

Nature of Invention: Chemical molecule and synthesis route

Applicant: QuantiVEX

Inventors: Shubham Singh, Pulkit

Chemical Formula: **(R-C₆H₄-SO₃Na)**

Chemical Name: Linear alkyl benzene sulphonate

Chemical synthesis routes:

a. Sulfonation of linear alkyl benzene

Raw materials

1. Linear alkyl benzene(R-C₆H₄) ,
2. SO₃ (sulphonating agent)
3. Hydrogen peroxide (a protic reagent and oxidising agent)
4. A Neutralizing agent

Sulfonation:

Reaction:



Mechanism:

Sulfonation of linear alkyl benzene (LAB) is an **electrophilic aromatic substitution (EAS)** reaction. The sulfonating agent, sulfur trioxide (SO₃), acts as an electrophile and attaches to the benzene ring of LAB.

- 1. Activation of Sulfur Trioxide (SO₃):**
 - a. SO₃ is a strong electrophile and reacts with benzene directly.
 - b. It forms a highly reactive intermediate due to the resonance stabilization of the benzene ring.
- 2. Formation of the Arenium Ion (σ-complex):**
 - a. The benzene ring donates electrons to SO₃, forming an unstable carbocation (arenium ion).
 - b. This intermediate is stabilized via resonance.
- 3. Proton Transfer and Formation of the Product:**
 - a. A base (often water or trace H₂SO₄) removes a proton from the arenium ion.
 - b. This restores aromaticity, leading to the formation of **linear alkyl benzene sulfonic acid**.

Reaction Conditions:

- **Temperature:** 0 - 90°C (typically controlled to prevent over-sulfonation), 25 – 50°C more preferable.
- **Pressure:** Up to 100 psi or more.
- **Solvent:** Can be carried out in a gas phase or liquid phase; sulfuric acid may be used as a solvent.
- **Reaction Type:** Highly **exothermic**, requiring efficient cooling.

Process Efficiency:

- Yield: 96~99%
- Purity: 96~98%

Sulfonation Process: Detailed Mechanism and Kinetics

The **sulfonation of Linear Alkyl Benzene (LAB)** follows **first-order kinetics**, with the reaction rate expressed as:

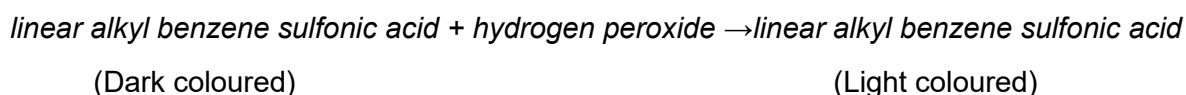
$$r = k[LAB][H_2SO_4]^2[H_2O]^{-1}$$

where excess sulfuric acid increases reaction speed, but excess water formation slows it down. The activation energy for the reaction is **18.75 kcal/mol**, as determined from Arrhenius modeling .

To ensure complete conversion (>98%), the molar ratio of **LAB:H₂SO₄** should be maintained at **1:5**. The **semi-batch reactor configuration** with **controlled acid addition** helps manage exothermic heat release. A **cooling jacket** is required to maintain **optimal reaction temperature (60°C)**.

Bleaching:

Reaction:



Mechanism:

1. Oxidation of Impurities:
 - a. Impurities (e.g., polymeric species, oxidized byproducts) contain conjugated double bonds, leading to colour.
 - b. H₂O₂ oxidizes these compounds, breaking their extended conjugation and rendering them colourless.

2. Radical Formation:

- Under suitable conditions, H_2O_2 decomposes to form hydroxyl radicals ($\cdot\text{OH}$).
- These radicals oxidize organic impurities to CO_2 and water.

Reaction Conditions:

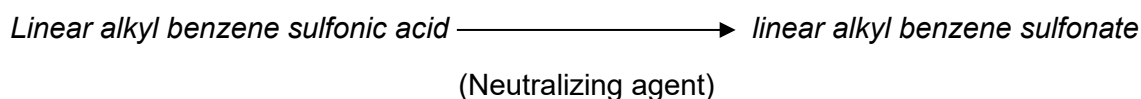
- Temperature:** 0 - 50°C
 - Higher temperatures accelerate oxidation but may degrade the product.
- Pressure:** Atmospheric pressure
- Catalyst:** Sometimes metal catalysts (e.g., sodium tungstate) are used to enhance bleaching efficiency.
- Solvent:** The reaction is typically carried out in an aqueous medium.

