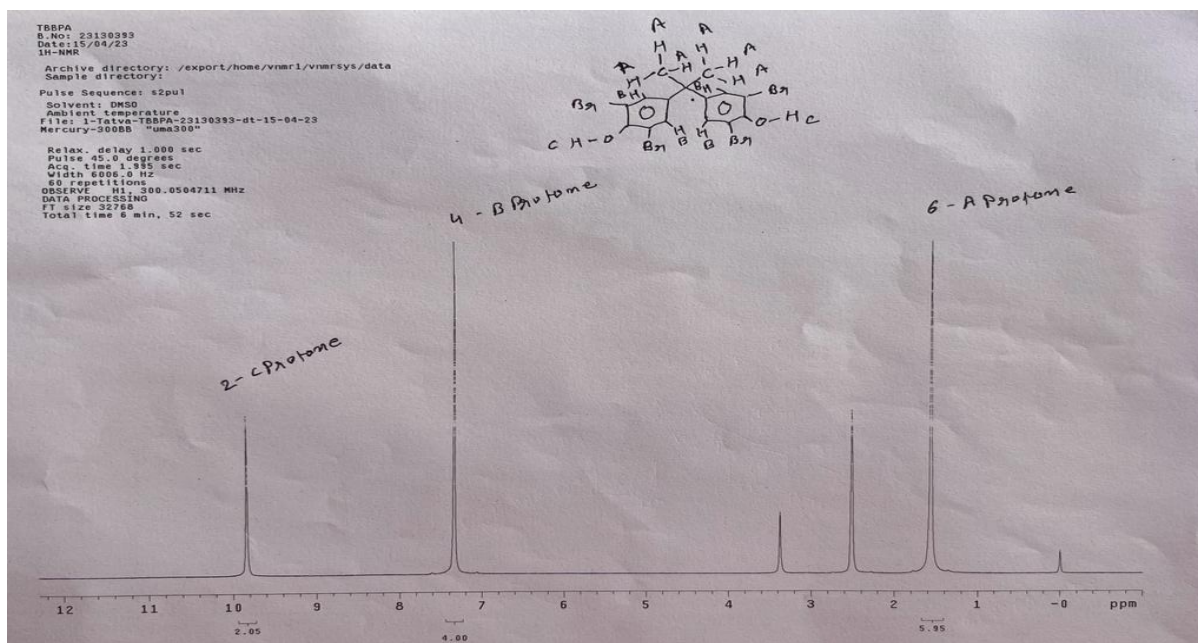


Figure 4 Conformation Peak of TBBPA

4.2 ^1H NMR

^1H NMR spectroscopy provided information about the structural characteristics and surrounding environment of TBBPA. This technique involves the excitation of magnetically active hydrogen nuclei in the sample using radio waves. The resulting NMR signal provided insights into the connectivity and chemical environment of the hydrogen atoms in TBBPA, aiding in its identification and structural analysis.

Figure 5- ¹H NMR of TBBPA

4.3 Mass spectroscopy

Mass spectrometry played a crucial role in determining the molecular weight and fragmentation pattern of TBBPA. Ionization of the TBBPA molecules was achieved by high-energy electrons, resulting in the formation of fragment ions. The mass spectrometer, specifically low-resolution mass spectrometry in this project, detected these ions and provided information on the molecular weight, molecular formula, and isotopic abundance of TBBPA. This technique helped confirm the identity of TBBPA and provided insights into its structural features.

