



Industrial Consultancy & Sponsored Research (IC&SR)

A CHROMATOGRAPHIC METHOD FOR SEPARATION OF β-DIBROMO-AND TRIBROMO-*MESO*-TETRAPHENYLPORPHYRINS IITM Technology Available for Licensing

Problem Statement

- A few isomers exist for H₂TPPBr₂, but their separation is hindered due to poor solubility in low polarity solvents, leading to resolution challenges in column chromatography.
- Current chromatographic methods involve resin, alumina, silica gel, and reverse phase supports, often demanding large amounts of low polarity solvents and proving to be timeconsuming and intricate processes.
- Hence, there is a need to selectively synthesize and efficiently separate H₂TPPBr_n (n = 2 and 3) derivatives.

Technology Category/ Market

Category - Chemicals, Porphyrinic materials

Applications - Porphyrinic materials and medicine, Chemical Synthesis, Materials Science, Catalysis, Renewable energy devices.

Industry - Chemical Manufacturing, Materials and Nanotechnology, Pharmaceuticals.

Market - The global chromatography columns market is projected to surpass US\$ 3.3 Billion by 2024, with a **CAGR of 8.4%** during the forecast period 2023 - 2030.

TRL (Technology Readiness Level)

TRL - 4: Technology validated in lab scale.

Research Lab

Prof. Bhyrappa, P. Department of Chemistry

CONTACT US

Dr. Dara Ajay, Head Technology Transfer Office, IPM Cell- IC&SR, IIT Madras

IITM TTO Website: https://ipm.icsr.in/ipm/

Intellectual Property

- IITM IDF Ref. 1265
- IN 338544 Patent Granted

Technology

- The present invention relates to a combined use of chromatographic method with moderate to more polar solvent / polar solvent mixtures for the separation of porphyrins in particular βdibromo and tribromo-meso-tetraphenylporphyrins.
 - The main aspect of the present invention is chromatographic separation of H_2TPPr_n (n = 2 and 3) mixture (as shown in Fig. 1,3) using single column consisting of a mixture of two chromatographic supports such as alumina/silica gel/charcoal/cellulose, with varying composition.
 - •Solvent Selection: Employs moderately to highly polar solvents or solvent mixtures (e.g., $CHCl_3$, CH_2Cl_2 , CCl_4 , toluene) to achieve precise separation of H_2TPPBr_n (n = 2 and 3) derivatives.
 - **Rapid and Controlled Process:** Enables separation completion within 12 hours, marked by colorless elution, and ensures clear differentiation between H₂TPPBr₂ and H₂TPPBr₃ derivatives.
 - Characterization Tools: Utilizes electronic absorption, ¹H NMR spectroscopy, and low-resolution ESI mass spectrometry for accurate identification and validation of separated compounds.
 - The process is rapid for the separation of 0.5 grams of a porphyrin mixture within few hours and total elution time taken is 10-12 hours.

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Email: smipm-icsr@icsrpis.iitm.ac.in

sm-marketing@imail.iitm.ac.in

Phone: +91-44-2257 9756/ 9719

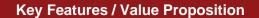
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Indian Institute of Technology Madras

- separation Having facile of two brominated porphyrins, H_2TPPBr_n (n = 2 and 3) from a single column consisting of a mixture of two chromatographic supports with varying composition.
- □ Useful for at least 0.5 g scale brominated porphyrin mixture in moderate to more polarity solvents / solvent mixtures within several hours.

 $X = Br, Y = H, H_2TPPBr_2$ 2,3-Dibromo-5,10,15,20-tetraphenylporphyrin $X = H, Y = Br, H_2TPPBr_2$ 2,12 or 2,13-Dibromo-5,10,15,20-tetraphenylporphyrin

FIG. 1. Depicts molecular structures of H₂TPPBr₂ derivatives.

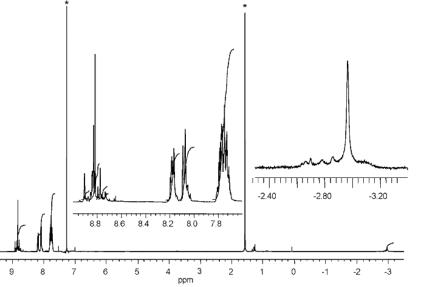


FIG. 2. Illustrates ¹H NMR spectrum of H₂TPPBr₂ in CDCl₃ at 298 K (* denotes impurity peaks).

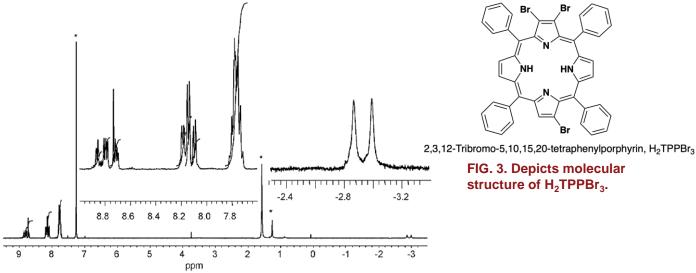


FIG. 4. Illustrates ¹H NMR spectrum of H₂TPPBr₃ in CDCI₃ at 298 K (* denotes impurity peaks).

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