Process Efficiency:

- Yield: 98~99%
- Purity: 96~98%

Neutralization:

Reaction:



Mechanism:

1. Acid-Base Reaction:

- The sulfonic acid ($-\text{SO}_3\text{H}$) group reacts with a base, donating a proton (H^+).
- This forms the corresponding **sulfonate salt** ($-\text{SO}_3^- \text{Na}^+$).

2. Salt Formation and Stabilization:

- The product (linear alkyl benzene sulfonate, or **LAS**) is **water-soluble** and **stable**.
- This reaction prevents further oxidation and degradation.

Reaction Conditions:

- Temperature:** Room temperature to 50°C
- Pressure:** Atmospheric pressure
- pH control:** The final solution must be **mildly alkaline (pH ~7-8)** to ensure complete neutralization.
- Agitation:** Stirring is essential to ensure uniform mixing.

Process Efficiency:

- Yield: 98~99%
- Purity: greater than 99

Purification Processes:

After neutralization, the LABS solution still contains impurities such as **residual salts, excess alkali, unreacted LAB, sulfones, sulfates, excess neutralizing agents and side products**. These must be removed for high product quality.

a) Removal of Unreacted LAB

- **Liquid-Liquid Extraction:** Using a solvent such as **methanol or ethanol**, LAB can be selectively extracted.
- **Vacuum Distillation:** Since LAB has a lower boiling point than LABS, distillation can separate it effectively.
- **Adsorption:** Activated carbon or silica gel can absorb residual LAB.
- **Vacuum Stripping:** unreacted LAB is separated using **vacuum distillation or thin-film evaporation**. This ensures a **high-purity LABS product** with minimal contamination.

b) Removal of Sulfones and Sulfates

- **Washing with Water or Brine:** Dissolves and removes **sulfates** and some polar impurities.
- **Solvent Purification:** Methanol or ethanol can help separate sulfones from LABS.
- **Filtration through Activated Carbon:** Removes sulfones and organic residues.

c) Filtration and Drying

- Once LABS is purified, it is filtered to remove any remaining solid impurities.
- **Common filtration techniques:**
 - **Vacuum Filtration:** Speeds up separation of solids.
 - **Membrane Filtration:** Removes ultra-fine particles.
- **Drying Methods:**
 - **Spray Drying:** Converts LABS solution into dry powder form. The liquid LABS is sprayed into a hot air chamber, where water evaporates, leaving behind dry LABS powder.
 - **Vacuum Drying:** Used when LABS must be dried without exposure to high heat.
- Activated carbon treatment may be applied to remove any **color impurities**.

d) Crystallization (for High Purity LABS)

- Dissolve LABS in a solvent (e.g., ethanol).
- Slowly cool the solution to allow pure LAS crystals to form.
- Filter and dry the crystals.

Purity Testing and Final Product Evaluation

After synthesis, the **quality of LAS** is determined using:

1. **FTIR/NMR spectroscopy**: Confirms sulfonate ($-\text{SO}_3^-$) formation.
2. **Titration of residual acid**: Ensures neutralization is complete.
3. **High-Performance Liquid Chromatography (HPLC)**: Detects byproducts to maintain commercial-grade purity.

Scaling Up: Industrial Considerations

For **large-scale production**, Falling Film Reactors (FFR) are used to improve **reaction efficiency and heat management**. The **final product is spray-dried** to produce LAS in **powdered form**, making it ideal for detergent formulations.

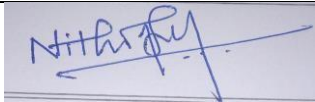
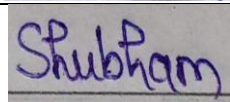
References:

- 1) <https://patents.google.com/patent/US2827484A/en>
- 2) <https://pmc.ncbi.nlm.nih.gov/articles/PMC8867685/>
- 3) https://hithaldia.in/eccn/vol6_1a/J5_6.pdf
- 4) <https://patentimages.storage.googleapis.com/1b/67/78/712514f5f0fc50/WO1997014676A1.pdf>

List the contributions of each author:

1. **Shubham Singh**: worked on selection of the chemical and selection of the manufacturing processes and contributed to the manufacturing analysis.
2. **Pulkit**: worked on studying the process, doing its manufacturing analysis.

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