

NOTES

N¹-Silver¹ Derivatives of Sulfanilamide and Some Related Compounds

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The N¹-silver derivative of sulfanilamide was prepared in small amount by Scudi² for the purpose of identifying sulfanilamide. The yield was not high and the method employed was impractical for the preparation of the silver salt in quantity.

For use in synthetic work, we have prepared the N¹-silver derivatives of sulfanilamide, N⁴-acetyl sulfanilamide and sulfapyridine in quantity, and present detailed procedures here for those interested in this field.

Experimental

N¹-Silver Sulfanilamide.—N¹-Sodium sulfanilamide was prepared in 97% yield from sulfanilamide and alcoholic sodium hydroxide by the procedure which Marshall, Bratton and Litchfield³ used for the preparation of N¹-sodium sulfapyridine.

Silver nitrate (14.0 g. or 0.082 mole) in 100 cc. of distilled water was added with vigorous stirring to a solution of N¹-sodium sulfanilamide (15.0 g. or 0.0773 mole in 100 cc. of water). Immediately a voluminous white precipitate formed which was filtered off with suction (using a fine filter paper) and washed with cold water until the washings were entirely free of silver ions. The washed precipitate was then spread upon a porous plate and allowed to dry in the dark for twenty-four hours. It was then removed from the plate and dried in the dark in a vacuum desiccator over potassium hydroxide sticks for forty-eight hours and finally in the oven at 100° for four hours.

Anal. Calcd. for C₆H₇O₂N₂SAg: Ag, 38.68. Found: Ag, 39.47, 39.25.

N¹-Silver N⁴-Acetylsulfanilamide.—N⁴-Acetylsulfanilamide⁴ (58.0 g. or 0.271 mole) was mixed with a small amount of water to make a thick paste. An equimolecular quantity of finely ground sodium hydroxide was then added to the paste. After the mixture had been stirred for a short time, complete solution was obtained. The solution was filtered and the filtrate poured into five times its volume of absolute ethyl alcohol and allowed to remain in the ice chest overnight. The heavy white precipitate which had formed was filtered off, washed with chilled absolute ethyl alcohol and dried in a vacuum desiccator over potassium hydroxide sticks. The N¹-sodium N⁴-acetylsulfanilamide was not purified but was converted directly into the N¹-silver derivative.

Sixty-two grams (0.262 mole) of the N¹-sodium salt was

dissolved in a small volume of distilled water. To the solution was added an aqueous solution containing an equimolecular quantity of silver nitrate. The N¹-silver derivative, which formed at once as a heavy white precipitate, was washed and dried exactly as described above for N¹-silver sulfanilamide.

Anal. Calcd. for C₈H₉O₂N₂SAg: Ag, 33.64. Found: Ag, 34.07, 34.17.

N¹-Silver Sulfapyridine.—N¹-Sodium sulfapyridine was prepared in 80% yield by the method of Marshall, Bratton and Litchfield.³

The sodium derivative, dissolved in a small volume of distilled water, was converted into N¹-silver sulfapyridine with aqueous silver nitrate as described under N¹-silver N⁴-acetylsulfanilamide. The silver salt, which was white and gelatinous when first precipitated, was washed and dried as described above.

Anal. Calcd. for C₁₁H₁₀O₂N₂SAg: Ag, 30.29. Found: Ag, 30.12, 30.14.

The yields of N¹-silver derivatives (based upon the N¹-sodium salts) were practically quantitative in all three preparations.

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The Germicidal Activity of Certain Organic Compounds¹

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The germicidal activity of some ten substances against *Escherichia coli*, *Bacillus subtilis*, *Staphylococcus aureus* and *Eberthella typhi* (Hopkin's strain) has been measured using the "wet filter paper method" as developed by Ruehle and Brewer,² except that the indications of growth were, in the case of *E. coli*, the formation of gas, in the case of *B. subtilis*, the growth of a pellicle, and in the case of *Staph. aureus* and *E. typhi*, turbidity or cloudiness. The periods of observation were thirty, sixty and ninety minutes.

Two tests were performed with each substance and each organism except in the case of *Escherichia coli*, where five to seven tests were carried out. Six of the substances were inactive, namely, 4-bromo-2-naphthol, 2-butyro-1-naphthol, 4-bromo-2-propionyl-naphthol, 2-propionyl-1-naphthol

(1) The nomenclature used in this paper is that proposed by Crossley, Northey and Hultquist, *THIS JOURNAL*, **60**, 2217 (1938).

(2) Scudi, *Ind. Eng. Chem., Anal. Ed.*, **10**, 346 (1938).

(3) Marshall, Bratton and Litchfield, *Science*, **88**, 597 (1938).

(4) Miller, Rock and Moore, *THIS JOURNAL*, **61**, 1198 (1939).

(1) Taken from a thesis submitted by Miss Emily R. Meserve in partial fulfillment of the requirements for the Degree of Master of Science in Chemistry in June, 1941.

(2) G. L. A. Ruehle and C. M. Brewer, *U. S. D. A. Circular No.* 198 (1931).