

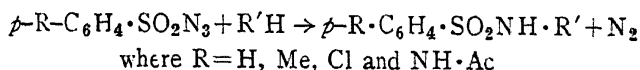
Organization, as in America and other European countries.

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ACTION OF SULPHONAZIDES ON HETEROCYCLIC COMPOUNDS

IN exploring the various possible methods of synthesis of sulphanilamido derivatives of heterocyclic compounds, we came across the reaction discovered by Curtius and Rissom¹ which appeared to give access to compounds otherwise difficult to prepare. This reaction, which can be represented as follows



has been extensively studied by Curtius and collaborators.¹ It has been found that in case R'H is benzene, toluene, xylene or naphthalene, the reaction proceeds as represented above, but with aniline, mono-methyl and dimethylaniline it is more complicated yielding other products also, one of them being the sulphonamide ($\text{R}\cdot\text{C}_6\text{H}_4\cdot\text{SO}_2\text{NH}_2$) arising from the azide. While pyridine yields a (2- or 3-substituted) benzenesulphonamido derivative, with quinoline the only crystalline product isolated is the sulphonamide corresponding to the starting azide.

We have studied, in the first instance, the action of acetsulphanilylazide² on thiazole, 2:4-dimethylthiazole, 2-hydroxy-4-methyl thiazole, pyridine and glyoxaline.

Thiazole and 2:4-dimethylthiazole in excess were kept boiling with acetsulphanilylazide till there was no more evolution of nitrogen (12 to 24 hours). In both the cases, the volume of gas collected corresponded roughly to one molecular equivalent. From the reaction products, the only crystalline product that could be isolated in the two cases was *p*-acetaminobenzenesulphonamide in yields of 25 and 5-10 per cent. respectively. Similarly *p*-toluenesulphonamide and 2:4-dimethylthiazole furnished only *p*-toluenesulphonamide in 75 per cent. yield. The action of acetsulphanilylazide on 2-hydroxy-4-methylthiazole led to no tangible product.

Pyridine on boiling with acetsulphanilylazide as described above furnished in poor yields an acetsulphanilamidopyridine, m.p. 280°, which appears to be the 3-substitution product (the m.p.s of the 2-, 3-, and 4-substituted products recorded in literature³ are 224-227°, 272-275° and 252° respectively).

Glyoxaline in sharp contrast to all compounds studied so far reacted very violently with one molecular equivalent of acetsulphanilylazide at 80 to 110°; one molecular equivalent of nitrogen was evolved in less than a minute and the product obtained was a tar. When, however, the reaction was carried out in small quantities carefully regulated, one molecular equivalent of nitrogen was evolved in about thirty minutes; the product obtained was a mixture from which two crystalline compounds were isolated (with very indefinite m.p.) none of which is identical with *p*-acetaminobenzenesulphonamide. The structures of these are under investigation.

We are studying the action of the sulphonazides on various other compounds.

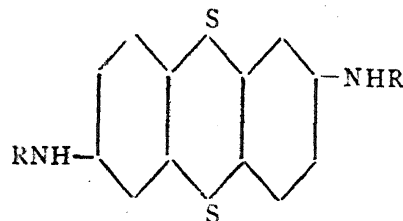
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March 29, 1943. K. GANAPATHI.

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SYNTHESIS OF SULPHANILAMIDE DERIVATIVES OF THIANTHRENE

SULPHANILAMIDO compounds possessing heterocyclic rings have come into great prominence as therapeutic agents with the discovery of sulpha-pyridine (B.P. 516288), sulphathiazole, etc. The disulphanilamido derivative of 2:6-diamino-thianthrene has now been prepared.

2:6-Diacetaminothianthrene was prepared according to the method of Ray.¹ The corresponding diamine was obtained from the diacetyl compound by hydrolysis with hydrochloric acid and neutralising with alkali (yellow needles, m.p. 120°C.). *p*-Acetaminobenzenesulphochloride reacts with 2:6-diaminothianthrene to yield the diacetyldisulphanilamide of thianthrene (I) which decomposes at 180°.



(I, R = $-\text{SO}_2\cdot\text{C}_6\text{H}_4\cdot\text{NHAc}$)

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March 12, 1943.

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