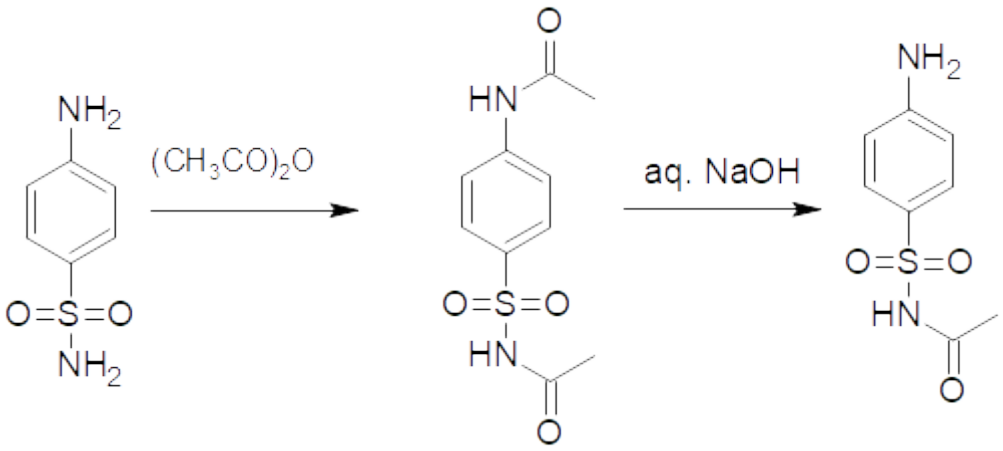
1. Sulfacetamide

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 then 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.

1. The crude N^1,N^4-diacetylsulfanilamide is mixed with a 50 ml of 2.5M sodium hydroxide solution in 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 formed precipitate is then isolated by suction. The isolated precipitate is then carefully treated with a saturated solution of sodium hydrogencarbonate (approx. 10 ml) in a beaker until the evolution of a gaseous CO_2 stops. The mixture is then filtered by suction. The **filtrate** is then made acidic again with acetic acid, then cooled in an ice bath, and the crystallization can be initiated by a friction of the beaker bottom with a glass rod. The solid is then isolated by suction. 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.