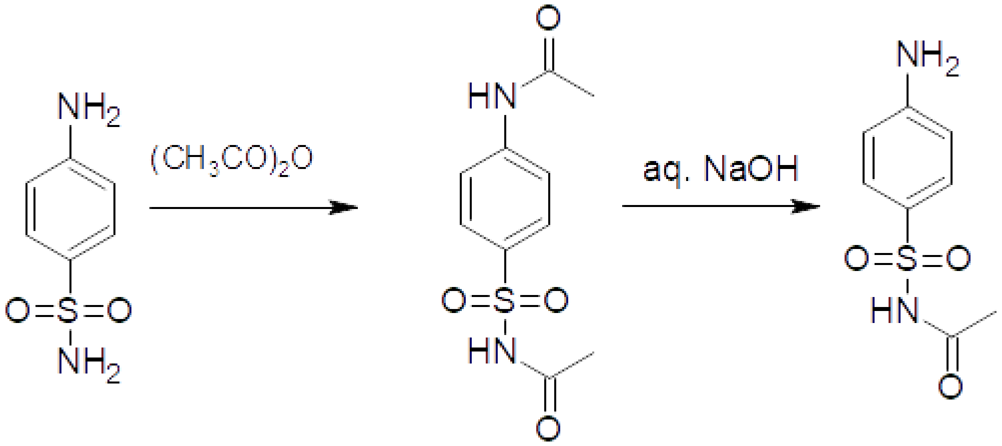
1. Sulfacetamide

Systematic name: *N*-(4-aminobenzensulfonyl)acetamide

Scheme of preparation:



Procedure

Step 1: N^1,N^4-diacetylsulfanilamide

Chemicals:

sulfanilamide [172.20]	8.6g	0.05 mol
acetic anhydride [102.09]	23 ml	0.24 mol
concentrated sulfuric acid		4 drops

To 8.6 g of 4-aminobenzenesulfonamide in a 250 ml round bottomed flask, 23 ml of acetic anhydride are carefully added under stirring by a slow cycling in a hand to avoid solidification. Then 4 drops of sulfuric acid are added and the mixture is refluxed under stirring on a magnetic stirrer equipped with an oil bath for 1 h, then cooled to room temperature and then 40 ml of cold water is added. Then it is closed by a stopper and shaked well during 1 minute which initiates formation of a white suspension. The solid N^1,N^4-diacetylsulfanilamide, formed as a precipitate, is then isolated by suction and washed with cold water (3-4 times); the yield is about 70-75%. The prepared wet intermediate is used for the second step without further purification.

(Properties: m. p. 253°C *well dried* (out of range of a melting point apparatus in our laboratory).

Step 2: *N*-(4-aminobenzensulfonyl)acetamide = sulfacetamide

Chemicals:

N^1,N^4-diacetylsulfanilamide [256.28]	cca12.81 g; cca 0.05 mol
NaOH [40.0]	50 ml of 2.5M solution
CH_3COOH [60.05]	50% solution q.s.
$NaHCO_3$	saturated solution q.s.

The crude N^1,N^4-diacetylsulfanilamide (9.2 g) is mixed with a 50 ml of 2.5M NaOH in of water. The mixture is then refluxed for 1 h in a flask under stirring on a magnetic stirrer equipped with an oil bath, then cooled under streaming water from the tap, and then neutralized to pH 4 - 5 with 50% acetic acid. The volume of the solution is then adjusted with water to approx. 80 ml in a beaker and carefully heated to boil under stirring with a glass rod. and again cooled with tap water outside of the flask. The formed precipitate is now isolated by suction filtration. If the solution is brown, or light-brown precipitate was beeng formed on the surface of the beaker when the solution was cold, small amount of activated charcoal is added and the mixture id boiled for 2 min. The hot mixture is then filtered through a folded paper filter to a beaker. This precipitate is then suspended in 10 ml of saturated sodium hydrogen carbonate ($NaHCO_{3)}$ solution and stirred vigorously with a glass rod. The filtrate is then cooled in a ice bath and the crystallization can be initiated by friction of the beaker bottom with a glass rod. The formed white suspension is then isolated by suction and washed wih cold water. Then it is put to dry on a Petri dish. Its m.p. is 181°C. The product is further characterized by TLC and ^{1}H-NMR spectra.