tion was concentrated to 100 ml and cooled. Total yield 52 g (63 %); m.p. 169°C. (Found: C 41.25; H 3.20; N 4.87. Calc. for $C_{10}H_{10}ClNO_5S$: C 41.17; H 3.43; N 4.80.)

4-Amino-2-hydroxybenzenesulfonamide (II), was prepared according to Thorpe and Williams [12] and recrystallised from propanol. M.p. 154–155°C. (Found: C 38.60; H 4.46; N 14.56. Calc. for $C_6H_8N_2O_3S$: C 38.30; H 4.28; N 14.89.)

2-(4'-Acetamido-2'-acetoxybenzenesulfonamido)-pyridine (III). The above acid chloride (5 g) and 2-aminopyridine (5 g) were dissolved in chloroform (50 ml), and the solution was refluxed for 10 min. The cooled solution was extracted three times with sodium carbonate solution. On acidification and cooling the sulfonamidopyridine separated as a white solid; yield 2 g. A better yield was obtained by the following procedure: The chloride (5 g) and 2-aminopyridine (2.1 g) were dissolved in pyridine (9 ml), and the solution was heated at 60–70°C for 10 min. The solution was diluted with 100 ml of water, acidified with acetic acid, and then neutralised with sodium bicarbonate. Yield 4 g (60 %). The product was recrystallised from glacial acetic acid; m.p. 247–249°C. (Found: C 51.05; H 4.20; N 11.68. Calc. for $C_{15}H_{15}N_3O_5S$: C 51.58; H 4.28; N 12.03.

2-(4'-Amino-2'-hydroxybenzenesulfonamido)-pyridine (IV). The foregoing compound (2 g) was dissolved in 2 N sodium hydroxide (10 ml), and the solution was refluxed for 4 h. On neutralisation of the cooled solution the deacetylated compound separated as a white solid. Yield 1.6 g. The compound was recrystallised from water with addition of carbon. It contained approximately half a molecule of water of crystallisation; it melted at 97–100°C, then solidified and melted again at 205°C. (Found: C 48.70; H 4.43; N 15.24, and after drying over phosphorus pentoxide: C 49.80; H 4.40; N 15.63. Calc. for $C_{11}H_{11}N_3O_3S.\frac{1}{2}H_2O$: C 48.20; H 4.80; N 15.15; and for $C_{11}H_{11}N_3O_3S$: C 49.81; H 4.18; N 15.84.)

4-Amino-2-hydroxybenzenesulfonanilide (V). The acetyl derivative was prepared in the same way as II. Yield 3.85 g from 5 g of the sulfonyl chloride (I). As analyses indicated a partial deacetylation, the compound was recrystallised from acetic anhydride. M.p. 208–213°C. (Found: C 54.80; H 4.70; N 7.98. Calc. for $C_{16}H_{16}N_2O_5S$: C 55.17; H 4.63; N 8.04.

The acetyl derivative (3 g) was dissolved in 1 N sodium hydroxide (20 ml) and refluxed for 2 h. On neutralisation and cooling the deacetylated compound separated. Yield 2.1 g; it was 1.3 g after three recrystallisations from water + ethanol. M.p. 175–176°C. (Found: C 54.60; H 4.66; N 10.27. Calc. for $C_{12}H_{12}N_2O_3S$: C 54.54; H 4.58; N 10.60.)

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Derivatives of *p*-Chlorobenzenesulfonic Acid

K. A. JENSEN and O. BUCHARDT

Chemical Laboratory of the University of Copenhagen, Copenhagen, Denmark

In the course of investigations on compounds with potential diuretic or antidiabetic activity we had to use N-(β-chloroethyl)-*p*-chlorobenzenesulfonamide as a starting material. A product supposed to have this composition has been described by Kulka [1]. However, we found that the product prepared according to Kulka and having the indicated melting point contained no aliphatic chlorine, and it was soon shown actually to be di-β-(*p*-chlorobenzenesulfonylaminoethyl)-sulfite, $(ClC_6H_4SO_2NHCH_2CH_2O)_2SO$. Kulka gives no Cl analysis of his product, but only C, H, and N values, with respect to which the chloride does not differ very much from the sulfite. The desired chloride was prepared from the corresponding hydroxy compound

and thionyl chloride with the addition of a trace of pyridine. When the preparation is carried out without pyridine, only the sulfite is obtained.

We have further prepared *p*-chlorobenzenesulfonyl derivatives of some amino acids: glycine, *α*-alanine, *β*-alanine, and phenylglycine, and transformed them into the corresponding acid chlorides. From these some amides were prepared.

Some attempts were made to cyclise the above-mentioned hydroxy and chloro compounds to benzothiazine derivatives, but without success.

Experimental

1. N-(β-Hydroxyethyl)-p-chlorobenzenesulfonamide. To a stirred solution of 106 g of *p*-chlorobenzenesulfonylchloride (0.5 mole) in 200 ml of chloroform, 31 g of ethanolamine (0.5 mole) was added dropwise. Then a solution of 25 g NaOH in 250 ml of water was added dropwise with cooling and stirring. The ethanolamide which separated in crystalline form was filtered off, washed with water and dried. A little more amide was recovered by evaporation of the chloroform layer. Yield 105 g (90 %), after crystallisation from chloroform 76 g. M.p. 104–105°C. (Found: C 40.65; H 3.73; N 5.98. Calc for $C_8H_{10}ClNO_3S$: C 40.78; H 4.28; N 5.95.

