

The projection co-ordinates are being refined. The third (x) co-ordinates have yet to be determined. But, even at this stage, it is apparent that the molecule has nearly a planar configuration. Only the O_3-C_9 bond in the 4-methoxy group seems to be significantly off the general plane of the rest of the molecule.

Further work on the complete structure determination is in progress.

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PREPARATION OF O-NITRO-ACETANILIDE

THE direct nitration of acetanilide results in the formation of *para*-compound in preponderant quantity. Turner,¹ therefore, prepared the *ortho*-compound by first sulphonating the acetanilide so that the *para* position is occupied by the sulpho-group, the product was nitrated and then the sulphonic acid group was removed by hydrolysis. Witt and Utermann² have prepared *ortho*- and *para*-nitro-acetanilides by nitrating acetanilide, in glacial acetic acid medium, with nitric acid. Menke³ synthesised O-nitro-acetanilide by treating aniline with copper nitrate in acetic anhydride medium. Roeder and Day⁴ prepared this compound by acetylating O-nitro-aniline. Recently Itaya *et al.*⁵ have prepared *ortho*-nitro-acetanilide by nitrating acetanilide with copper nitrate in acetic anhydride medium at 50° C.

During the course of our investigations, we have developed a simple method for the preparation of this compound in 95% yield.

Experimental.—One kilogram of acetanilide (7.408 Moles) was suspended in 5 litres of acetic anhydride (7.467 Moles) in a ten-litre open mouth stainless steel vessel fitted with an efficient stirrer, dropping funnel and a thermometer. The suspension was cooled in an ice-salt mixture. The suspension of the crystals was dissolved in subsequent reaction. 1.2 litres of HNO_3 , sp. gr. 1.418, were cooled to 0–5° C. and added slowly into the cooled solution of acetanilide in acetic anhydride at 5° to 10° C. with vigorous stirring. The reaction

vessel was kept cool with mixture of ice and salt during the whole operation. The addition was completed in 5 hours and then the reaction mixture was allowed to stand for one hour with stirring. The reaction being exothermic, extreme precautions were taken not to allow the temperature of the mixture to rise, by slow addition of nitric acid and efficient cooling for two hours in the beginning when 1/3 of the acid was added, later on, the reaction proceeded smoothly. After completing the reaction, the reaction mixture was poured in ice-water when an orange-yellow precipitate of O-nitro-acetanilide separated.

The precipitate was filtered and washed with water till it was free from acid. The yield of the crude compound was 98%. The material was crystallised from hot water to get a pure O-nitro-acetanilide, m.p. 93° C. The weight of the pure material was 1267 gm. This method gives mainly O-nitro-acetanilide, the overall yield being 95%. The identity of the material was confirmed by estimation of nitro-group, carbon, hydrogen and nitrogen analysis, and by preparation of derivatives.

Analysis: Calculated for $C_8H_8O_3N_2$: C, 53.33; H, 4.44; N, 15.56.

Found: C, 53.45; H, 4.50; N, 15.51.

On hydrolysis with 10% HCl it gave O-nitro-aniline m.p. 72° C. and on reduction in neutral medium it gave O-azoxy-acetanilide, m.p. 185° C. Infrared spectrum was recorded on a Perkin Elmer Infracord model No. 137 using potassium bromide pellet. The I.R. spectrum was found identical with that of this compound prepared by Witt and Utermann² method, showing peaks at 5.9 μ , 6.65 μ , 7.42 μ and 7.87 μ .

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