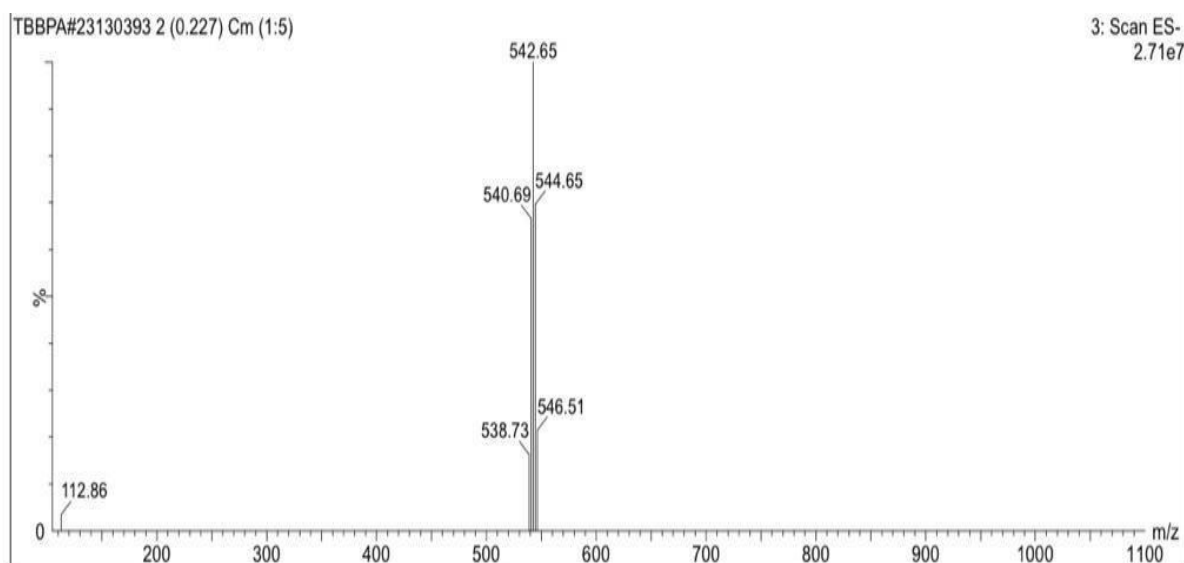


Figure 6 Mass spectrum of TBBPA

By combining the results obtained from HPLC, ¹H NMR spectroscopy, and mass spectrometry, a comprehensive characterization of TBBPA was achieved. The HPLC analysis quantified the amount of TBBPA present, while NMR spectroscopy provided structural details and confirmed its identity. Mass spectrometry complemented the findings by providing information on the molecular weight and fragmentation pattern. Together, these techniques enabled a thorough understanding of the properties and composition of TBBPA in the study.

4.4 Validation

Following the HPLC analysis of the TBBPA product, a validation process was conducted to assess the reliability and accuracy of the analytical method used. This validation included evaluating method precision, system precision, and linearity.

4.4.1 Method precision

Method precision was determined by analyzing multiple replicate samples of the TBBPA product using the same HPLC method. The objective was to assess the repeatability and consistency of the method in producing consistent results. The samples were prepared and analyzed under identical conditions, and the measured concentrations of TBBPA were compared. The method precision was evaluated by calculating statistical parameters such as percent relative standard deviation (%RSD) to determine the precision of the method.

4.4.2 System precision

System precision aimed to assess the reproducibility and consistency of the HPLC system itself. To evaluate system precision, multiple injections of the same TBBPA sample were made using the HPLC system. The retention time and peak area of TBBPA were monitored across these injections, and statistical parameters such as %RSD were calculated to determine the precision of the HPLC system.

4.4.3 Linearity

Linearity analysis was performed to evaluate the relationship between the concentration of TBBPA and the response of the HPLC system. A series of TBBPA standards with varying concentrations were prepared and analyzed using the HPLC method. The peak areas or heights corresponding to each concentration were recorded, and a calibration curve was constructed. The linearity of the method was assessed by analyzing the correlation coefficient (R^2) and the linearity range of the calibration curve.

By conducting method precision, system precision, and linearity analysis, the validity and reliability of the HPLC method for analyzing TBBPA were assessed. These validation steps provided crucial information about the precision, accuracy, and linearity of the method, ensuring the quality and credibility of the analytical results obtained for the TBBPA product.

Chapter 5 - Conclusion and Future aspects

Conclusion

In this project, the characterization of tetrabromobisphenol A (TBBPA) was successfully carried out using various analytical techniques including HPLC, ^1H NMR spectroscopy, and mass spectrometry. The HPLC analysis provided accurate quantification of TBBPA, while ^1H NMR spectroscopy offered insights into its structural features and chemical environment. Mass spectrometry aided in determining the molecular weight and fragmentation pattern of TBBPA. The characterization of TBBPA using a combination of HPLC, ^1H NMR spectroscopy, and mass spectrometry proved to be effective in providing a comprehensive understanding of its properties and composition. The analytical methods utilized in this study demonstrated good precision, system precision, and linearity, ensuring the reliability and accuracy of the results obtained. The validation process confirmed the robustness of the HPLC method for TBBPA analysis.

However, it is important to address the health and environmental concerns associated with TBBPA. The widespread usage and potential accumulation of TBBPA and its derivatives in various environmental compartments raise concerns about their impacts on human health and ecological systems. Further research is needed to understand the potential toxicity and persistence of TBBPA in the environment, as well as its potential for bioaccumulation in organisms.

To mitigate these concerns, it is crucial to explore alternative flame retardant materials that possess reduced environmental and health risks while maintaining effective fire safety properties.

In the future, it is essential to continue investigating the occurrence and fate of TBBPA in diverse environmental matrices, as well as its potential transformation products and metabolites. By developing sensitive analytical methods and sample preparation techniques, we can enhance our ability to detect and monitor TBBPA and its derivatives in the environment, enabling better risk assessment and management strategies.

By addressing the health and environmental concerns associated with TBBPA and advancing our understanding of alternative flame retardant materials, we can work towards a safer and more sustainable approach to fire safety without compromising human health or environmental security.

Future prospects

Based on the findings of this project, several future prospects can be considered. Further research can focus on exploring the degradation pathways and transformation products of TBBPA under different environmental conditions. Additionally, studies can be conducted to investigate the potential health and environmental impacts of TBBPA and its derivatives. Developing more sensitive analytical methods and sample preparation techniques for diverse environmental matrices can enhance our understanding of the occurrence and fate of TBBPA in the environment. Furthermore, efforts can be directed towards the development of alternative flame retardants that possess reduced environmental and health risks compared to TBBPA, addressing the concerns associated with its usage.

By continuing to investigate and expand our knowledge of TBBPA, its analytical methods, and its environmental implications, we can contribute to the development of safer and more sustainable flame retardant materials.

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