2. Di-β-(p-chlorobenzenesulfonylaminoethyl)-sulfite. To N-(*β*-hydroxyethyl)-*p*-chlorobenzenesulfonamide (11.3 g) suspended in benzene (25 ml) was added 22 ml of freshly distilled thionyl chloride, and the mixture was heated under reflux for 3 h. Benzene and excess thionyl chloride were removed *in vacuo;* absolute ethanol (25 ml) was added and again distilled off *in vacuo*. The residue was recrystallised from ethyl acetate. White plates (4.7 g) of m.p. 150°C. (Found: C 37.25; H 3.57; Cl 13.67; S 18.60. Calc. for $C_{16}H_{18}Cl_2N_2O_7S_3$: C 37.14; H 3.50; Cl 13.70; S 18.59.)

3. N-(β-Chloroethyl)-p-chlorobenzenesulfonamide. N-(*β*-Hydroxyethyl)-*p*-chlorobenzenesulfonamide (45 g), thionyl chloride (90 ml) and pyridine (0.5 ml) were refluxed for 4 h. Excess thionyl chloride was removed *in vacuo;* 50 ml of absolute ethanol was added and subsequently removed *in vacuo*. The residue was recrystallised from ethyl acetate + petroleum ether. Yield 45 g (90 %). M.p. 84–85°C. (Found: C 38.00; H 3.42; Cl 27.55.Calc. for $C_8H_9Cl_2NO_2S$: C 37.82; H 3.57; Cl 27.90).

4. p-Chlorobenzene sulfonyl derivatives of amino acids. To a solution of the amino acid (0.1 mole) dissolved in 0.1 N sodium hydroxide (200 ml), a solution of *p*-chlorobenzenesulfonyl chloride (21.2 g, 0.1 mole) in chloroform (100 ml) was added, and the mixture was stirred vigorously for 2 h at room temperature and for an additional half hour at 40°C. The aqueous layer was separated and filtered. The acid was separated by addition of excess hydrochloric acid. The precipitate was filtered and recrystallised from water.

Yields: p-*Chlorobenzenesulfonylglycine.* M.p. 173°C. The compound has already been prepared by Nikolenko [2].

p-*Chlorobenzenesulfonyl-α-alanine.* M.p. 155°C. (Found: C 41.15; H 3.85; N 5.14. Calc. for $C_9H_{10}ClNO_4S$: C 41.19; H 3.81; N 5.30).

p-*Chlorobenzenesulfonyl-β-alanine.* M.p. 157.5°C. (Found: C 40.80; H 3.74; N 5.19. Calc. for $C_9H_{10}ClNO_4S$: C 41.19; H 3.80; N 5.30.)

p-*Chlorobenzenesulfonylaminophenylacetic acid.* M.p. 181–182°C. (Found: C 51.60; H 3.68; N 4.47. Calc. for $C_{14}H_{12}ClNO_4S$: C 51.62; H 3.71; N 4.30.)

p-*Chlorobenzenesulfonylaminoacetyl chloride.* The acid (12 g, 0.05 mole) was dissolved in thionyl chloride (50 ml) and the mixture refluxed for 2 h. Excess thionyl chloride was removed *in vacuo* and the residue crystallised from benzene. Yield 10 g (85 %). M.p. 130–131°C. (Found: C 36.10; H 2.79; N 5.54; Cl 25.80. Calc. for $C_8H_7Cl_2NO_3S$: C 35.85; H 2.63; N 5.23; Cl 26.45.)

In the same way were prepared:

α-(p-Chlorobenzenesulfonylamino)-propionyl chloride. Recrystallised from *n*-heptane. Decomp. at 100–120°C. (Found: C 38.90; H 3.15. Calc. for $C_9H_9Cl_2NO_3S$: C 38.50; H 3.20.)

β-(p-Chlorobenzenesulfonylamino)-propionyl chloride. Recrystallised from benzene + *n*-heptane. Decomp. at 100–120°C. (Found: C 39.10; H 3.79. Calc. for $C_9H_9Cl_2NO_3S$: C 38.50; H 3.20.)

The following amides were prepared by reaction of the acid chloride with an excess of the amine in benzene solution; they were recrystallised from water:

p-*Chlorobenzenesulfonylglycine isopropylamide.* M.p. 178°C. (Found: C 45.15; H 5.20. Calc. for $C_{11}H_{15}ClN_2O_3S$: C 45.13; H 5.20.)

p-*Chlorobenzenesulfonylglycine n-butylamide* M.p. 146°C. (Found: C 46.95; H 5.53. Calc. for $C_{12}H_{17}ClN_2O_3S$: C 47.28; H 5.62.)

p-*Chlorobenzenesulfonyl-β-alanine isopropylamide.* M.p. 143°C. (Found: C 47.50; H 5.68; N 9.10. Calc. for $C_{12}H_{17}ClN_2O_3S$: C 47.28; H 5.62; N 9.19.)

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