



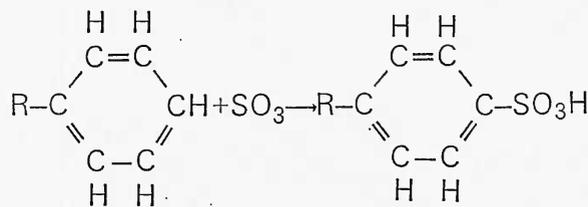
A UNILEVER LABORATORY EXPERIMENT NUMBER 2

The Preparation of Sodium Alkylbenzene Sulphonate

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The Preparation of Sodium Alkylbenzene Sulphonate

Sodium alkylbenzene sulphonate is a common soapless detergent and its molecular structure contains the specific features of a water-soluble head (the sulphonate ion) and a water-insoluble tail (the hydrocarbon paraffin chain). It is common practice for the hydrocarbon to be partly aromatic, i.e. an alkylbenzene, in order to facilitate sulphonation, which takes place at the benzene ring para to the paraffin side-chain.



The 'best' size for the side-chain R for good detergent behaviour is of the order of C_{12} and such material is often referred to as dodecylbenzene. In commercial material there is usually a mixture of sizes which, in relative abundance, rise to a maximum at C_{12} . The hydrocarbon has a flash point in the range $265\text{-}285^\circ\text{C}$ and it presents no fire hazard.

Two levels of experimental working are dealt with, quantities for the lower level being given first. This level corresponds with one type of Quickfit or similar apparatus available in School sets.

If it is preferred to measure quantities rather than weigh them, the Sp. Gr. of the hydrocarbon should be taken as 0.87, and that of 20% oleum as 1.92, in order to make the necessary calculations.

Apparatus

Only a simple set-up is illustrated, but those with more sophisticated apparatus will be able to modify it accordingly.

A 100 ml (300 ml) round-bottomed, wide-necked flask to take inlet and outlet tubes and the stirrer; or a 3-necked flask if available.

A stirrer (glass or polythene-covered steel)

A 50 ml (100 ml) separating funnel

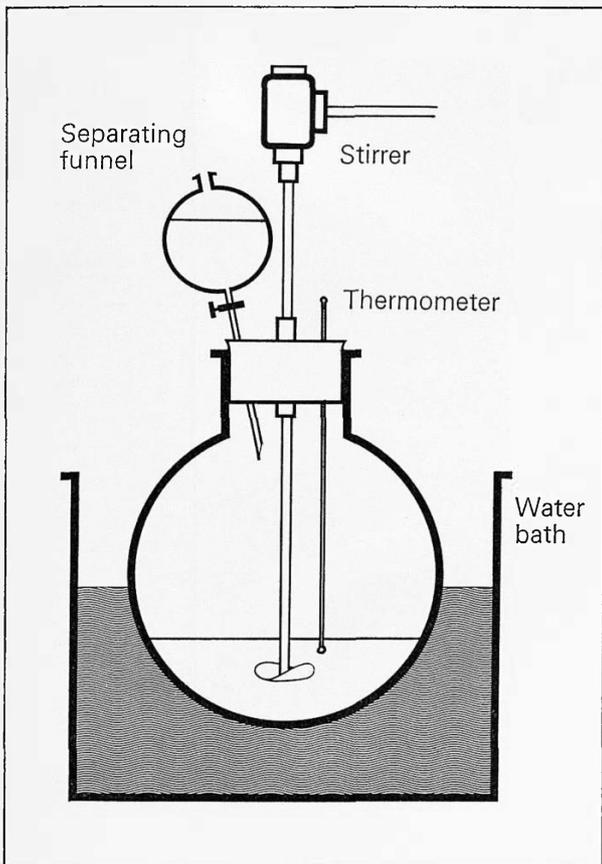
A water bath to contain the flask

Reactants

Alkylbenzene hydrocarbon (this will be supplied by Unilever Education Section)

Oleum (20% free sulphur trioxide)

Caustic soda: dissolve 3.2 g (8 g) sodium hydroxide in 6 ml (15 ml) distilled water.



Sulphonation

GREAT CARE MUST BE TAKEN WHEN HANDLING OLEUM. A PROTECTIVE LABORATORY OVERALL COAT, RUBBER GLOVES AND GOGGLES MUST BE WORN. ALL OPERATIONS SHOULD BE CARRIED OUT IN A FUME CUPBOARD IF POSSIBLE.

Weigh 20 g (50 g) hydrocarbon into the reaction flask. Put iced water into the bath and fix the flask in the water. Insert the thermometer and stirrer in the flask, and cool the contents to below 10°C.

Fix the separating funnel in position and transfer 18.8 g (47 g) oleum to the funnel. Keeping the cold water in the bath, add the oleum to the flask at such a rate that the temperature does not rise above 56°C and is $55 \pm 1^\circ\text{C}$ at the end of addition. Add crushed ice to the bath if necessary. Stir the reaction mixture vigorously throughout.

Replace the cold water in the bath with hot water, to maintain the temperature of the reaction mixture at $55 \pm 1^\circ\text{C}$ for a period of 30 minutes.

Remove the hot water, refill with iced water and cool the contents of the flask to below 20°C.

Neutralization

Weigh 8 g (20 g) finely crushed ice into a 100 ml (300 ml) beaker and fix the beaker in the water bath. Surround the beaker with ice-water mixture. Fix the stirrer, add the caustic soda and agitate. Insert the thermometer.

During neutralization it is important that the temperature should be kept below 50°C. Add 10 g (25 g) of the sulphonic acid as quickly as possible, consistent with keeping the temperature at this level. Now adjust to pH 6.5-7.5, with the aid of BDH universal indicator paper, using sulphonic acid or caustic soda as required. For testing, remove a spot of the mixture by means of a glass rod, and apply it to the indicator paper.

Note: the weights have been calculated to give a final moisture content of 60%, below which agitation becomes difficult.



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Chemicals
Plantations

The method of preparation of sulphonate paste described in this experiment is basically the same as that used by Unilever in the large-scale production of soapless detergents.



Unilever Laboratory Experiment
Number 2
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