NOTES

N^1-Silver[1] Derivatives of Sulfanilamide and Some Related Compounds

By Charles E. Braun and Jack L. Towle

The N^1-silver derivative of sulfanilamide was prepared in small amount by Scudi[2] 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 N^1-silver derivatives of sulfanilamide, N^4-acetyl sulfanilamide and sulfapyridine in quantity, and present detailed procedures here for those interested in this field.

Experimental

N^1-Silver Sulfanilamide.—N^1-Sodium sulfanilamide was prepared in 97% yield from sulfanilamide and alcoholic sodium hydroxide by the procedure which Marshall, Bratton and Litchfield[3] used for the preparation of N^1-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 N^1-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 $C_6H_7O_2N_2SAg$: Ag, 38.68. Found: Ag, 39.47, 39.25.

N^1-Silver N^4-Acetylsulfanilamide.—N^4-Acetylsulfanilamide[4] (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 N^1-sodium N^4-acetylsulfanilamide was not purified but was converted directly into the N^1-silver derivative.

Sixty-two grams (0.262 mole) of the N^1-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 N^1-silver derivative, which formed at once as a heavy white precipitate, was washed and dried exactly as described above for N^1-silver sulfanilamide.

Anal. Calcd. for $C_8H_9O_3N_2SAg$: Ag, 33.64. Found: Ag, 34.07, 34.17.

N^1-Silver Sulfapyridine.—N^1-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 N^1-silver sulfapyridine with aqueous silver nitrate as described under N^1-silver N^4-acetylsulfanilamide. The silver salt, which was white and gelatinous when first precipitated, was washed and dried as described above.

Anal. Calcd. for $C_{11}H_{10}O_2N_3SAg$: Ag, 30.29. Found: Ag, 30.12, 30.14.

The yields of N^1-silver derivatives (based upon the N^1-sodium salts) were practically quantitative in all three preparations.

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[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).

The Germicidal Activity of Certain Organic Compounds[1]

By H. L. Cole, C. C. Prouty and Emily R. Meserve

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-propionylnaphthol, 2-propionyl-1-naphthol

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[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).

ethyl ether, 2-propyl-1-naphthol ethyl ether and 2-aceto-1-naphthol. 2-Propionyl-1-naphthol was active only against *E. typhi*, killing it in sixty minutes. 2-Ethyl-1-naphthol, 4,6-diethylresorcinol and 2-propyl-1-naphthol killed all of the organisms in thirty minutes, except that the last substance did not kill *E. coli*. These more promising substances are being studied further.

Acknowledgment is made to Miss Catherine Ulrich and to the late G. L. A. Ruehle for cultures of the organisms, and to Professor C. M. Brewster for the purification and preparation of the organic compounds studied.

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Partition Ratios of Some Organic Acids between Water and Ethyl Ether[1]

BY O. C. DERMER, WENDELL G. MARKHAM AND H. M. TRIMBLE

In the course of investigating the use of partition ratios for the identification of organic acids, it became clear that literature dealing with these ratios is fragmentary and even untrustworthy for some common acids. The work here reported concerns the measurement of the distribution of sixteen organic acids between water and ethyl ether at 25°. The results, expressed in the form used by the "International Critical Tables,"[2] are set forth in Table I. The ratios are uncorrected for any difference in association or dissociation in the two phases.

TABLE I

PARTITION RATIOS

Acid	Concn. in ether layer, C_e, N	Ratio C_w/C_e
Acrylic	0.0574	0.504 ± 0.002
	.115	.482 ± .002
	.302	.451 ± .002
	.604	.440 ± .002
Adipic	.0267	1.95 ± .02
	.0670	1.90 ± .02
	.108	1.88 ± .02
	.138	1.84 ± .02
Barbituric	.00080	38 ± 3.0
	.00124	43 ± 2.0
	.00183	43 ± 2.0
Benzenesulfonic	.00028	310 ± 40.0
	.00032	540 ± 40.0
	.00063	690 ± 40.0
	.00115	750 ± 40.0
Bromoacetic[a,b]	.0802	0.306 ± 0.002
	.163	.286 ± .002
	.410	.265 ± .002
	.820	.250 ± .002
Chloroacetic[a,b]	.0671	.457 ± .002
	.138	.439 ± .002
	.351	.394 ± .002
	.708	.373 ± .002
Crotonic[a]	.0741	.253 ± .001
	.151	.238 ± .001
	.380	.213 ± .001
	.755	.188 ± .001
Dichloroacetic[a,b]	.0908	.176 ± .002
	.190	.139 ± .002
	.488	.112 ± .002
	.942	.102 ± .002
Formic[a]	.0264	2.78 ± .03
	.0530	2.80 ± .03
	.139	2.75 ± .03
	.286	2.62 ± .03
Furoic	.0392	0.286 ± .003
	.0791	.265 ± .003
	.203	.237 ± .003
	.342	.220 ± .003
Glycine	.00086	106 ± 4
	.00108	162 ± 4
	.00129	313 ± 4
	.00148	485 ± 4
Glycolic[a,b]	.00248	34.7 ± 0.8
	.00488	35.2 ± .8
	.0121	35.7 ± .8
	.0244	35.5 ± .8
α-Hydroxyisobutyric	.0154	4.49 ± .05
	.0313	4.39 ± .05
	.0808	4.19 ± .05
	.179	3.85 ± .05
Succinic[a]	.0100	7.75 ± .04
	.0201	7.79 ± .04
	.0512	7.67 ± .04
	.104	7.52 ± .04
Tartaric[a]	.00034	280 ± 50
	.00075	260 ± 10
	.00172	282 ± 5
	.00329	294 ± 5
Trichloroacetic[a,b]	.101	0.236 ± 0.003
	.187	.174 ± .003
	.475	.122 ± .003
	.920	.101 ± .003

[a] For literature see ref. 2. [b] *Cf.* ref. 3.

It is difficult to compare our findings with literature values because nearly all other investigators used either more dilute solutions or differ-

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[1] This paper is based upon a thesis submitted by Mr. Markham in partial fulfillment of the requirements for the degree of Master of Science at the Oklahoma Agricultural and Mechanical College in 1941.

[2] "International Critical Tables," Vol. III, McGraw-Hill Book Co., New York, N. Y., 1928, pp. 422–433.