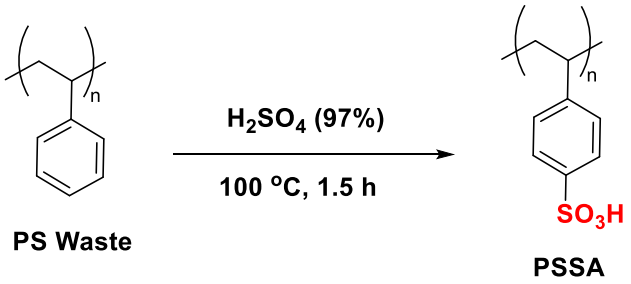
Experimental Protocol for the Preparation of Polystyrene Sulfonic Acid (PSSA)



PS Waste **PSSA**

1 g of expanded polystyrene (PS) waste is first dissolved in the minimum amount of ethyl acetate in a beaker. The resulting solution is transferred to a round-bottom flask, and the solvent is evaporated using a rotary evaporator with a water bath set at 35 °C. The flask is left connected to the rotavapor for an additional 20–30 minutes to ensure complete removal of the solvent.

The reduced PS is then scraped from the walls of the flask using a spatula and evenly distributed across the bottom to minimise volume. The flask is clamped to a stand, and 10 mL of concentrated sulfuric acid is added along with a Teflon-coated magnetic stirring bar.

The reaction mixture is heated at 100 °C for 1.5 hours using a heating block. It is recommended to use a glass rod during the reaction to help submerge any PS fragments not fully in contact with the acid. **Note: Do not use a metal spatula, as it will dissolve in hot sulfuric acid.**

After the reaction, the mixture is cooled in an ice-water bath, and ice-cold water is slowly added. A rubbery solid precipitates, which is then filtered under vacuum and washed thoroughly with deionised water until the filtrate reaches neutral pH.

The solid is dried overnight in an oven at 70 °C, yielding a yellowish-white product. The resulting PSSA is insoluble in deuterated solvents and is characterised by FTIR spectroscopy. The number of acidic sites is determined by titration with a 0.01 M NaOH solution using phenolphthalein as an indicator.

Typical yields range from 0.9 to 1.2 g of PSSA, with degrees of sulfonation between 30% and 45%.